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Synthesis and Characterization of New Heterocyclic Based Organic Crystal

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Abstract

Synthesis of 3-[3-(2,3-dichlrophenyl)prop-2-enyl]-2H-chromen-2-one is a heterocyclic organic crystal using Claisen-Schmidth condensation reaction. Functional group present in the sample was investigated by their vibrational modes using FTIR spectrometer. Thermal stability of crystal was investigated by thermogravimetric analysis. UV absorption of crystal was determined by Shimadzu UV- 1800 spectrometer wavelength range from 400 to 4000cm-1.

Keywords: FTIR• UV-ViS • XRD

Introduction

Heterocyclic organic compounds are one of the most exciting scientific investigations to understand the basic units of matter and their properties. Heterocyclic compounds are organic compounds which have a ring structure containing atoms such as sulfur, oxygen or nitrogen as a part of the ring in addition to carbon. Chalcones are synthesized by condensing ketones with aromatic aldehydes in the presence of suitable bases. They are very useful intermediates for the synthesis of five, six and seven-membered heterocyclic compounds. A heterocyclic ring may comprise of three or more atoms which may be aliphatic or aromatic. Different characterization techniques give rise to different properties of the crystal [1,2].

Crystal Synthesis

The titled compound was synthesised by Claisen-Schmidt condensation method , A mixture of 3 acetyl coumarin (1eq) and dichlorobenzaldehyde (1eq) and a few drop of NaOH in the were kept for stirring by using stirrer 48 hours at room temperature with ethanol as a solvent , product was tested by TLC for purity later it was mixed with ice cold water by slowly stirring the mixture , finally it was filtered required powder remained in filter paper collected as sample. Purified it by recrystallization.

Characterization

Ftir spectra

The analysis of the compound using FTIR was carried out. A FTIR instrument with 400-4500 cm-1 SHIMADZU FT-IR–8400 spectrophotometer was used . If we start examining from the bottom right, the peaks which are having wave number at 747.00 cm-1 is confirmed the formation of title compound this transmittance band is for C-Cl bending [3] (Table 1)(Figure 1, Figure 2).

Wavenumber (cm-1)	Tentative assignment
405	Benzene ring
747, 715	C-Cl bending ,ortho distributed
1484	C-C stretching
1610	C-C stretching
1738	Aromatic ring C=C stretch
2004	CH stretch indicates acids (weak bond)
2321	Symmetric and asymmetric stretching band -O-CH(weak bond)

Table 1. Wavenumber and tentative assignment.

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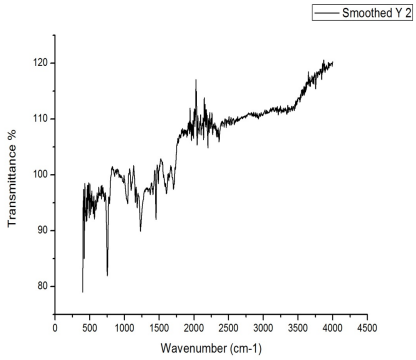

Figure 1. Transmittance %.

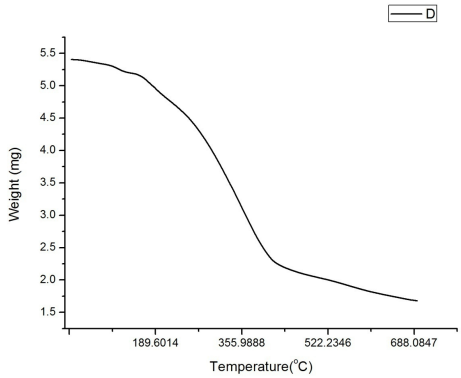

Figure 2. Thermal analysis.

Thermal analysis

Thermogravimetric analysis of sample show maximum rate of decomposition at 3500C. Corresponding to maximum rate of weight loss. There is less weight loss from 3500C to 6800 C. This indicates that crystal is stable from 3500C-6800 C temperature it helps in NLO application (Figure 3).

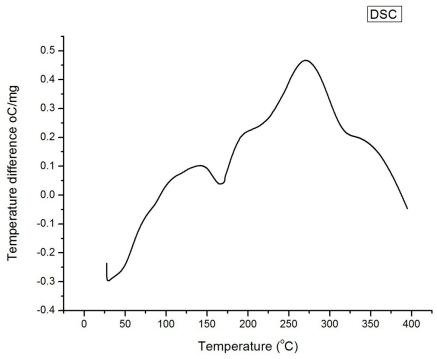

Figure 3. The differential scanning calorimetry.

Differential thermo gravimetric (DTG) analysis for titled compound in powder form placed in a platinum crucible were carried out in an nitrogen atmosphere using a SDT Q 600 TA instrument , heating rate of 10 oC /min in the temperature range 0-400oC. The differential scanning calorimetry graph is shown in figure 3, it shows endothermic transition at 178 0C indicating the melting point of the crystal [3](Figure 4).

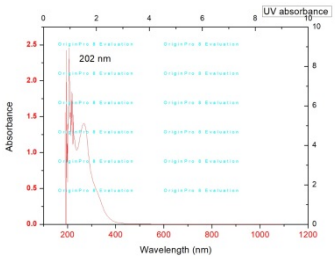

Figure 4. The UV–Vis absorption spectrum.

UV absorption

The UV–Vis absorption spectrum is shown in figure 4, generally, chalcones derivative absorb light in the UV region and transmit in the remaining region. The crystal has strong absorption band in the UV region due to n-p* and p-p* transition and is attributable to the excitation in the aromatic ring and presence of C=O group. The absence of the absorption in the visible region is a key factor for this compound to be exploited for NLO application at room temperature. It is seen in fig that maximum absorption shown in 202nm [4-6]

Conclusion

The title compound was synthesized using Clasien-Schemid condensation reaction it was conferred by FTIR spectrometer by knowing functional group and double bond is also conferred by fig 1. Thermal studies revealed that crystal can remain in stable state from 350- 650oC. DSC study confirms melting point is 350oC, it shown in fig 3. The good transparency in visible region and high UV absorption indicates that promising candidate for NLO application.

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