



# Structural, Optical and Thermal Characterization of Synthesised Heterocyclic Organic Compound

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## Authors' contributions

*This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.*

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## ABSTRACT

A heterocyclic organic compound of 3-[(2z)-2-methyl-3-(4-nitrophenyl) prop-2-enoyl]-2H-(1-benzopyran-2-one) (MNB) is synthesized using Claisen-Schmidt condensation reaction. Structural characterization and presence of functional groups are carried out using powder X-ray diffractogram and FTIR spectroscopic studies respectively. Thermogravimetric analysis and differential thermal analysis are carried out to determine the melting property and it shows good thermal stability among organic crystal showing result at 166°C. Second Harmonic Generation efficiency study is carried out using Nd: YAG laser, this revealed that titled compound is good for NLO characterization and applicable in optoelectronics. PL wavelength present in visible region conclude that sample shows fluorescence property.

**Keywords:** Heterocyclic organic compound; powder XRD; Photoluminescence; second harmonic generation.

## 1. INTRODUCTION

Organic compounds are heterocyclic in which have a ring structure containing atoms such as

sulphur, oxygen or nitrogen as a part of the ring in addition to carbon. A heterocyclic ring may comprise of three or more atoms which may be aliphatic or aromatic. Heterocyclic compound is

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an aromatic ketone and that forms the central core for variety of important optical compounds. The presence of heterocycles in all kinds of organic compounds of interest mainly in biology, pharmacology, optics, electronics, materials sciences and so on is very well known [1]. Chalcones are known as the key intermediate in the synthesis of various organic compound, in this article compound synthesised in laboratory using Claisen-Schmidt reaction. As the source intensity increase, the oscillating dipoles generates harmonic frequencies. Thus, frequency doubling or SHG and higher order frequency generates [2]. Since time responsibility is fast in the organic materials these are very interesting in nonlinear optical characterization. Also, they are inexpensive, easy to process, shows high UV absorption, exhibit thermal & chemical stability thus organic compound is interesting subject among all. The structural requirement of organic material boost to NLO phenomena. Hence presence of  $\pi$  bonds in organic material confer a high polarizability & rapid charge redistribution, these crystals are good for optoelectronics application [3].

## 2. MATERIALS AND METHODS

The titled compound was synthesised by Claisen-Schmidt condensation reaction, A mixture of 3 acetyl coumarin (1eq) and Nitro benzaldehyde (1eq) and a few drop of NaOH were added keep stirring by using magnetic stirrer for 48 hours at room temperature with ethanol as a solvent, product was tested by TLC for purity later it was mixed with cold water to form precipitation, finally it was filtered required powder remained in filter paper [4,5,]. A nitro group is replaced the hydrogen from 3 nitro benzaldehyde by releasing water molecule, now the product is heterocyclic in nature confirms this by FTIR characterization.

## 3. RESULT AND DISCUSSION

### 3.1 Powder XRD Analysis

Synthesised organic crystal is characterised by powder XRD, this was carried by Shimadzu XRD-700 diffractometer with Copper  $K_{\alpha}$  ( $\lambda = 1.5405 \text{ \AA}$ ) radiation using a tube voltage and current of 40 kV and 30 mA respectively. Sample was scanned over the range of  $0^{\circ}$  -  $90^{\circ}$ , over the rate of  $5^{\circ}/\text{min}$  [6]. XRD of newly synthesised sample shows peak at  $9.7, 16.4, 22.5$  degree since broad peak observed between  $15$  to  $35$  degrees. Few broad and few narrow peaks observed in graph

can conclude that synthesised compound shows semi crystalline nature. Crystallite size is calculated using the Scherrer equation  $\tau = k\lambda/(\text{FWHM}) \cos\theta$ . ( $k = 0.9$  broadening constant) powder XRD study of titled compound have  $4.99$  nm average crystallite size.

### 3.2 Ftir Spectra Study

The synthesised compound's bonds were studied using FTIR. This was carried out using instrument SHIMADZU FT-IR – 8400 spectrophotometer and analysis was done from  $4500 \text{ cm}^{-1}$  to  $500 \text{ cm}^{-1}$ . If we start examining from the bottom right, the peaks which are having wave number ranging from  $735 \text{ cm}^{-1}$ - $808 \text{ cm}^{-1}$  are representing benzene ring and shows strong intensity. Peaks with wave number ranging from  $1346 \text{ cm}^{-1}$ - $1523 \text{ cm}^{-1}$  represents the availability of nitro(C-N) compounds as functional groups, they are at lower wavenumbers than usual because the nitro group is conjugated with the benzene ring. Peaks with wave number  $1740 \text{ cm}^{-1}$  –  $1710 \text{ cm}^{-1}$  represents about the presence of Ketone group (C=O stretch) gives two strong intensity bands. And wave number ranges from  $2935$ - $3068 \text{ cm}^{-1}$  represents presence of N-O stretch [7,8] Thus bending bonds occurs at lower frequency and stretching bonds presents at higher frequency concluded that product contains both aldehyde and ketone.

### 3.3 UV-Vis Absorption Study

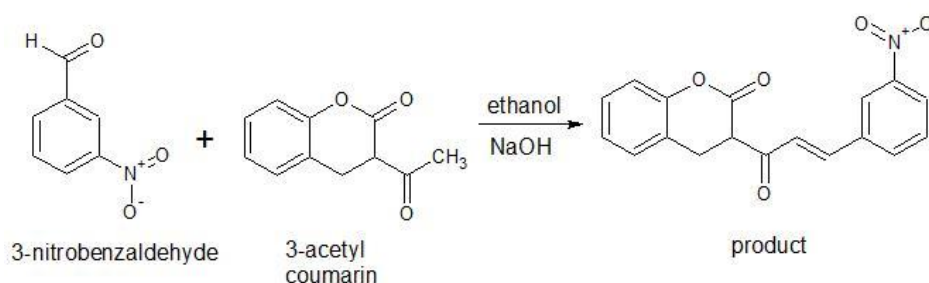
UV- Vis absorption study done for newly synthesised organic crystal and graph is plotted between wavelength vs absorbance is given in Fig (4). UV-VIS absorption spectra were recorded using Shimadzu UV- 1800 spectrometer. Absorption spectra carried over the range of  $200$ - $800 \text{ nm}$  using ethanol as solvent for MNB crystal [9]. From the absorption spectra it is noted that peak found at  $269.2 \text{ nm}$  this is lambda maximum. Hence we calculated optical absorption energy using the formula  $E=hu$ , it is  $4.67 \text{ eV}$  [10]. The horizontal line from  $400 \text{ nm}$  to  $800 \text{ nm}$  reveals about titled compound is good transmittance in visible region this confirms compound shows good optical property [11]. Crystal can be used as potential candidate for optical electronic device fabrication [12].

### 3.4 Thermal Alalysis of MNB

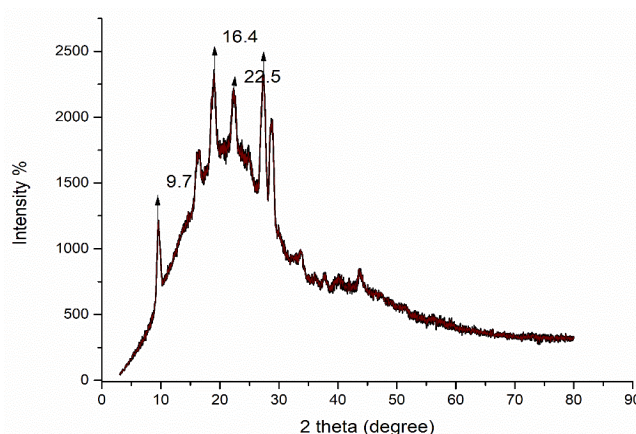
Using SDT Q- 100 instrument the thermal analysis carried out for MNB sample in the

temperature range from ambient temperature to 700 C and response curve of sample shown in Fig. 5. and Fig. 6. In TG analysis the known gram of compound is heated. Fig. 5 illustrates weight loss of 25% shows at 150 C and Fig. 6 illustrates that large endothermic peak observe at 166 , Hence up to 150 C material is stable , above this material losses its weight drastically 75% and

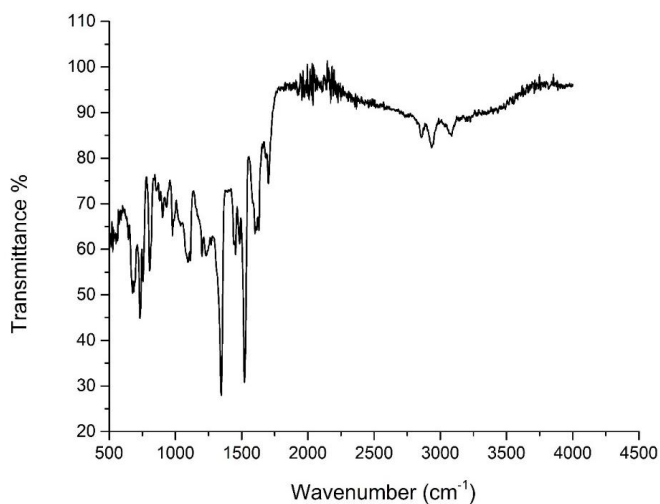
compound decomposes ,phase change from liquid to air. The DTA curve shows major endothermic peak which is melting point of sample noted at 166 C phase change occurs from solid to liquid. This sample shows exothermic peak 269 C, after that compound decomposes and many secondary bonds breaks [9,10,13] .



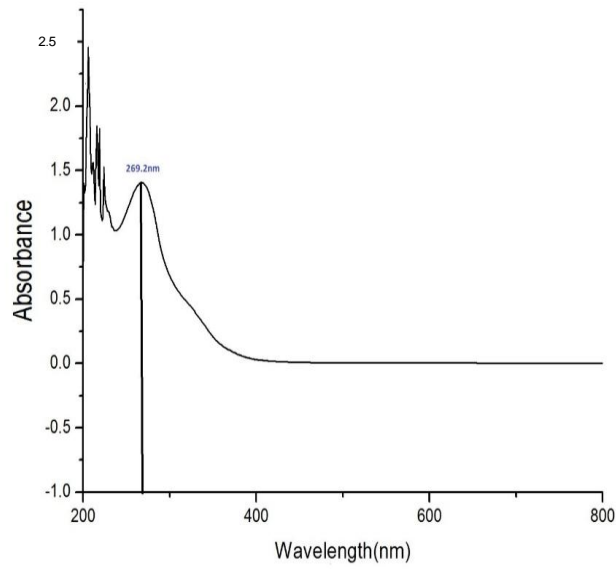
**Fig. 1. Schematic representation of material synthesis**



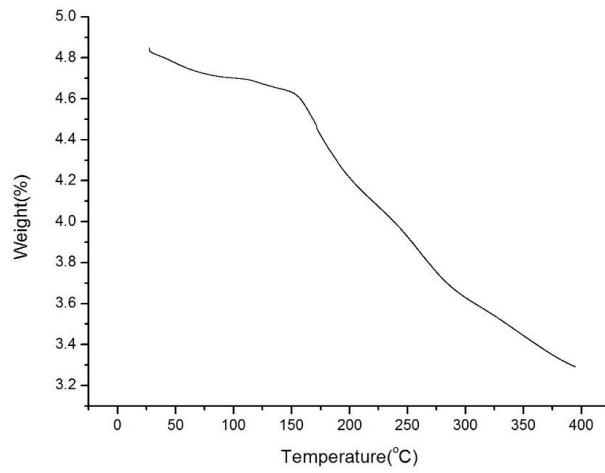
**Fig. 2. Powder XRD of MNB crystal**



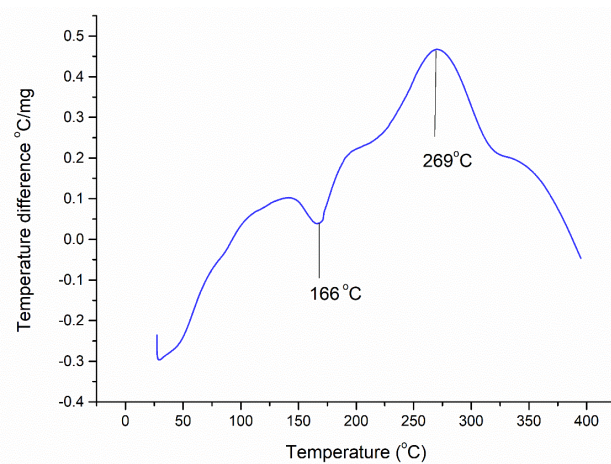
**Fig. 3. FTIR of MNB crystal**



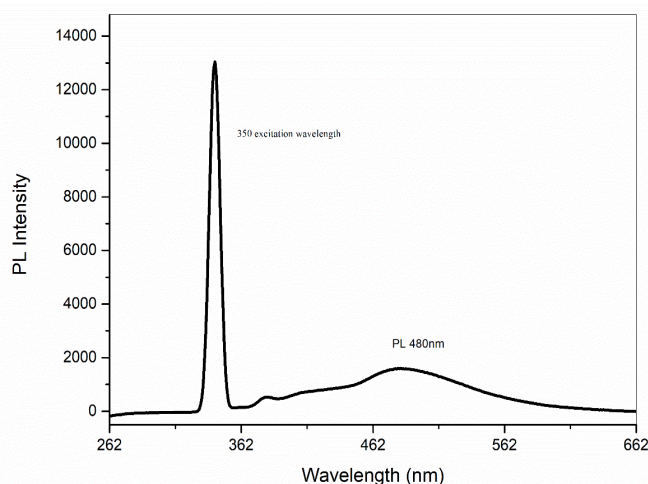
**Fig. 4. UV- Vis absorbance spectra of MNB dissolved in ethanol solvent**



**Fig. 5. TG curve of MNB crystal**



**Fig. 6. DTA of MNB crystal**



**Fig. 7. Photoluminescence of MNB dissolved in ethanol solvent**

### 3.5 Second Harmonic Generation Efficiency

Second harmonic generation (SHG) efficiency was determined by a powder technique developed by Kurtz & Perry. Powdered sample inserted in a closed microcapillary tube later exposed to laser light with input beam 2.5mJ/P. The SHG output is finally detected by photomultiplier tube & displayed on oscilloscope [14]. The emission green light confirms the generation of second harmonics. High molecular dipole moment generally forces the molecules to pack Centro symmetrically in sample but SHG requires non-centro symmetry property in crystal, generally organic crystal's molecules connected through weak intermolecular hydrogen bond and such molecules usually stabilize the non-centrosymmetric crystal packing [8,3]. Because of non-centro-symmetric nature the titled compound shows good SHG efficiency. The output power was measured for the titled sample be 0.2mV. For maximum SHG efficiency crystal should possess phase matching properties that is the propagation speed of fundamental and harmonic wave should be identical in crystal.

### 3.6 Photoluminescence

Using "Labsolution RF-600 series" software an emission spectrum of titled compound was studied from 200nm – 800nm, data interval 1nm, scan speed 2000nm/min. An excitation energy given at 350 nm, thus secondary excitation shown at 650nm in Fig.7. The titled compound shown PL peak at 480nm this is emission wavelength. Thus concluded that blue mixed

green photoluminescence emission observed in MNB crystal [2]. Since PL wavelength present in visible region conclude that sample shows fluorescence property.

## 4. CONCLUSION

The primary objective of paper is synthesis of good quality MNB crystal and it is achieved by Claisen-Schmidt condensation method. Functional group present in the synthesised compound were studied using FTIR and crystallite size and nature of sample is studied by powder XRD, crystallite size is calculated using the Sherrer formula  $\tau = k\lambda/(\text{FWHM})\cos\theta$ . Thermal stability of crystal is found to be 150°C is confirmed by TG study. Melting point is found at 166°C is confirmed by DTA graph shown in Fig 6. UV-Vis spectroscopy study reveals that titled compound have very good transmittance in visible region and good absorption at UV region calculated the optical energy using relation  $E=hc/\lambda_{\text{max}}$ . This confirms that sample shows good NLO property. SHG study reveals that the crystal has a good NLO property. Since PL wavelength present in visible region conclude that sample shows fluorescence property.

## DISCLAIMER

The products used for this research are commonly and predominantly use products in our area of research and country. There is absolutely no conflict of interest between the authors and producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. Also, the research was not funded by

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### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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