Original Research Article

Synthesised Heterocyclic Organic Compound and XRD, Thermal, Optical Characterization Studies on it. .

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-Schmidth condensation method. 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 its thermal stability. Second Harmonic Generation efficiency study is carried out using Nd : Yag laser, this revealed that titled compound is good for NLO characterization.

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 sulfur, 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 a 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. intensity increase, the oscillating dipoles generates harmonic frequencies. Thus, frequency doubling or SHG and higher order frequency generates. [3]. 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 πbonds in organic material confer a high polarizability & rapid charge redistribution [11].

2.MATERIALS AND METHODS

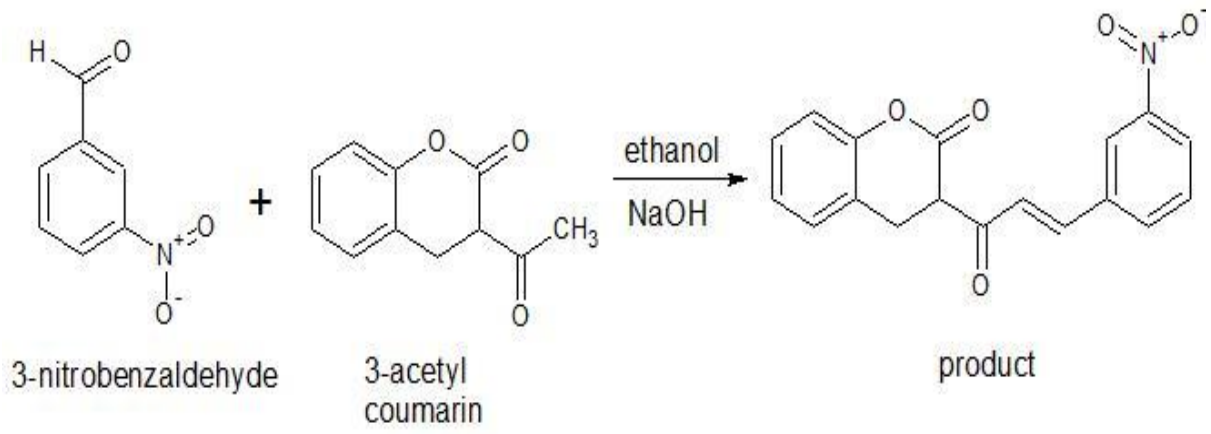

Fig.1 Schematic representation of material synthesis.

The titled compound was synthesised by Claisen-Schmidt condensation method , A mixture of 3 acetyl coumarin (1eq) and Nitro benzaldehyde (1eq) and a few drop of NaOH were added keep stirring by using 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[2]. 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.

2 theta (diffraction angle)	λ(nm) (wavelength	"d"	FWHM	Crystallite size(A^0)
20.6223	15.406	4.30348	0.205	0.783103098
22.4685		3.95387	1.6523	0.720963506
27.4041		3.25194	0.1271	0.596772103
29.7921		2.99649	0.7465	0.54893755
41.6221		2.16809	0.149	0.395008923
47.8098		1.90093	1.7182	0.363511134
56.598		1.62485	0.11	
73.5203		1.28712	0.1305	0.318831832
				0.269752273
				AVG= 0.499610052

Table – 1, Crystallite size calculation using Sherr formula.

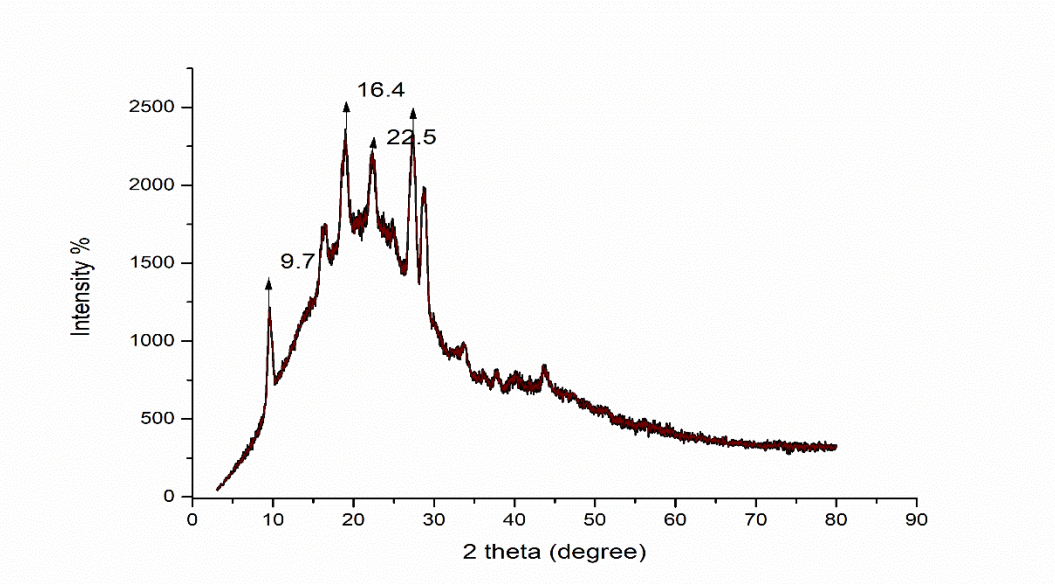

Fig-2 Powder XRD of MNB crystal

Synthesised organic crystal is characterised by powder XRD, this was carried by Shimandzu XRD-700 diffractometer with CuKα (λ = 1.5405 Å) radiation using a tube voltage and current of 40 kV and 30 mA respectively. Sample was scanned over the range of 0^0 -90^o, over the rate of 5^o/min[4]. XRD of newly synthesised sample shows peak at 9.7,16.4,22.5 degree since broad peak observed between 15 to 35 degree. Since broad and few narrow peak observed in graph can conclude that synthesised compound shows semi crystalline nature . Data are given in table 1. Crystallite size is calculated using the Sherr formula $\tau = k\lambda/(FWHM)\cos\theta$. (k = 0.9 broadening constant) powder XRD study of titled compound indicates the 4.99 nm average crystallite size.

3.2 FTIR SPECTRA STUDY

The analysis of the compound using FTIR was carried out using instrument SHIMADZU FT-IR – 8400 spectrophotometer and analysis was done from 4500 cm^{-1} to 500 cm^{-1} . If we start examining from the bottom right, the peaks which are having wave number ranging from 735 cm^{-1}-808 cm^{-1} are representing Benzene ring and shows strong intensity. Peaks with wave number ranging from 1346 cm^{-1}-1523 cm^{-1} represents the availability of

nitro 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 1199 cm^{-1} - 1089cm^{-1} tells about the presence of Ketone group(C=O) gives two strong intensity bands. And wave number ranges from 2935-3068 cm^{-1} represents presence of O-H group [5,7].

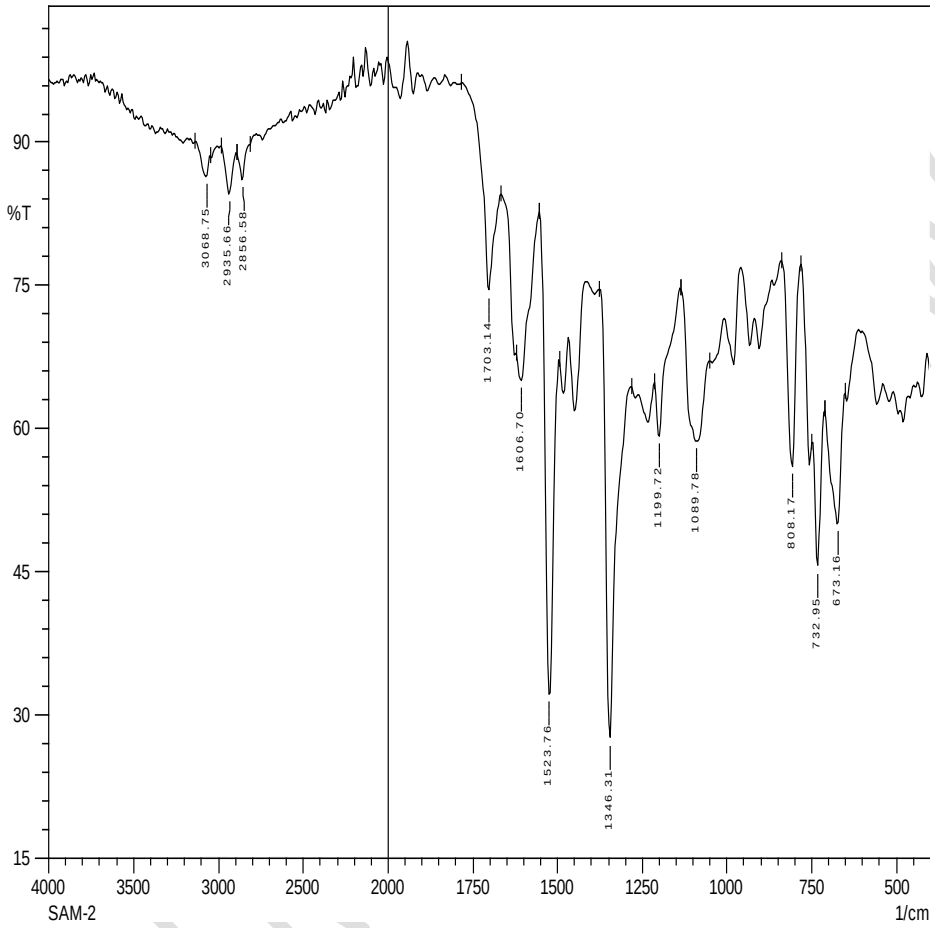

Fig. 3 FTIR of MNB

3.3 UV-ViS ABSORPTION STUDY

UV- ViS absorption study done for newly synthesised organic crystal mentioned above and graph is plotted between wavelength vs absorbance is given in fig(4) . UV-ViS absorption spectra was recorded using Shimadzu UV- 1800 spectrometer . Absorption spectra carried over the range of 200-800 nm [6]. From the absorption curve it is noted that peak found at 269nm this is lambda maximum. Hence we calculated optical absorption energy using the formula $E=h\upsilon$, it is 4.67eV.[8] The horizontal line from 400nm to 800nm tells about titled compound is good transmittance in visible region [12].

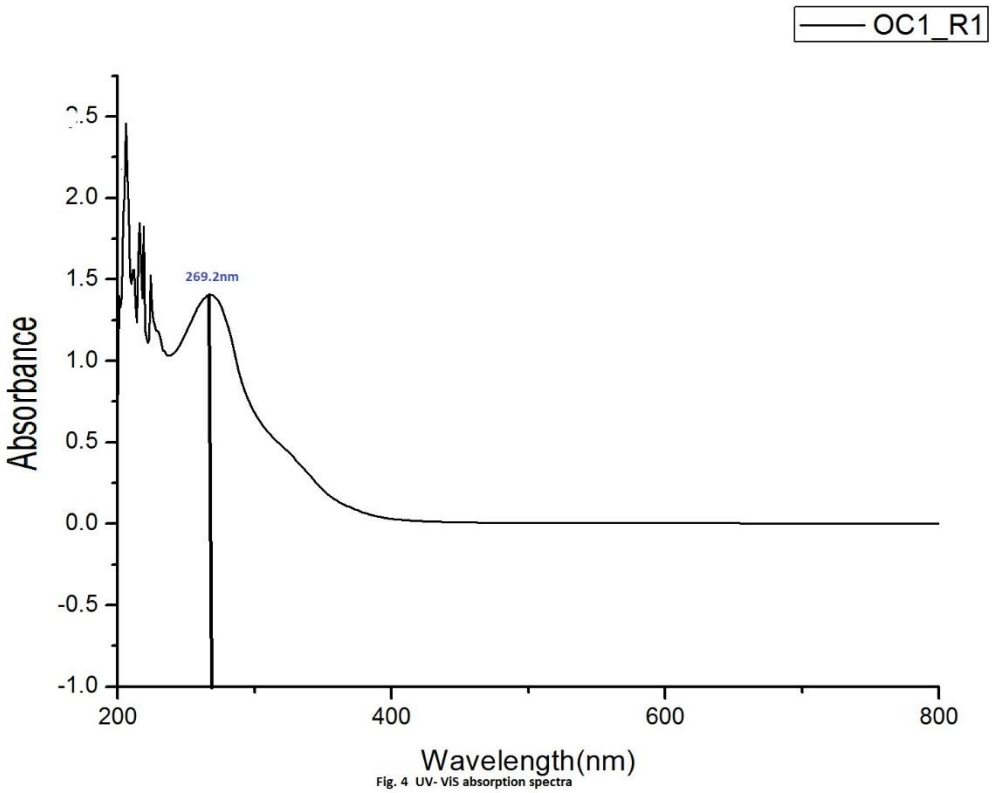

Fig. 4 UV- ViS absorption spectra

Fig.4 UV- ViS absorbance spectra

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^{o}C and response curve of sample shown in fig.5. and fig.6. In TG analysis the known gram of compound is heated. Figure 5 illustrates very small weight loss 10% shows at 150^{0}C.Hence material is stable up to 150^{0}C, above this material losses its weight drastically 90%and compound decomposes. The DTA curve shows major endothermic peak which is melting point of sample noted at 166^{0}C . This sample shows exothermic peak 269 ^{o}C after that compound decomposes and many secondary bonds breaks [6,8,9] .

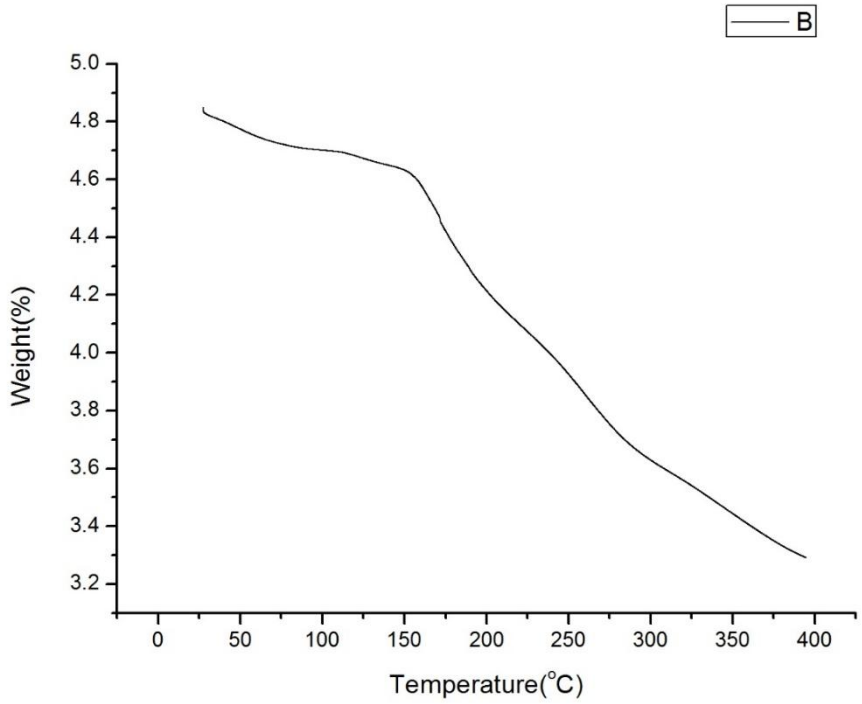

Fig.5 TG curve of MNB

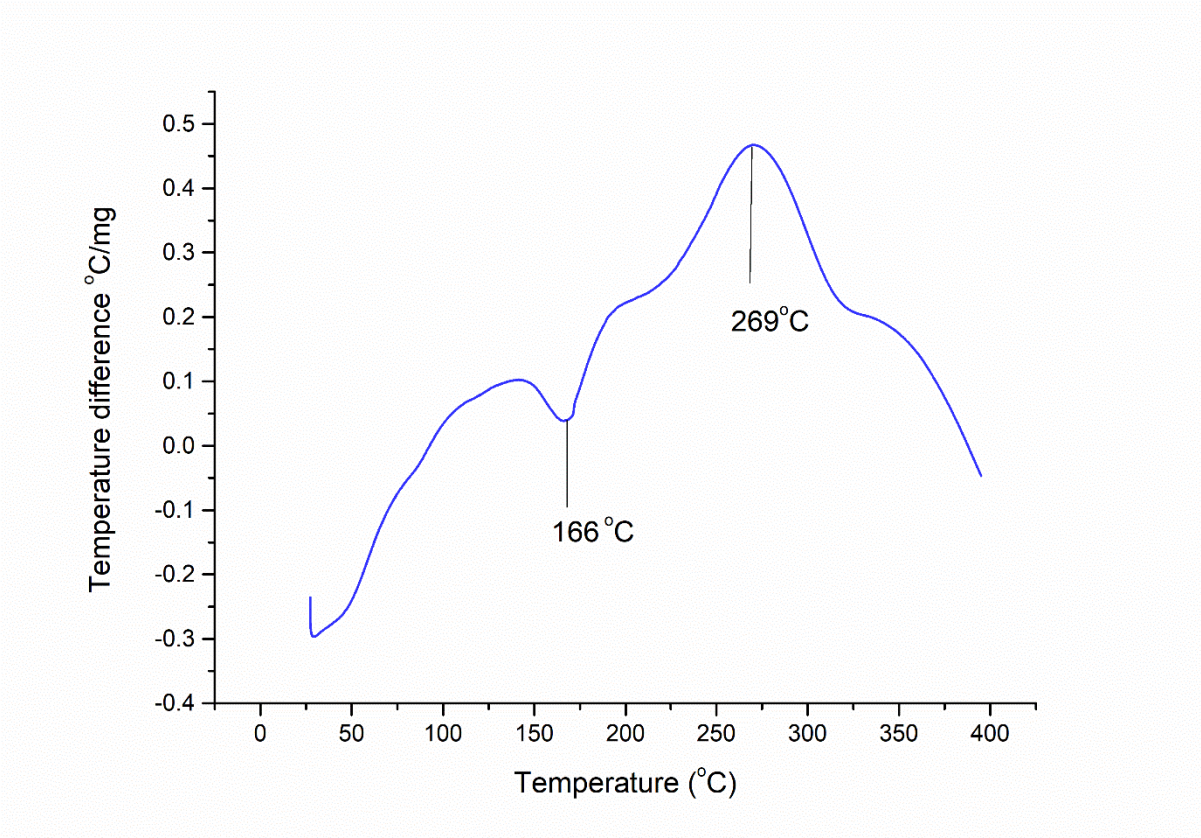

Fig.6 DTA curve of MNB

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 [10]. The emission green light confirms the generation of second harmonics. High molecular diploe moment generally forces the molecules to pack centro symmetrically but SHG requires non centro symmetry , generally organic crystal's molecules connected through weak intermolecular hydrogen bond and such molecules usually stabilize the non-centrosymmetric crystal packing .[7][11] 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 i.e the propagation speed of fundamental and harmonic wave should be identical in crystal .

3.6 PHOTOLUMINESCENCE

Using "Labsolution RF-600 series "software an emission spectra of titled sample 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[3].

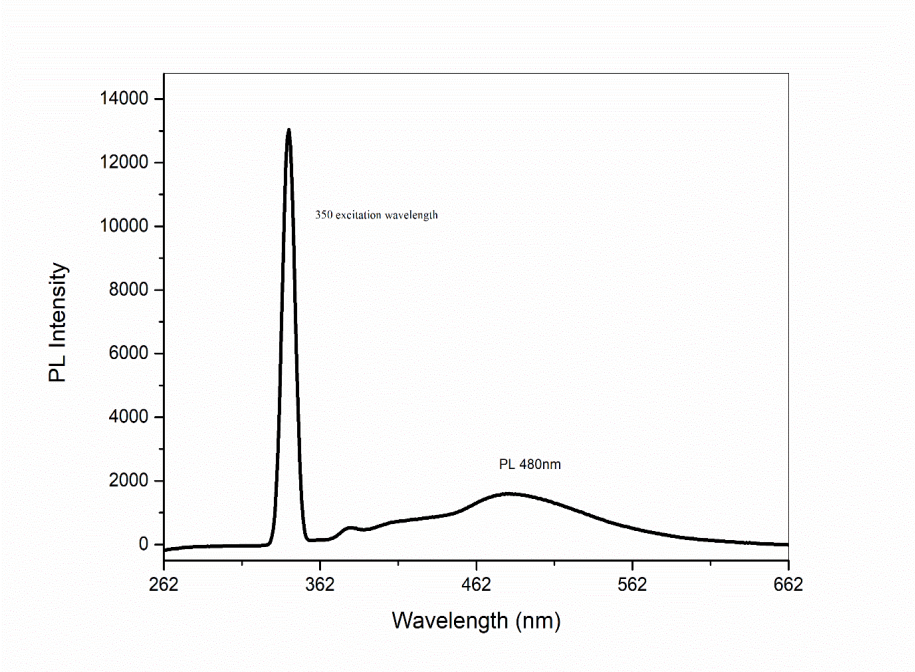

Fig.7 Photoluminescence curve

4. CONCLUSION

The primary objective of synthesis of good quality MNB crystal it is achieved by Claisen-Schmidt condensation method. Functional group present in the synthesised compound were studied using FTIR. MNB is semi crystallite in nature its crystallite size is calculated using the Sherr formula $\tau = k\lambda/(FWHM)\cos\theta$.. Thermal stability of crystal is found to be $160^{O}C$. 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 $E=hc/\lambda_{max}$. SHG study reveals that the crystal has a good NLO properties. Photoluminescence study reveals that sample exhibits blue mixed green emission.

COMPETING INTERESTS DISCLAIMER:

Authors have declared that no competing interests exist. 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 the producing company rather it was funded by personal efforts of the authors

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