

# Crystal Growth and Characterization of Nickel (II) Cinnamaldehyde Sulfate Optical Crystal by Slow Evaporation Method

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This paper describes the growth of Nickel (II) cinnamaldehyde sulfate (NCS) in an entirely new metal-organic nonlinear optical crystal at ambient temperature using the slow evaporation method. The produced crystals were characterized by single-crystal, powder X-ray diffraction techniques, UV-Visible, FT-IR, TG/DTA, SEM. Single crystal XRD analyses were performed on the grown crystal in order to validate the lattice attributes. In order to determine the functional groups of the developed crystal, Fourier transform infrared (FTIR) analysis was performed. After analyzing the UV-vis NIR spectrum, 3.25 eV was determined to be the calculated band gap energy. Thermal gravimetric differential thermal analysis (TG-DTA) was used to examine the generated crystal's thermal behavior. This indicates that weight loss does not occur up to 160°C. The SEM

and EDAX examination also revealed the surface shape of the NCS crystal. A range of 280–900 nm is examined for the grown crystal in the fluorescence investigation. The effectiveness of the formed crystal's second harmonic generation (SHG) was examined using the Kurtz-Perry powder technique.

**Keywords:** Crystal growth, UV- Vis, FTIR, Single X-ray Diffraction, SEM-EDAX, TG/DTA, Fluorescence studies.

## 1. Introduction

Nonlinear optical (NLO) crystals have emerged as a possible alternative to their inorganic counterparts due to their superior features, such as high NLO coefficients and quick reaction times. The benefit of metal-organic materials is that they have a high laser damage threshold due to structural changes in their optical properties, in addition to having rich nonlinear optical performance [1-2]. Finding and creating new materials with extraordinary nonlinear optical characteristics for useful applications has received a lot of attention lately. Nonlinear optical (NLO) materials are essential for optical switching, modulation, colour display, medical diagnostics, computing, and information processing and frequency conversion in modern telecommunication and optoelectronics technologies. The aforementioned features prompted material scientists to search for innovative materials for nonlinear optical applications.

Every day, scientists use novel synthesis strategies to find new organic, inorganic and semi organic complex molecules. To date, a large number of crystals exhibiting nonlinear optical properties have been produced, along with closely related isomorphs. The change in the electronic sector has led to a greater demand for crystals, which has made improvements in both technical and economic aspects necessary. Because of their easy molecular arrangement customization, higher  $\beta$  values, high optical damage resistance, multifunctional substitution, low dielectric constant and maneuverability for specific device applications, organic NLO crystals have superior second and third-order NLO properties over inorganic crystal [3-5].

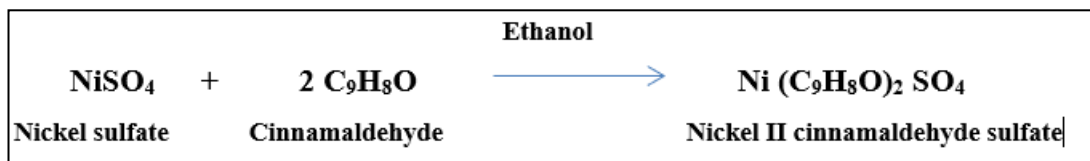
A vast worldwide effort has been made in recent years to create metal-organic compounds with very effective NLO characteristics. Using organic molecules as NLO materials has the benefit of providing a wide range of design options through the introduced of desired substituents and the selection of acceptable reactants for conducting chemical processes. Adding an electron-donating (donor) or electron –with drawing (acceptor) group to the  $\pi$  –electron conjugated system is a popular method to boost the molecule's nonlinear polarization. Nonlinear polarization, which is typically accompanied by a conjugated system in organic materials, is the outcome of the interaction between strong electromagnetic waves and laser light. In this study, we synthesized a high-quality; transparent metal-organic compound Nickel (II) cinnamaldehyde sulfate (NCS) at room temperature using a slow evaporation approach. Cinnamaldehyde is a naturally-occurring organic compound responsible for the unique flavor and aroma of cinnamon. Cinnamon aldehyde is also known as the sweetest discovered element because it tastes so good. Aromatic aldehyde means the class of chemical compound. Nickel sulfate is the metal compound which is composed of Nickel (Ni), Sulfur(S) and Oxygen (O) with the chemical formula ( $\text{Ni SO}_4$ ), a blue crystalline solid, is the most often seen of its numerous hydrated forms [6]. Organic and metal compound was synthesized to form Condensed Cinnamaldehyde with Nickel Sulfate. Similarly, a non-

Centro symmetric single crystal with space group P was generated by synthesizing cinnamaldehyde with Nickel Sulfate. The previously mentioned details have been taken into consideration when creating a new NLO materials. The ring nitrogen atom in the complex's hydrogen bonding is what causes salts to develop, and similar effects previously been investigated in arrange of organic types. Various characterization experiments were performed on the produced NCS crystal, including single crystal XRD, PXRD, FTIR, UV-visible absorbance, fluorescence, and NLO properties were executed. The production and development of cinnamon aldehyde nickel sulfate are reported here.

## 2. Materials and Methods

### 2.1 Experimental Section

To form a NCS crystal, 0.154g of Nickel sulfate is dissolved in 10 ml distilled water and 0.264 ml of cinnamon aldehyde were combined added in the 100ml beaker, then added 5 ml of ethanol. Slowly diffuse the cinnamaldehyde solution into the nickel sulfate solution to create a homogenous solution, the components of the solution were agitated two hours stir at 50<sup>0</sup>c using a magnetic stirrer [7-8]. Allow the mixture to stand at room temperature for 2-3 days. The resulting solution was then filtered through Whatman filter paper with a 15micrometer pore size [3]. The NCS –saturated solution was then transferred to a beaker with a perforated lid to regulate the rate of evaporation, and it was left to crystallize at room temperature. After ten to twelve days, a single crystal of NCS was produced using the slow evaporation approach depicted in figure 1. High grade crystals were taken out for investigations into characterization. The following equation describes nickel II cinnamaldehyde sulfate.



Ethanol was used as a solvent in a recrystallization process to purify the final product. This procedure is used to increase the transparency and purity of the grown crystal. The shape of the as grown NCS crystal is shown in figure.1 respectively.

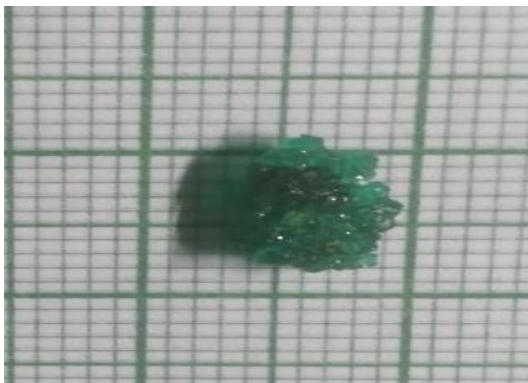


Figure 1: Grown Crystal of NCS

2.2 Characterization Studies:

Various studies on the characterization of NCS are performed to explore its crystal growth, structural, thermal, optical and nonlinear optical applications [9]. The unit cell parameters of NCS were obtained from a single crystal X-ray diffraction study carried out in Enraf nonius cad 4-mv diffractometer with MoKa( $\lambda=0.7070 \text{ \AA}$ ). The NCS crystal underwent FTIR spectrum was recorded in KBr pellets using a Perkin-Elmer spectrometer in the range of 400–4000  $\text{cm}^{-1}$ . Thermal stability of the title compound was studied by TG/DTA analysis NETZSCA STA 409 instrument at a heating rate 20<sup>0</sup> C per minute from 50<sup>0</sup>C to 500<sup>0</sup>C. An NCS crystal was recorded for optical behavior from 200 to 800 nm a VARIN CARY 5E UV-Vis Spectrophotometer. The NLO efficiency of the formed crystal was tested using KURTZ powder method and an ND:YAG laser Quanta Ray Model Lab-170-10.

3. Results and Discussion

3.1 Single Crystal X-Ray Diffraction and Fundamental Parameters:

The ENRAF NONIUS CAD 4-MV31 Diffractometer, was used to collect data on a single crystal [10]. The generated crystal is identified as belonging to tetragonal crystal system with space group P by single crystal x-ray Diffraction refinement performed on the grown Cinnamaldehyde Nickel Sulfate single crystal is shown in fig.2 and the crystallographic data [5],[6].Table.1 shows the calculated lattice parameters for the Central nervous system  $\alpha=90^0, \beta=90^0, \gamma=90^0$  and the cell volume  $V=844 \text{ \AA}^3$ . The Crystal that has developed has a density of 2.178g  $\text{cm}^3$ .

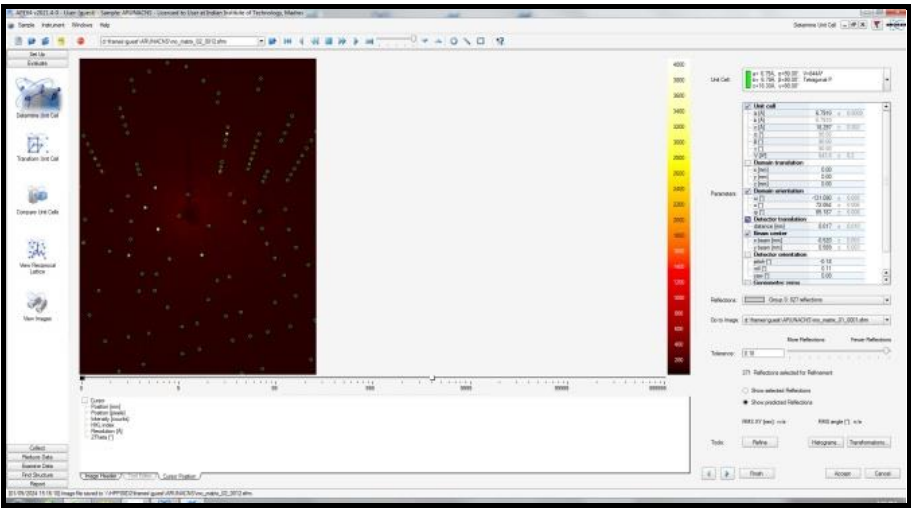


Figure 2: Single XRD of NCS

Table1. Single XRD Spectrum of NCS

| Crystal System | A    | B    | C     | Alpha | Beta | Gamma | Volume             |
|----------------|------|------|-------|-------|------|-------|--------------------|
| Tetragonal     | 6.79 | 6.79 | 18.30 | 90°   | 90°  | 90°   | 844 $\text{\AA}^3$ |

### 3.2 Powder X-Ray Diffraction

The  $2\theta$  values from powder diffraction spectra were utilized to index the peaks. Figure.3 depicts the PXRD diffractogram recorded for the newly formed crystal. Precise peaks at specific Bragg angles indicate a crystalline substance [11]. Investigation was focused on the purity as well as the crystallinity of the synthesized compound. The thin powder sample of NCS single crystal was subjected to PXRD measurements. The observed data for as synthesized sample ranged from 10-90 degree ( $2\theta$ ) at room temperature. Distinguishable peaks at a constant  $2\theta$  value identified through powder X software indexing confirmed the state of crystallization of NCS crystals. The crystal under study showed strong diffraction patterns. Based on PXRD, this data reveal that formed NCS crystal is characterized by multiple diffracted planes, with the most intense being (001), implying a higher density packing compared to other planes.

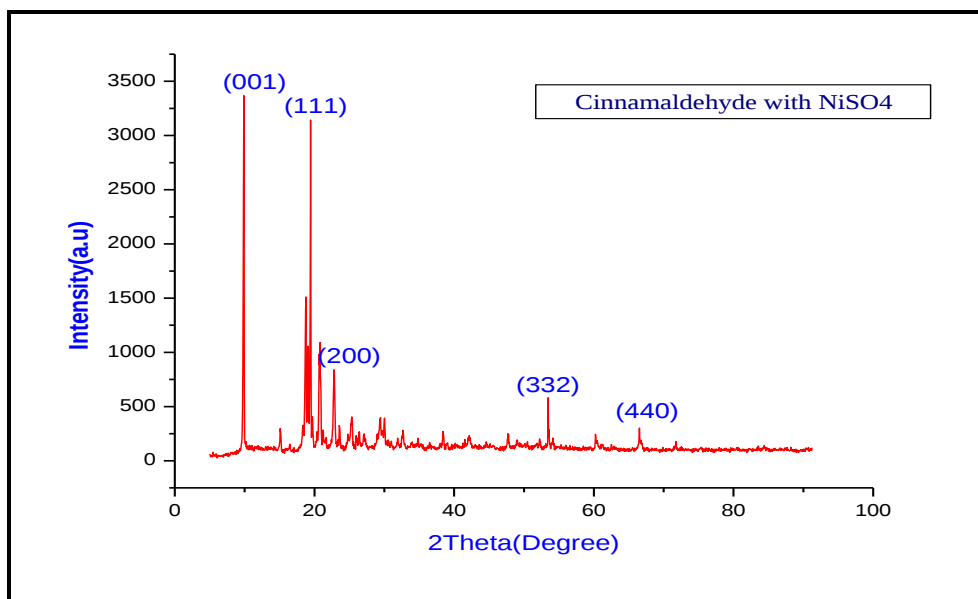


Figure 3:PXRD Spectrum of NCS

### 3.3 FT-IR Spectral studies of NCS Crystal

Figure.4 shows the FTIR spectrum of the NCS crystal. The Table shows the measured vibrational wavenumber as well as suggested allocations. A Prominent peak in the NCS FTIR spectra is found at  $3390\text{ cm}^{-1}$  which is due to olefinic C-H stretching. Sulfate's H=O stretching vibration was detected at  $3074\text{ cm}^{-1}$ , While the C-H stretching vibration was observed at  $2926\text{ cm}^{-1}$ . The FTIR spectrum indicates that the  $\text{-CH=CH-}$  bending mode occurs at  $981.72\text{ cm}^{-1}$  and the C=O vibration is found to lie at  $1629.98\text{ cm}^{-1}$ . The vibration of C-S is at  $630.12\text{ cm}^{-1}$ . The C-H Stretching of the aldehyde group and the CH of the planar vibration mode of the NCS Crystal are responsible for the significant absorption shown at  $1066\text{ cm}^{-1}$  and  $626\text{ cm}^{-1}$  [12-13]. The Table.2 shows the FT-IR spectrum of Nickel II Cinnamaldehyde sulfate.

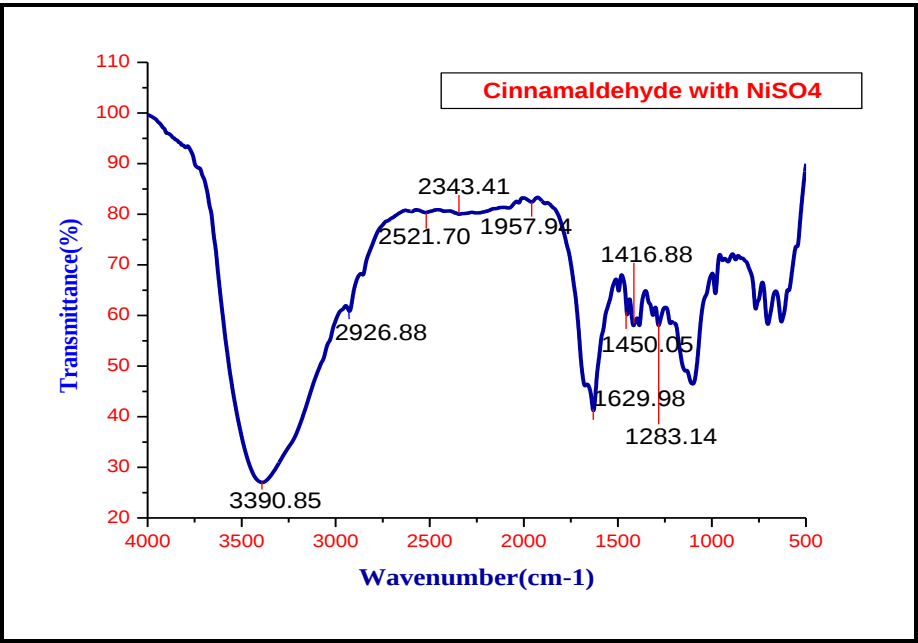


Figure 4: FT-IR Spectrum of NCS

Table. 2. FT-IR Spectrum of NCS

| Wavenumber(Cm <sup>-1</sup> ) | Vibrational assignment          |
|-------------------------------|---------------------------------|
| 3390                          | olefinic CH stretching          |
| 2926.88                       | C-H stretching in plane bending |
| 1957.94                       | CH out of plane aromatic        |
| 1629.98                       | C=O Stretching                  |
| 981.72                        | -CH=CH-                         |
| 630.12                        | C-S Stretching                  |

3.4 Optical Properties

3.4.1 UV-Visible Studies of NCS Crystal

Figure 5(a) and 5(b) show the optical spectral absorption and transmittance spectrum of the NCS. The NCS crystal has strong optical transmission across the visible spectrum and the lower cut-off wavelength of 278.60 nm observed. Because the absorption of ultraviolet and the visible light requires needs the stimulation of electrons from the ground state to higher energy levels, UV-Vis spectral analysis also provides crucial information on the formed crystal’s electrical structure [14-15]. The transition peak for NCS crystal from the absorbance spectrum is tabulated in table 3.

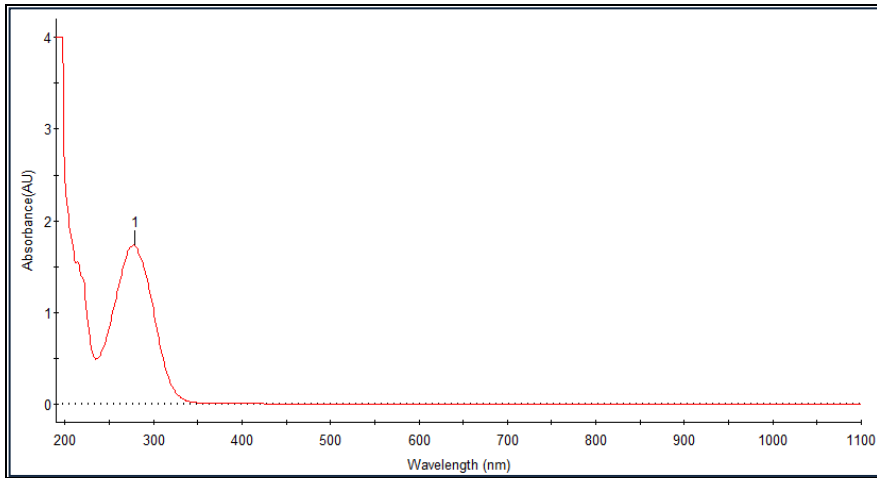


Figure 5: (a)

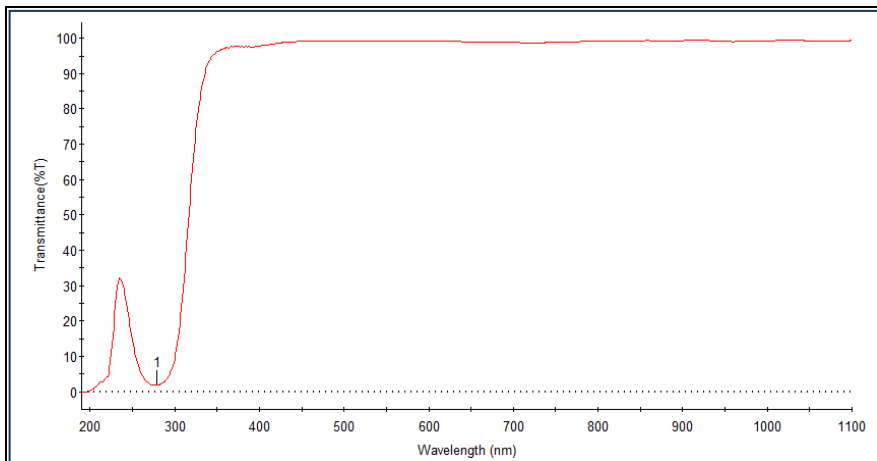


Figure 5: (b)

Figure 5: UV-Visible spectrum of NCS

Table 3: UV –Vis spectra table of NCS

| $\lambda_{\max}$ Value (nm) | Transition |
|-----------------------------|------------|
| 278.60                      | n-s*       |

### 3.4.2 Optical Band Gap

Tweezers were used to prepare the sample before placing it on a glass slide. The sample was then covered with a fresh glass slide, each with taps on both sides to keep it in place. The band gap was also determined in grown NCS crystals [16]. The expanded trend line in figure.6 illustrates the energy band gap of the rough mixed crystal of NCS. The band gap of a rough mixed crystal with Cinnamaldehyde doped Nickel Sulfate is estimated to be 2 eV, implying

that the line crossed the X-axis. Cinnamaldehyde doped Nickel Sulfate crystals. The predicted band gap for Cinnamaldehyde doped Nickel sulfate is 3.25 eV.

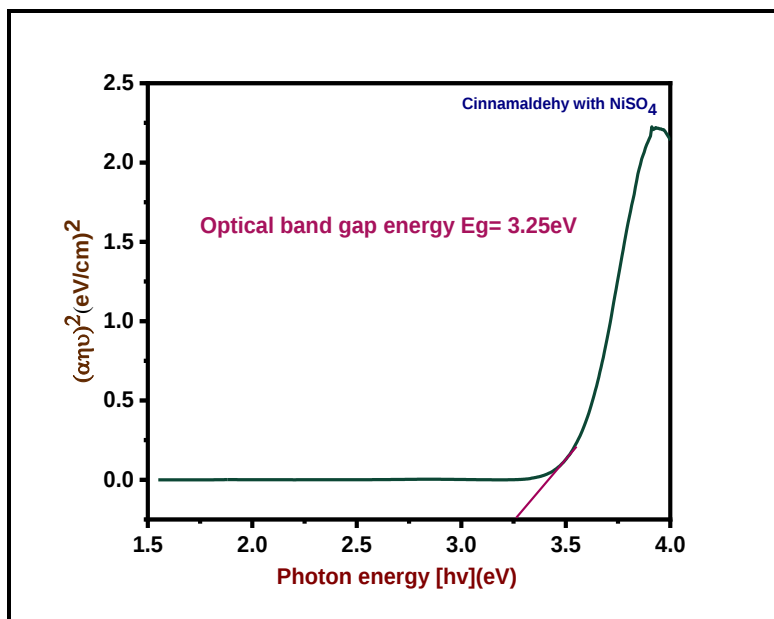


Figure 6: Band gap Energy of NCS

### 3.4.3 Fluorescence investigations

In aromatic compounds, Fluorescence is a common phenomenon. It contains numerous conjugated double bonds and exhibits remarkable resonance stability. The NCS crystal was crushed to completion using a mortar and pestle. In luminescence experiments, powder samples are typically used for aromatic compounds with many conjugated double bonds and high resonance stability. A mortar and pestle were used to completely smash the NCS crystals. Powder samples were used in luminescence experiments [17-18]. A Perkin Elmer fluorescence spectrometer (Model: LS 45) was employed as the excitation source. The detector's response was flat from 200 to 900 nm. The PL spectrum of NCS grown crystals. The excitation wavelength was 396 nm. The results show that the NCS crystal emits blue-violet fluorescence light.

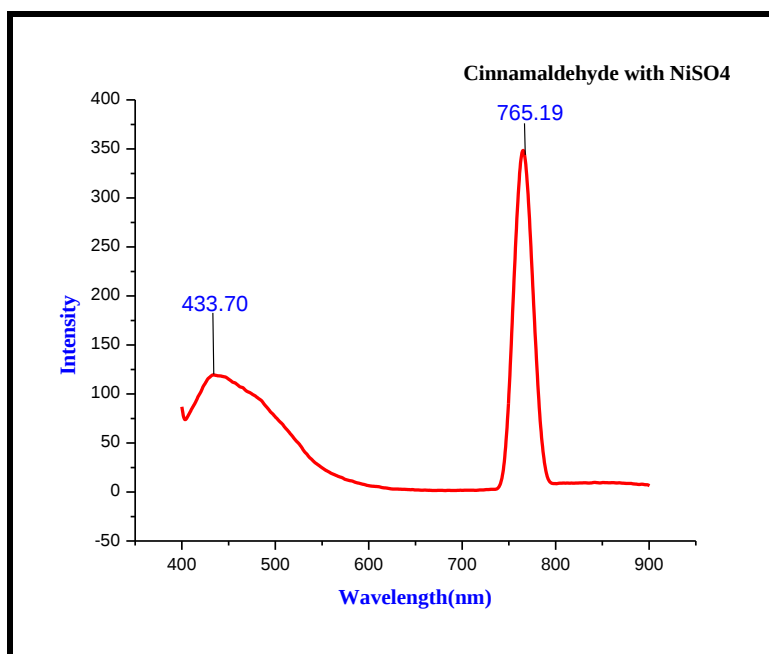


Figure 7: Fluorescence Emission Spectra of NCS

### 3.5 Thermal Analysis (TG-DTA) of NCS crystal

The melting point and thermal stability of the NCS crystal are determined using thermo gravimetric-differential thermal analysis (TG-DTA). Figure.8 shows the temperature range and characteristic curve in the nitrogen environment, which is between room temperature 0°C and 500°C at a heating rate of 20 k min<sup>-1</sup>. The material that was first removed for TG-DTA analysis weighed 33.590 g. The NCS was found to be crystal stable up to 130°C on thermal examination. The initial endothermic peak, which showed up at 130°C and coincided with a 5.5% weight loss, verifies that the NCS molecules did not include any water molecules. This second significant weight loss (endothermic peak) at 271.6°C is caused by the breakdown of volatile molecules in the NCS crystal, including N, Cl, and CO<sub>2</sub>. The peak sharpness indicates the purity and high crystallinity of the compound. Because of its strong thermal stability, the compound is suitable for use in Non-linear optical properties. [19].

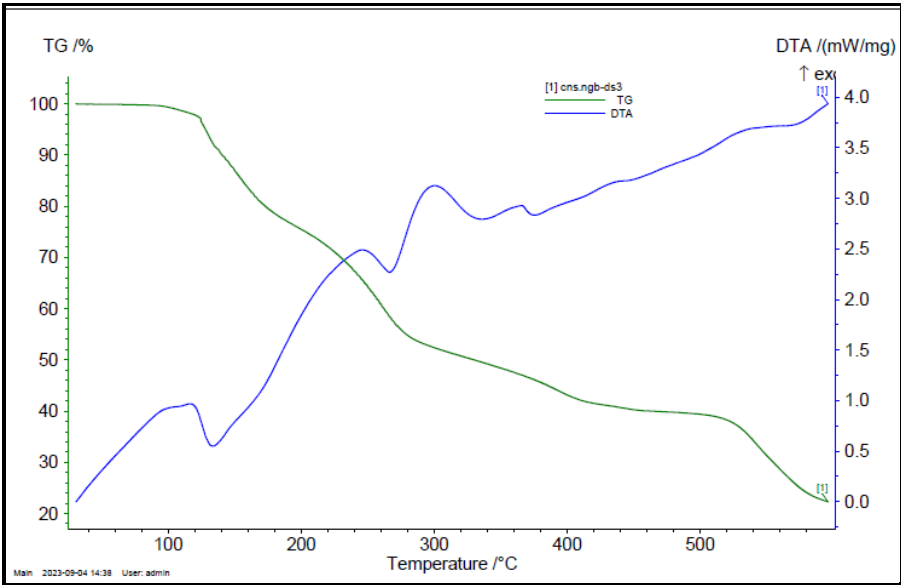
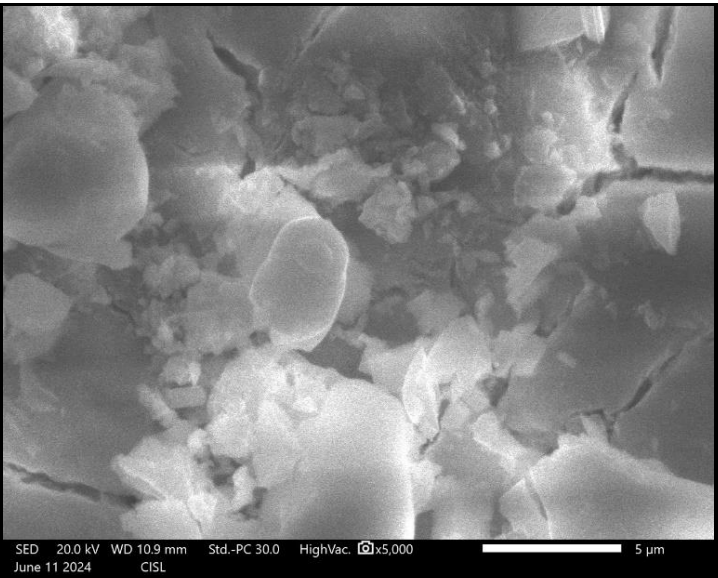


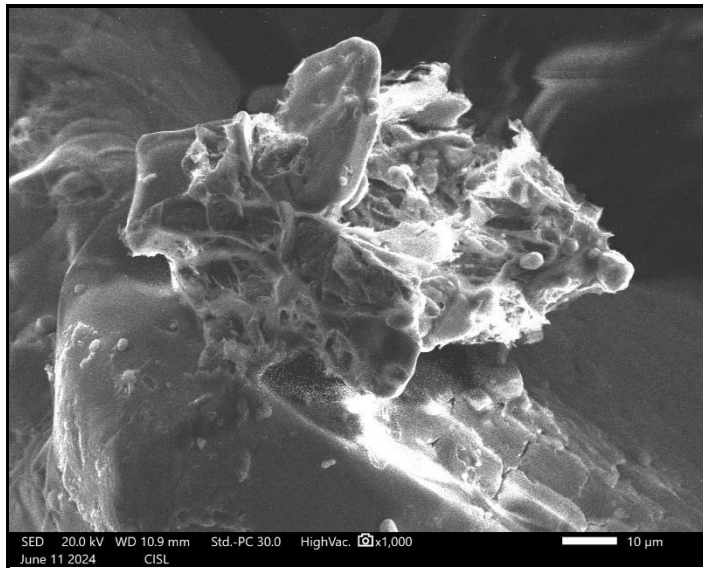
Figure 8: TG-DTA Spectrum of NCS

3.6 SEM and EDAX analysis

Fig.9 (a and b) show SEM micrographs of NCS crystals at 5 and 10  $\mu\text{m}$  resolutions, respectively. Figure 9a shows that the surface of the formed crystal is quite smooth, with a low density of flaws. Figure 9b, with better resolution 10, depicts layers of atoms of varying sizes, SEM pictures give qualitative signals into chemical composition and distribution [20-23].



(a) 5  $\mu\text{m}$  resolution



(b)10μm resolution

Figure 9: SEM micrographs of NCS Crystal

Quantitative investigation of the grown NCS crystal was conducted using Energy dispersive X-ray analysis (EDAX) Fig.10 displays the EDAX spectra of several elements in the crystal. The weight percentages of Carbon, Oxygen, Sulfur and Nickel are 53.07, 44.71, 1.50, 0.72 respectively is shown in table.4, based on the spectra's peaks [24-26].

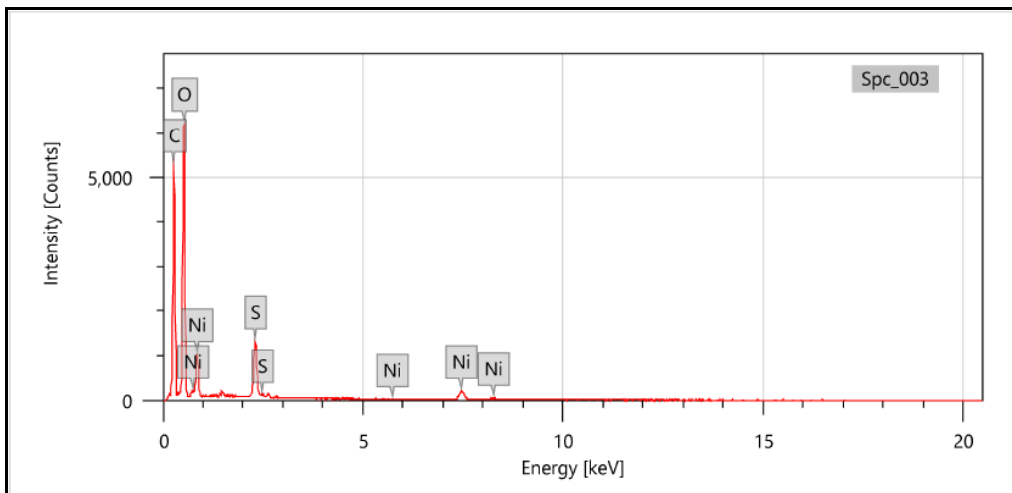


Figure 10: Energy Dispersive x-ray analysis of NCS Crystal

Table.4. Energy Dispersive x-ray analysis of NCS Crystal

| Element | Line          | Mass%      | Atom%      |
|---------|---------------|------------|------------|
| C       | K             | 44.16±0.14 | 53.07±0.16 |
| O       | K             | 49.56±0.25 | 44.71±0.23 |
| S       | K             | 3.34±0.04  | 1.50±0.02  |
| Ni      | K             | 2.95±0.007 | 0.72±0.02  |
| total   |               | 100.00     | 100.00     |
| Spc_003 | Fitting ratio |            | 0.0176     |

3.7 NLO Studies

The modified Kurtz-Perry powder technique is a useful tool for determining the second harmonic generation conversion efficiency of nonlinear optical materials. The powder sample was shown with a Q-switched high Energy Nd:YAG Laser(QUANTARAY Model LAB-170-10),Which generated light with a fundamental wavelength of 1064 nm and a pulse width of 6 nanoseconds. The laser beam with 0.50 mJ of input energy was permitted to strike the powder sample. A photomultiplier tube detected the SHG signal with a green wavelength emission at 532 nm and showed it on a storage oscilloscope with a0.70mJ energy output. The green light emission verified the existence of the second harmonic generation. The KDP sample was utilized as a reference material. The output energy of reference is shown in table.5.

The KDP sample was utilized as a reference [27-30]. The reference (KDP) has output energy of 7.5 MJ. The title crystal has a second harmonic generation efficiency that is 0.5 times greater than that of KDP.As a result, the title compound is among the most promising NLO materials for optical applications.

Table.5 SHG Efficiency of Nickel II Cinnamaldehyde sulfate with respect to KDP

| Input energy (joule) | Output energy(milli joule) | Nickel II Cinnamaldehyde sulfate |
|----------------------|----------------------------|----------------------------------|
| 0.50                 | 7.5                        | 13.5                             |

4. Conclusion

As per using the slow evaporation solution method (SEST) technique for growing a single crystal of NCS. The Single XRD research indicated that the space group of NCS crystal is P, and it therefore belongs to a tetragonal crystal system. To evaluate the crystalline purity as well as to determine the cell parameter of a NCS crystal, PXRD was used to identify the sharp and intense peaks in the PXRD precursor suggests a high crystallinity of NCS crystal. FTIR correction for identification of functional groups and vibrational assignments UV-Vis NIR probe to study the material susceptibility showed a decrease in absorption from 400 up to about 1100 nm, where the cut-off wavelength was at approximately 278.60 nm and a band gap that could be considered equal or higher than this energy(3.25 eV).The observed PL emission spectra are plotted for the crystal in blue wavelength region .The NLO efficiency of the synthesized materials is 0.5 times greater than that of KDP. The thermal behavior of the crystalline form obtained was studied by TGA-DTA analysis.

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