

Synthesis, Thermal and Crystallographic analysis of 1,1'-diylbis(2-methyl-4-phenylene)bis(2-nitrobenzoate)cyclohexane

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ABSTRACT

The crystal structure of 1,1'-diylbis(2-methyl-4-phenylene)bis(2-nitrobenzoate) cyclohexane (NBM-2) has been determined by X-ray crystallographic technique. Single crystals were grown by slow evaporation technique. NBM-2 crystallized in the triclinic crystal lattice having space group P-1. Both the phenyl rings are perpendicular to the cyclohexane ring having chair conformation. NBM-2 showed both inter-and intramolecular interactions. The structure of NBM-2 has also been supported by FT-IR, ¹HNMR, ¹³C NMR, MS techniques. The thermal behaviour of NBM-2 were studied by TGA and DSC techniques at heating rate of 10°C min⁻¹ in nitrogen atmosphere. Thermal stability and kinetic parameters were determined according to Anderson-Freeman method and discussed

Keywords: Bisphenols; Crystal structure; X-ray diffraction analysis; Thermal analysis

1. INTRODUCTION

Bisphenols are used for a class of chemical compounds bearing two hydroxyphenyl moieties connected via a carbon or sulfur bridge [1]. For many decades, they are widely used in the manufacturing of epoxy resins and polycarbonates to different everyday objects like water bottles, coatings or electronic devices [2]. Bisphenols have a wide spectrum of biological activity including fungicides, antibacterial, antioxidant, antiviral activity, antagonistic activity for an estrogen receptor and act as a selective estrogen receptor modulator [3-7]. 1,1'-Bis(4-hydroxyphenyl)cyclohexane has been used for the isolation of methylhydrazine from its aqueous solution [8] and proved to be useful for efficient separation of isomers, which could not be separated by the fractional distillation technique because of their narrow boiling points [9,10]. Bisphenols leading to ladder-like structures are structurally comparable to metalloorganic ladder frames [11]. Bisphenols can have more than one independent molecule or formula unit in a unit cell giving rise to a higher Z' value [12] and the polymorphism occurs due to a different Z' value [13-15]. By adding electron donating methyl groups to the aromatic rings of bisphenols they become more lipophilic and sterical demanding. Their respective solubility, bioavailability and biological activity are directly connected to the molecular structure and intermolecular interactions of the particular derivative in the solid state and in solution [16]. The knowledge of methylated bisphenols and their analogues is rather limited [17]. To the best of our knowledge no work has been reported

on synthesis and specifics on the structural study of 1,1'-diylbis(2-methyl-4-phenylene)bis(2-nitrobenzoate)cyclohexane which prompted us to carry out synthesis and molecular study of this compound. The synthesis of these compound was followed by measurement of their analytical data and subsequent spectroscopic analyses using ^1H NMR, ^{13}C NMR spectra, mass spectra and elemental analyses techniques to confirm the presence of the supposed ring systems, presence of nitro and methyl substituents as well as the signals for the existence of various protons. A single crystal X-ray diffraction analysis was carried out for the compound in order to establish the crystal as well as molecular structures and to understand possible intermolecular interactions.

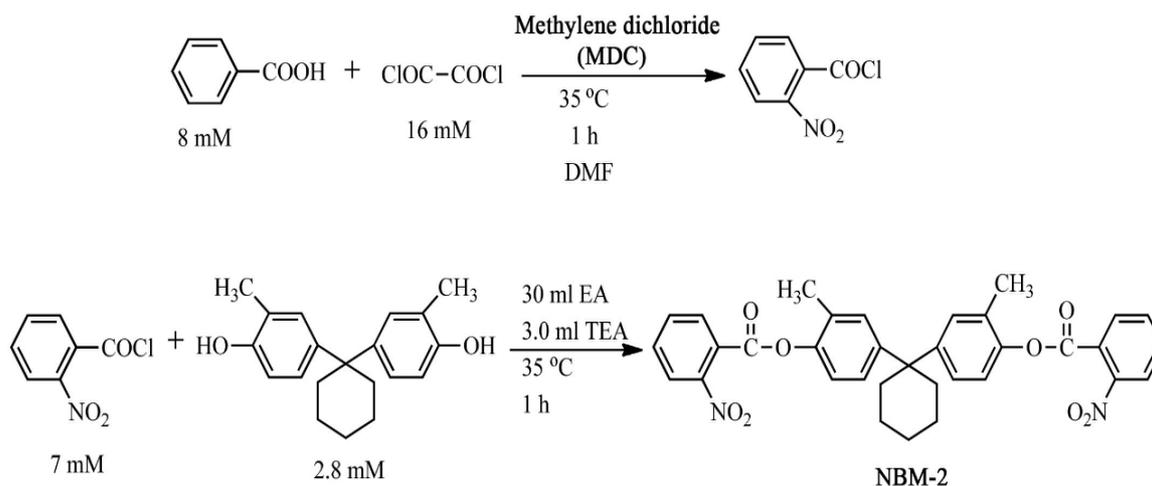
2. EXPERIMENTAL

2.1. Materials

The solvents and chemicals used in the present investigation were of laboratory grade and were further purified according to literature methods [18]. 1,1'-bis(3-methyl-4-hydroxyphenyl)cyclohexane (MEBC) was synthesized and crystallized four times from ethyl acetate according to our previous publication [19]. 2-Nitrobenzoyl chloride was synthesized by reacting 2-nitrobenzoic acid and oxalylchloride in methylene dichloride at room temperature [20].

2.2. Measurements

Melting point was determined in an open capillary and are uncorrected. Fourier transform infrared spectrum (FT-IR) was recorded from KBr pellet over the range 4000-400 cm^{-1} on a Shimadzu FT-IR-8400. Nuclear Magnetic resonance (^1H NMR) spectrum was scanned on a Bruker Avance III 400 MHz NMR spectrometer using CDCl_3 as a solvent and TMS as an internal standard. Carbon-13 nuclear magnetic resonance (^{13}C NMR) spectrum was recorded on a Bruker Avance Spectrospin 400MHz spectrometer using CDCl_3 as a solvent and TMS as an internal standard. Mass spectrum (MS) was scanned on a Shimadzu GC-MS QP 2010 spectrometer by using EI (0.7kV) detector. The ion source temperature was 220 $^\circ\text{C}$ and interface temperature was 240 $^\circ\text{C}$. Thermogravimetric analysis (TGA) was carried out on a Shimadzu DTG-60H at the heating rate of 10 $^\circ\text{C min}^{-1}$ in a nitrogen atmosphere of flow rate 100 mL min^{-1} . Differential scanning calorimetric (DSC) measurement was carried out on a Shimadzu DSC 60 at the heating rate of 10 $^\circ\text{C min}^{-1}$ in a nitrogen atmosphere of flow rate 100 mL min^{-1} . About 2.390 mg of the sample was accurately weighed, enclosed in an aluminium DSC crucible using a crimper. The compound was prepared following the procedure given below and as shown in Scheme 1.



Scheme 1. Synthesis of NBM-2.

2.3. Preparation of 1,1'-diylbis(2-methyl-4-phenylene)bis(2-nitrobenzoate)cyclohexane (NBM-2)

A 250 mL two neck round bottomed flask equipped with a mechanical stirrer and a dropping funnel was placed in a thermostat bath. To this flask 2.8 mM MEBC in 10 mL ethylacetate (EA) and 3.0 mL triethylamine (TEA) were placed and dissolved with stirring. To this solution 7.0 mM 2-nitrobenzoyl chloride in 20 mL ethylacetate was added dropwise through a dropping funnel over a period of 5 min. The solution was stirred at 35°C for 1 h. Crude product was isolated, filtered, washed well with hexane and dried in an oven at 50°C and was crystallized four times from ethylacetate.

2.4. Spectral data of 1,1'-diylbis(2-methyl-4-phenylene)bis(2-nitrobenzoate)cyclohexane (NBM-2)

White solid, yield 81%, m.p. 119-121 °C; IR (KBr) ν cm^{-1} : 3095, 2933, 2860, 1754, 1606, 1535, 1498, 1365, 1253, 1120, 1035, 854; ^1H NMR (400 MHz, CDCl_3) δ ppm : 1.28-1.39 (m, 6H, $-\text{CH}_2$), 2.25 (s, 6H, $-\text{CH}_3$), 2.28 (s, 4H, $-\text{CH}_2$), 7.14-7.21 (m, 6H, Ar-H), 7.68-7.78 (m, 4H, Ar-H), 7.90 (d, 2H, Ar-H, $J=1.2$ Hz), 7.92 (d, 2H, Ar-H, $J=0.8$ Hz); ^{13}C NMR (400 MHz, DMSO) δ ppm: 16.3(C11), 22.8(C2), 26.3(C1), 37.2(C3), 45.6(C4), 121.03(C13), 124.20(C9,C17), 125.94(C10), 127.29(C14), 129.59(C6), 130.07(C7), 132.22(C15), 133.20(C16), 146.59(C18), 146.76(C5), 148.18(C8), 163.83(C12); MS (m/z): 594[M^+]

3. X-RAY DIFFRACTION ANALYSIS

The single crystal X-ray diffraction data of compound was collected at 293 K on a Rigaku SCX mini diffractometer with graphite monochromated Mo- $\text{K}\alpha$ radiation ($\lambda=0.71075$ Å) as the excitation source. Data collection and reduction were performed using Crystal Clear software [21]. The reflections of single crystal was measured in a 2θ range of 2.53-25.00°. Total 6510 independent reflections were measured. REQAB multi-scan empirical absorption

corrections were adopted for data processing. The crystal structure was solved by direct method and the non-hydrogen atoms were subjected to anisotropic refinement by full-matrix least-squares on F^2 using SHELXL-97 [22]. All H atoms were placed in their calculated positions and included in the refinement using the riding model. The final least square cycle of refinement gave $R_1 = 0.1110$ and $wR_2 = 0.3553$. The crystallographic data and structural refinements are listed in Table 1 Selected bond lengths and bond angles of NBM-2 are listed in Table 2. The selected torsion angles, intra- and intermolecular interactions are shown in Table 3. The crystal structure of NBM-2 is shown in Fig. 1. Crystallographic data of NBM-2 are deposited to the Cambridge Crystallographic Data Centre (CCDC No. 948669)

Table 1. Crystallographic data and structural refinement of NBM-2.

Deposition number	CCDC 948669
Empirical Formula	$C_{34}H_{32}N_2O_8$
Formula weight	594.62
Crystal color, Habit	Colorless, Prism
Crystal system	Triclinic
Crystal size (mm^3)	0.60×0.56×0.45
Temperature (K)	293
Wavelength (λ)	0.71075 (Å)
Space group	P-1
Unit cell dimensions	$a = 9.981(2)$ Å, $\alpha = 109.597(8)^\circ$ $b = 12.253(3)$ Å, $\beta = 104.769(7)^\circ$ $c = 12.943(3)$ Å, $\gamma = 90.973(6)^\circ$
Volume (Å^3)	1432.8(6)
Z value	2
Calculated density (g/cm^3)	1.378
Absorption coefficient (cm^{-1})	0.988
$F(000)$	624.00
θ range for data collection ($^\circ$)	0.95 – 27.4
Index ranges	$-12 \leq h \leq 12$, $-15 \leq k \leq 15$, $-16 \leq l \leq 16$
Reflections collected/unique	14642/6510 [$R_{int} = 0.0201$]
Refinement method	Full-matrix least-squares on F^2
Goodness-of-fit on F^2	1.486
Residuals: $RI^a(I > 2.00 \sigma(I))$	0.0950
Residuals: R (All reflections)	0.1110
Residuals: wR_2^a (All reflections)	0.3553
Largest diff. peak and hole ($e \text{ Å}^{-3}$)	0.69 and -0.33
S^a	1.486
n_p	397

Table 2. Selected torsion angles ($^{\circ}$) of NBM-2.

Atoms	Angles ($^{\circ}$)	Atoms	Angles ($^{\circ}$)
C9-C23-C27-C33	-53.1(4)	C9-C21-C30-C33	60.3(4)
O2-C18-C19-C36	6.1(4)	O1-C13-C25-C41	-9.7(4)
C20-C11-C14-N10	-7.2(5)	C29-C37-C38-N7	-9.6(5)
C20-O1-C13-C25	106.6(3)	C29-O2-C18-C19	-77.4 ⁰

Table 3. Selected intra- and intermolecular interactions of NBM-2.

Bond	Distances (\AA)	Bond	Distances (\AA)
O1-O15	2.811(5)	O4-O44	2.804(6)
O ₁₇ -O ₁₇ ⁷	2.919(7)	O8-O8 ⁵	2.884(5)

4. RESULTS AND DISCUSSION

4.1. Chemistry

Friedel-Crafts condensation reaction of cyclohexanone and o-cresol in presence of an acid catalyst gave 1,1'-bis(3-methyl-4-hydroxyphenyl)cyclohexane (MEBC). Further condensation of MEBC with 2-nitrobenzoyl chloride in presence of triethylamine and ethylacetate gave final product (NBM-2). These compounds were characterized by FTIR, ¹H NMR, ¹³C NMR, and MS techniques. Good quality single crystals of NBM-2 were prepared by slow evaporation method using ethyl acetate. Crystals grown are stable for a long-time in normal conditions of temperature and humidity. The chemical formula was determined when resolving the crystal structure by X-ray diffraction. In the present work, we report the synthesis and spectroscopic analysis of the NBM-2.

4.2. Crystal structure analysis

Single crystal X-ray diffraction analysis of NBM-2 revealed triclinic crystal lattice with space group P-1 and density (Z) of 2.

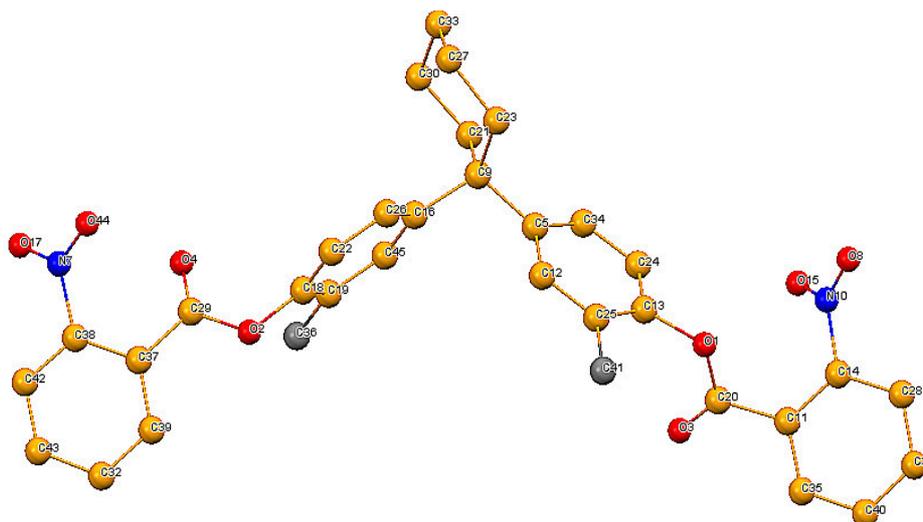


Fig. 3. Crystal structure of NBM-2.

From Fig. 3, it is clear that cyclohexane ring adopted the stable chair conformation because it is completely strain-free. The conformation of the cyclohexane ring is described in terms of torsion angles of $C_9-C_{23}-C_{27}-C_{33} = -53.1(4)$ and $C_9-C_{21}-C_{30}-C_{33}$ [$60.3(4)$], respectively. The presence of the 2-nitro benzoate and methyl group substituents are responsible for quite higher value of dihedral angle. The values of dihedral angle and C-X (X=cyclohexane ring) bond length between the phenyl rings is $108.2(2)^\circ$ and 1.536 \AA are somewhat higher than that of mother compound [106.15° 1.544 \AA] confirming steric effect of methyl substituent between the plane of phenyl rings [23]. The cyclohexane rings of neighbouring molecules closely packed through nonspecific and weak van der Waals interactions because they do not have hydrogen-bonding donor/acceptor groups. The interatomic distances $1.342(4) \text{ \AA}$ for C13-C24, $1.358(6) \text{ \AA}$ for C42-C43 and $1.359(6) \text{ \AA}$ for C31-C40 are nearly same to that of a typical C-C double bonds (1.34 \AA). The bond angles for C24-C13-C25, C32-C43-C42 and C28-C31-C40 are $122.8(3)^\circ$, $120.9(4)^\circ$ and $120.7(4)^\circ$ respectively are nearly same to 120° , which indicated that C13, C31 and C42 adopted sp^2 hybrid orbit to form C=C bonds. The C-N distances [$1.452(4) \text{ \AA}$ for C14-N10, $1.459(5) \text{ \AA}$ for C38-N7] are similar to a typical single bond C-N (1.45 \AA) suggesting that intra-annular π conjugated system is occurred. Due to the existence of a conjugated system, the N-O bond distances [N10-O8, N10-O15, N7-O17 and N7-O44 are of values $1.205(4) \text{ \AA}$, $1.192(4) \text{ \AA}$, $1.188(5) \text{ \AA}$ and $1.208(5) \text{ \AA}$] are shorter than that of a typical bond length of N-O (1.24 \AA). Each methyl groups present on phenyl rings can be oriented in *synperiplanar* (*cis*) or *antiperiplanar* (*trans*) conformation with respect to adjacent C=C of the phenyl rings. The torsion angle O-C=C-C close to 0° corresponds to the *cis* configuration and close to 180° corresponds to *trans* configuration. The observed torsion angles $O_2-C_{18}-C_{19}-C_{36} = 6.1(4)^\circ$ and $O_1-C_{13}-C_{25}-C_{41} = -9.7(4)^\circ$ are close to 0° confirming *cis* arrangement of methyl groups. The conformation of the nitro group with respect to the carbonyl groups is described in terms of the torsion angles $C_{20}-C_{11}-C_{14}-N_{10} = -7.2(5)$ and $C_{29}-C_{37}-C_{38}-N_7 = -9.6(5)$, respectively are close to 0° confirming *cis* conformation. The spatial arrangement of the carbonyl groups with respect to methyl group adopted a *trans/cis* configuration as indicated by torsion angles $C_{20}-O_1-C_{13}-C_{25} = 106.6(3)^\circ$ and $C_{29}-O_2-C_{18}-C_{19} = -77.4^\circ$. This orientation of the carbonyl groups can probably be attributed to the intramolecular interactions (O...O) with $O_4-O_{44} = 2.805(6) \text{ \AA}$ and $O_1-O_{15} = 2.8119(5) \text{ \AA}$. The nitro groups form dimer with neighbouring molecule through intermolecular interactions having bond

distances $O_{17}-O_{17}$ [7] = 2.919(7) Å⁰ and O_8-O_8 [5] = 2.884(5) Å⁰ which are similar to the standard geometry [$O...O$ = 2.868(1) Å⁰]. The structure also exhibits intermolecular C-H...O interactions with adjacent molecules having bond distances 3.205 Å⁰, 3.162 Å⁰ and 3.205 Å⁰ respectively, as illustrated in Fig. 4.

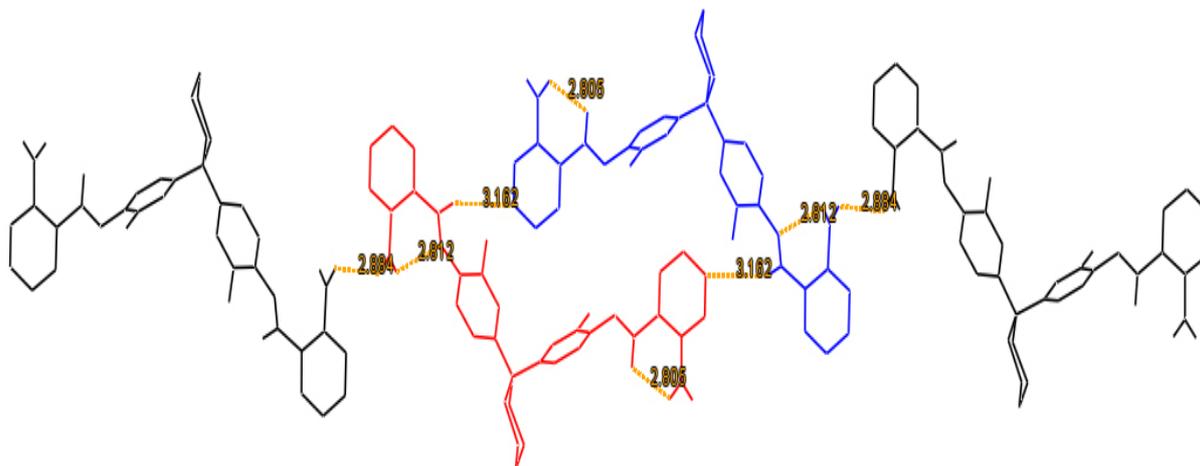


Fig. 4. C-H...O and O...O interactions between the adjacent molecules.

These hydrogen interactions link the molecules into chains and help in stabilizing the crystal structure and give rise to a 3D network as shown in Fig. 5.

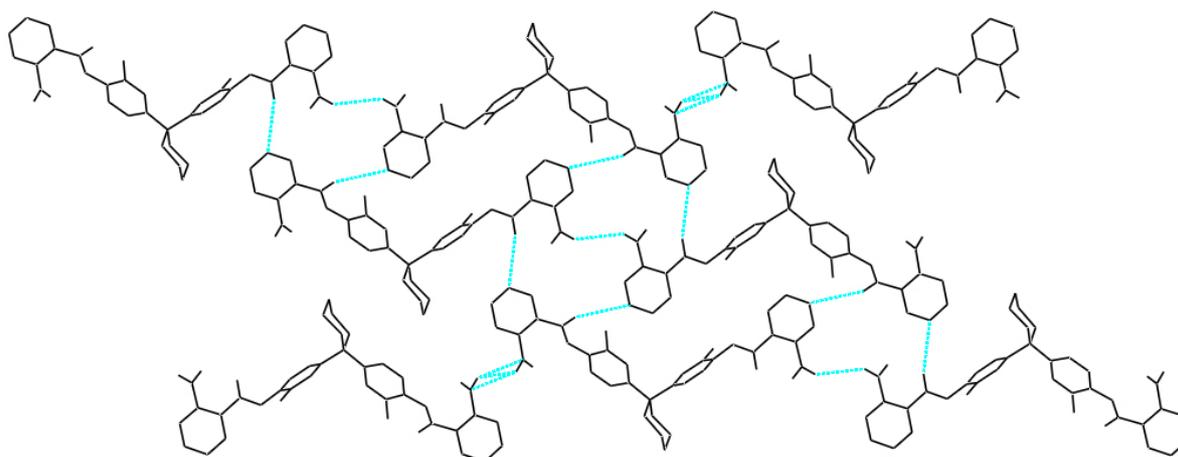


Fig. 5. The 3D architecture of molecules.

4.3. Thermal analysis

DSC thermogram of NBM-2 at 10 °C min⁻¹ heating rate in nitrogen atmosphere is presented in Fig. 4. The sharp endothermic transition at 119.40 °C is due to melting transition. Observed heat of melting and entropy are 30.84 kJmol⁻¹ and 78.56 JK⁻¹mol⁻¹, respectively. TGA thermogram of NBM-2 at 10 °C min⁻¹ heating rate in nitrogen atmosphere is presented in Fig. 5.

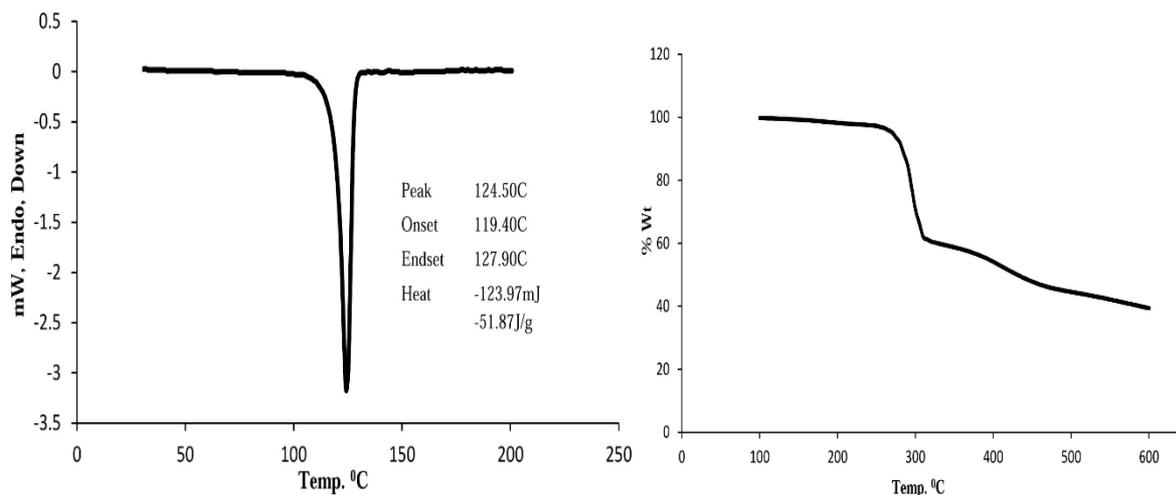


Fig. 6 & 7. DSC & TGA themogram of NBM-2.

NBM-2 is thermally stable up to 270 °C and followed two-step degradation reactions involving 43.9 % weight loss in first step (270-315 °C) and 13.8% weight loss in second step (340-485 °C). A 39.4 % residue was remained at 500 °C. The observed temperature of maximum weight losses are 295° and 414.1 °C, respectively for the first and second step degradation reactions. Associated kinetic parameters such as order of reaction (n), activation energy (E) and frequency factor (A) were determined according to the Anderson-Freeman method [24].

$$\Delta \ln \frac{dw}{dt} = n \Delta \ln W - \left(\frac{E}{RT} \right) \Delta \left(\frac{1}{T} \right) \quad (1)$$

$$A = \frac{E\beta}{RT^2} e^{E/RT} \quad (2)$$

$$\Delta S^* = R \ln \frac{Ah}{kT} \quad (3)$$

Where dw/dt is the rate of decomposition, W is the active mass, β is the heating rate, R is gas constant, h is Planck's constant, T is temperature and k is Boltzmann constant. The least square values of E , A , n , ΔS^* and regression coefficients R^2 are 339.9 kJmol⁻¹, 3.71×10^{29} s⁻¹, 0.8, 315.8 JK⁻¹ mol⁻¹, 0.97 and 178.5 kJmol⁻¹, 9.68×10^{12} s⁻¹, 1.0, -2.41 JK⁻¹ mol⁻¹ and 0.98, respectively for first and second steps. A large and positive magnitude of ΔS^* suggested that the transition state is in disorderly state, while negative magnitude of ΔS^* confirmed that the transition state is in orderly state [25,26]. Degradation process is a complex process, and involves a variety of reactions such as cleavage, rearrangement, branching, crosslinking, etc. Ester, methyl and nitro substituents are weak points in the molecule so preferential degradation starts from such points and free radicals form, which may undergo above mentioned reactions and degrade at elevated temperatures. A 39.4% residue above 500°C confirmed formation of highly thermally stable cross-linked product.

5. CONCLUSION

Spectral data supported the expected structure of NBM-2, which crystallized in triclinic crystal lattice having space group P-1 with crystallographic parameters: $a = 9.981(2) \text{ \AA}$, $b = 12.253(3) \text{ \AA}$, $c = 12.943(3) \text{ \AA}$, $\alpha = 109.597(8)$, $\beta = 104.769(7)$, $\gamma = 90.973(6)$. NBM-2 is thermally stable up to about $270 \text{ }^\circ\text{C}$ and followed two step decomposition reactions with apparently first order degradation kinetics.

Supplementary material

CCDC depositions 948669 contains supplementary crystallographic data of NBM-2 for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif by e-mailing datarequest@ccdc.cam.ac.uk, or contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ.

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