Available online www.worldscientificnews.com

World Scientific News

An International Scientific Journal

WSN 216 (2026) 93-107 EISSN 2392-2192

Synthesis, Characterization and Mesomorphic Study of Homologous Series Linking Azo-Ester Bearing tert-Butyl Terminal Group

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https://doi.org/10.65770/LRWY4450

ABSTRACT

A new homologues series of azo-ester containing tert-butyl terminal group has been synthesised and investigated for its mesomorphic properties. The series consists of total eleven homologues (A_1-A_{14}) in which lower homologues (A_1-A_3) are non-mesomorphic in nature due to lack of molecular flexibility. The higher homologues (specifically A_4–A_{14}) demonstrate consistent enantiotropic nematic phases. The liquid crystal phase remains stable at broad range of temperatures. The phase transition temperatures and characteristic textures of homologues were identified via polarised optical microscopy (POM) using heating stage and differential scanning calorimetry (DSC). The observed texture of nematic phase is schlieren or threaded. Thermal, analytical and spectral (IR, ^{1}H NMR, Mass analysis) data confirmed the molecular structure.

Keywords: Mesomorphic, Azo-ester, Nematic, Homologues.

(Received 12 April 2026; Accepted 15 May 2026; Date of Publication 24 June 2026)

1. INTRODUCTION

Liquid crystals have a distinct phase that integrates the mobility of liquids and structural anisotropy of solid-state crystals. Such characteristics are crucial to their utility in tuneable optical systems and high-performance material synthesis. [1-3]. The temperature sensitive nature of thermotropic liquid crystals and mesophase transition makes them optimal applicants for study, particularly regarding their versatile physical properties [4-6]. Recent studies highlight a move toward rational design of novel liquid crystals, where structural modifications are exploited to modulate phase stability, thermal ranges and optoelectronic parameters. [7-9] Considerable focus has been guided towards the azobenzene framework, bent-core mesogens, and functionalised liquid crystalline material. [10-11] A recent investigation highlights the illustration of structural property relationships, including substituent identity, chain length, and various linkage like azo (N=N), Schiff (CH=N), chalcone (CO–CH=CH) and ester (-COO-) to enhance mesomorphic thermal stability and performance. [12-16]

The Mesomorphic behaviour of azobenzene based materials is greatly affected by distinct terminal groups attached to their aromatic centres. [17] The introduction of bulky or sterically demanding moieties is a very effective approach to regulate intermolecular forces. [18-19] Hence, optimising thermotropic stability and regulate the phase transition profile of the resultant liquid crystals. In relation to the tert-butyl group it has attracted attention due to substantial steric bulk and non-linear, branched geometry. [20-22] The integration of this moiety at terminal position can interrupt the dense molecular packing through steric hindrance and promote the nematic phase over the smectic phase, afterwards decreasing in melting point and expansion of mesophase stability over an expanded temperature range. [23]

In the present series A, eleven homologues (A_1-A_{14}) were studied. In which lower homologues (A_1-A_3) exhibit non-liquid crystalline behaviour due to lack of molecular flexibility, whereas A_4-A_{14} shows an enantiotropic nematic phase. [24] There is no evidence of smectic phase because of bulky terminal group which provides steric hindrance and prevent layered molecular packing. Mesomorphic characteristic were evaluate using polarized optical microscopy (POM) and differential scanning calorimetry (DSC). [25-26]

2. EXPERIMENTAL

Material and Method

The following materials were acquired from BLD pharma for synthesis: 4-Hydroxy benzoic acid, 4-tert butyl aniline, Phenol, DCC, DMAP, and potassium hydroxide (KOH). Alkyl bromides (R-Br), were purchased from Spectro. Chem Ltd. Further hydrochloric acid (HCl), methanol, ethanol and dichloromethane (DCM) were purchased from JSK Chem Ltd.

Synthesis of Part: A

4-Hydroxy benzoic acid (1eq) was dissolve in methanol and treated with potassium hydroxide (2 eq), stirred for few minutes, and then add appropriate alkyl halide (R-X, 1.2 eq), reflux the reaction mixture for 16 hours and monitored by TLC. Upon completion of reaction add 10 % KOH solution to the reaction mixture and stirred for few more hours. The reaction mixture then poured into cold water and neutralise it with hydrochloric acid, filter the precipitate of 4-alkoxy benzoic acid. [27]

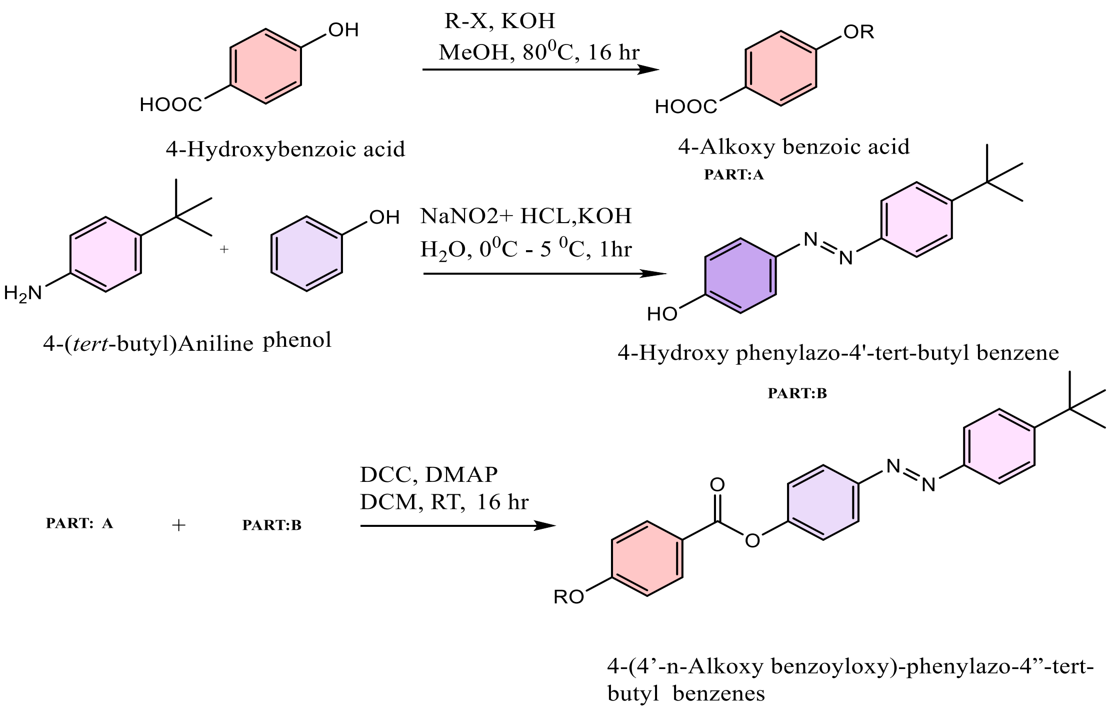
Synthesis of Part: B

4-tert butyl aniline (1 eq) was dissolved in water and HCL in a 500 mL beaker. The mixture was stirred in an ice bath until the solid completely dissolved. A calculated amount of sodium nitrite ($NaNO_2$) was dissolved in water and then slowly added to the reaction mixture, which was stirred for 30 minutes to complete the diazotization process. Now take another beaker, phenol and potassium hydroxide, and dilute in water. Now add both the solutions slowly into the cooled reaction mixture and stirred for few hours. After completion of reaction yellow precipitates were observed, which were filtered and dried under reduced pressure. Recrystallize using ethanol to remove colour impurity. [28]

Synthesis Of Final Product

Take 4-*n*-alkoxy benzoic acid (Part A) (1 eq) in DCM now add DCC (1.1 eq) and allow to react at room temperature for 1 hour. After white precipitate was observed, add part B (1 eq) and DMAP catalytic amount stirred the reaction mixture for 16 hours at room temperature. [29] After the completion of reaction filter out insoluble DCU and evaporate the DCM and add ethanol into the RM to fallout the desired product. [30]

Reaction Scheme



Scheme 1. Synthesis of homologues series A.

Spectral Data

- IR data of Hexayloxy *derivative:*
  FT IR in cm^{-1}: 2978 and 2929 (C-H, str. Of tert-butyl / alkyl chain), 1772 & 1730 (C=O, str. Of ester), 1685 & 1646 (azo conjugated carbonyl vibration), 1557 & 1509 (C=C str. of aromatic ring and –N=N– linkage), 1475 & 1457 (C–H bending of alkyl group), C–H deformation of tert-butyl group), 1263(-C-O-C str. Of ether).
- *IR data of Heptyloxy derivative:*
  FT IR in cm^{-1}: 3285 (aromatic -C–H str.), 2948 & 2915 (C–H str. of alkyl chain), 1674 (C=O str. of ester group), 1588 & 1548 (–N=N– azo str. and -C=C- str. of aromatic ring), 1446 & 1360 (C–H bending of tert-butyl/alkyl group), 1274 &1253 (C–O str. of ester linkage)
- *Mass data of propyloxy derivative:* 417.2 (M$^+$)
- *Mass data of butyloxy derivative:* 431.2 (M$^+$)
- *^{1}H NMR of hexyloxy derivative:*
  ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.23 – 8.13 (m, 2H), 8.05 – 7.97 (m, 2H), 7.92 – 7.86 (m, 2H), 7.60 – 7.53 (m, 2H), 7.43 – 7.35 (m, 2H), 7.05 – 6.97 (m, 2H), 4.08 (t, *J* = 6.6 Hz, 2H), 1.86 (p, *J* = 6.7 Hz, 2H), 1.52 (tt, *J* = 9.6, 5.0 Hz, 3H), 1.41 (s, 13H), 0.99 – 0.91 (m, 1H).

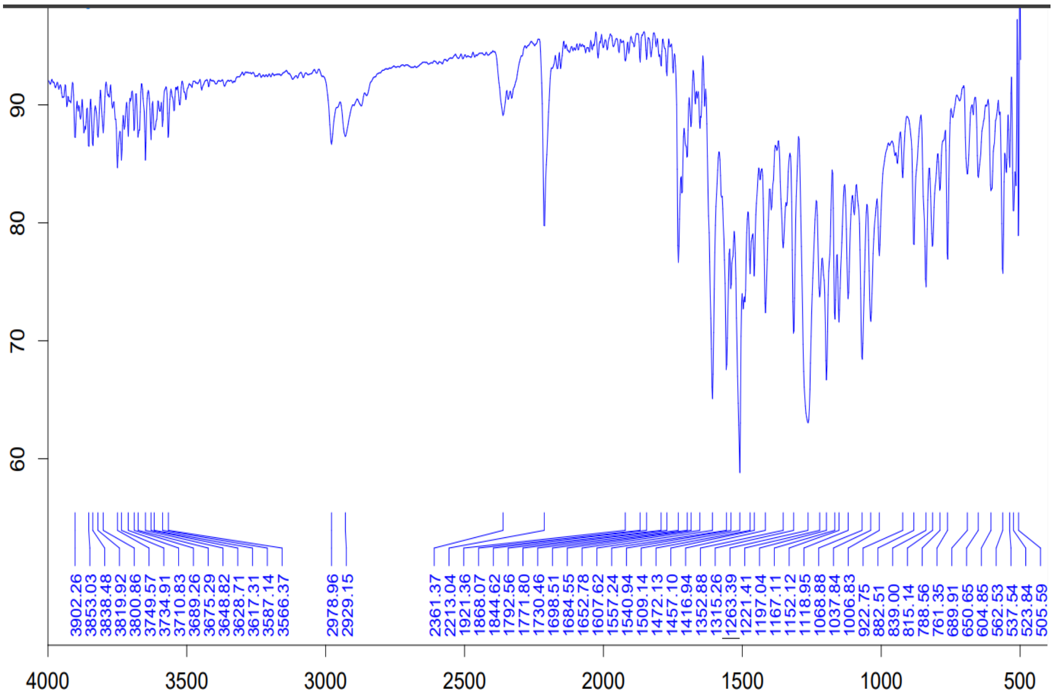


Figure 1. IR in Cm^{-1} for hexyloxy derivative.

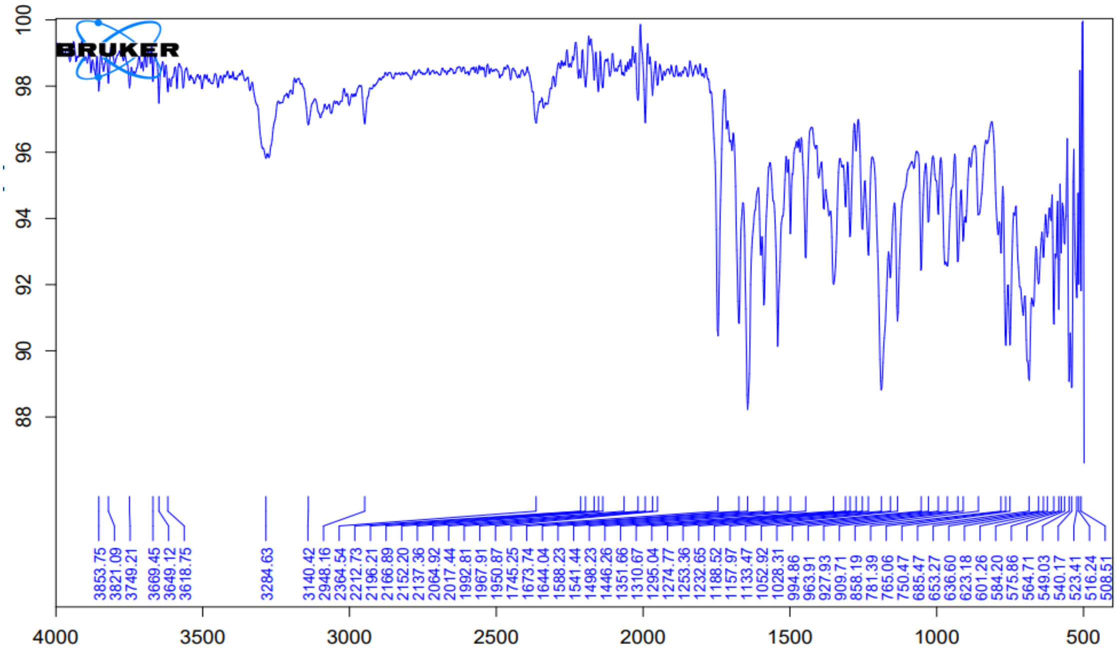


Figure 2. IR in Cm^{-1} for heptyloxy derivative.

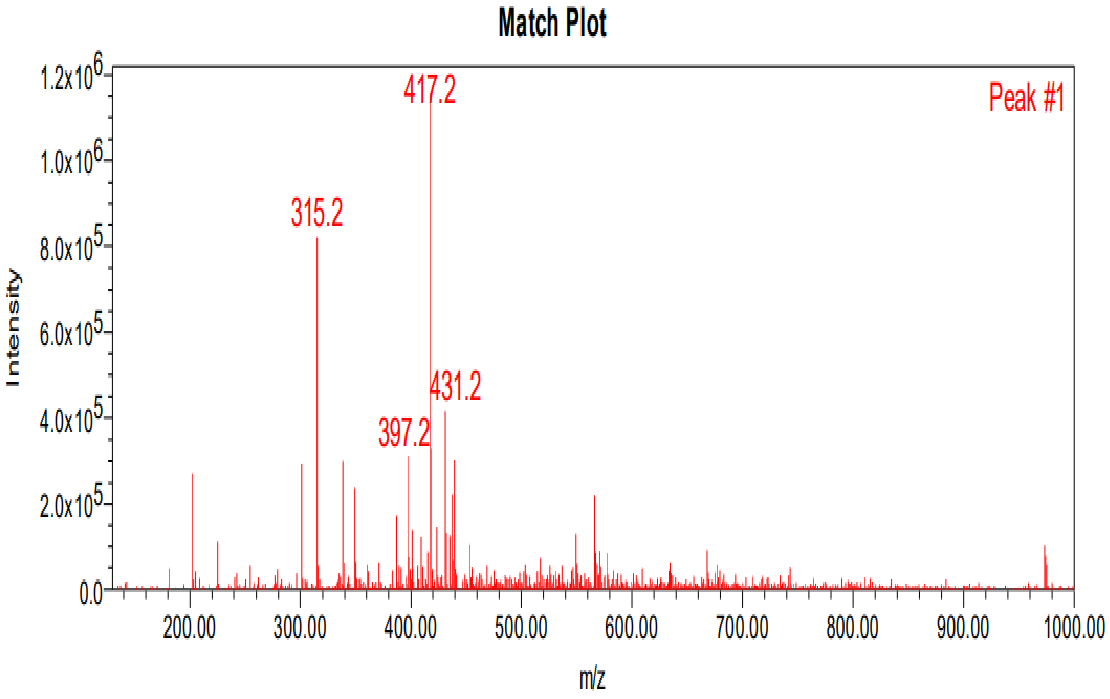


Figure 3. Mass of propyloxy derivative.

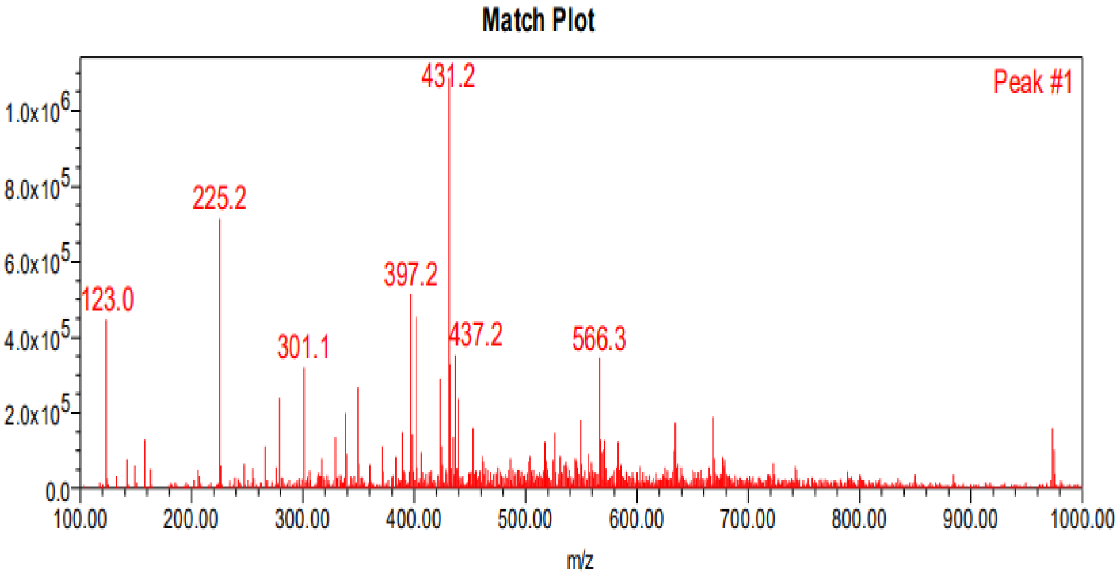


Figure 4. Mass of butyloxy derivative.

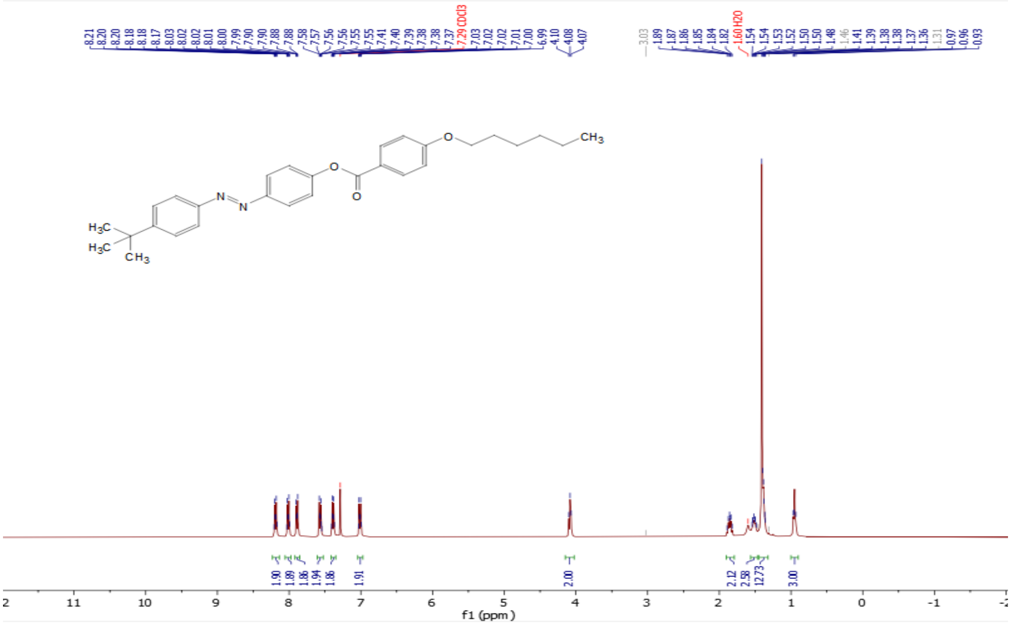


Figure 5. NMR of hexyloxy derivative.

Characterization

A few selected compounds from the series were characterized using standard analytical techniques. The FT-IR spectra were recorded on a Bruker Alpha II FTIR spectrometer in the 4000–400 cm⁻¹ range, and the ^{1}H NMR spectra were obtained using a Bruker AV Neo 400 MHz spectrometer with $CDCl_3$ as the solvent. The thermal behavior of the compounds was examined using a Differential Scanning Calorimeter (Shimadzu DSC-60, Singapore, version 10.8).

The mesomorphic textures were observed under a Nikon Eclipse 400/TU Plan ELWD 20X/0.40 polarized optical microscope (POM). Because all members of the series possess the same molecular core and vary only by the length of their terminal alkyl chains, confirming the structure of a few representative compounds is sufficient to validate the entire series. This approach is commonly followed in the study of liquid crystalline homologous series.

3. RESULTS AND DISCUSSION

The mesomorphic characteristics of present series (A_1-A_{14}) appraised through polarising optical microscopy and differential scanning calorimetry. The result obtained from polarising optical microscopy are presented below.

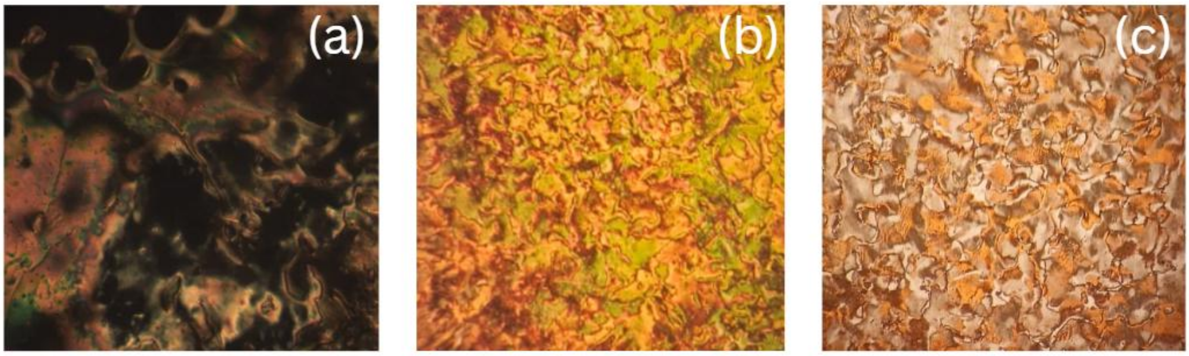


Figure 6. (a) Nematic phase of A_8 at 140.9 °C on heating cycle. (b) Nematic phase of A_4 at 135.6 °C on heating cycle. (c) Nematic phase of A_6 at 120.8 °C on heating cycle.

As given in figure, the textures displayed in Fig. 6 (b) and (c) represents classic schlieren patterns, distinguished by erratic, thread-like formations. These specific characteristics serve as reliable diagnostic markers for the nematic (N) phase, as they reflect a state of long-range orientational alignment in the absence of any structural positional order. While Fig. 1(a) shows dark region with bubble like pattern which indicates N-I transition phase just before clearing point.

The transition temperatures for the newly synthesized azo-ester series demonstrate that their liquid crystalline (mesomorphic) properties rely on the length of their terminal alkyl chains. The shortest homologues (A_1-A_3) melt directly from a solid crystal to an isotropic liquid, without showing liquid crystal phase because their short chains cannot generate the necessary structural directionality (anisotropy). However, liquid crystalline phases emerge starting with A_4 (Table 1). The delay in mesomorphism is caused by the tert butyl group's steric hindrance. Which inhibit close molecular packing and decrease intramolecular anisotropic interactions essential for liquid crystalline state. [31]

Table 1. Transition temperature of homologous series A

Compound	**No of carbon in alkyl chain (C_nH_{2n+1})**	**Transition Temperature in ^{0}C**		
		Sm	**N**	**I**
A_1	1	-	-	150
A_2	2	-	-	169.2
A_3	3	-	-	173.3
A_4	4	-	112.5	159.9
A_5	5	-	116.2	143.7
A_6	6	-	106.7	146.4
A_7	7	-	108.6	135.6
A_8	8	-	105.2	142.3
A_{10}	10	-	91.2	129.8
A_{12}	12	-	96.3	127.4
A_{14}	14	-	94.8	124.5

A clear correlation was observed between alkyl chain length and phase transition temperature (Fig. 2). The nematic–isotropic transition temperature fluctuate unpredictably from 159.9 °C for (A_4) to 124.5 °C for (A_{14}). The observed reduction in thermal stability arises from increased conformational entropy within the alkyl chain length. As the alkyl chain extends, significant steric hindrance and conformational entropy are introduced. The N-I transition temperature reveals downward trend, while the Cr-N transition temperature exhibits non-linear irregularities, indicates that the crystalline phase is strongly governed by conformational packing effects. [32]

Given figure 2 clearly shows influence of alkyl chain length on mesomorphic behaviour. The lower homologues are non-mesomorphic because it exhibits direct crystalline to isotropic (Cr-I) transition.

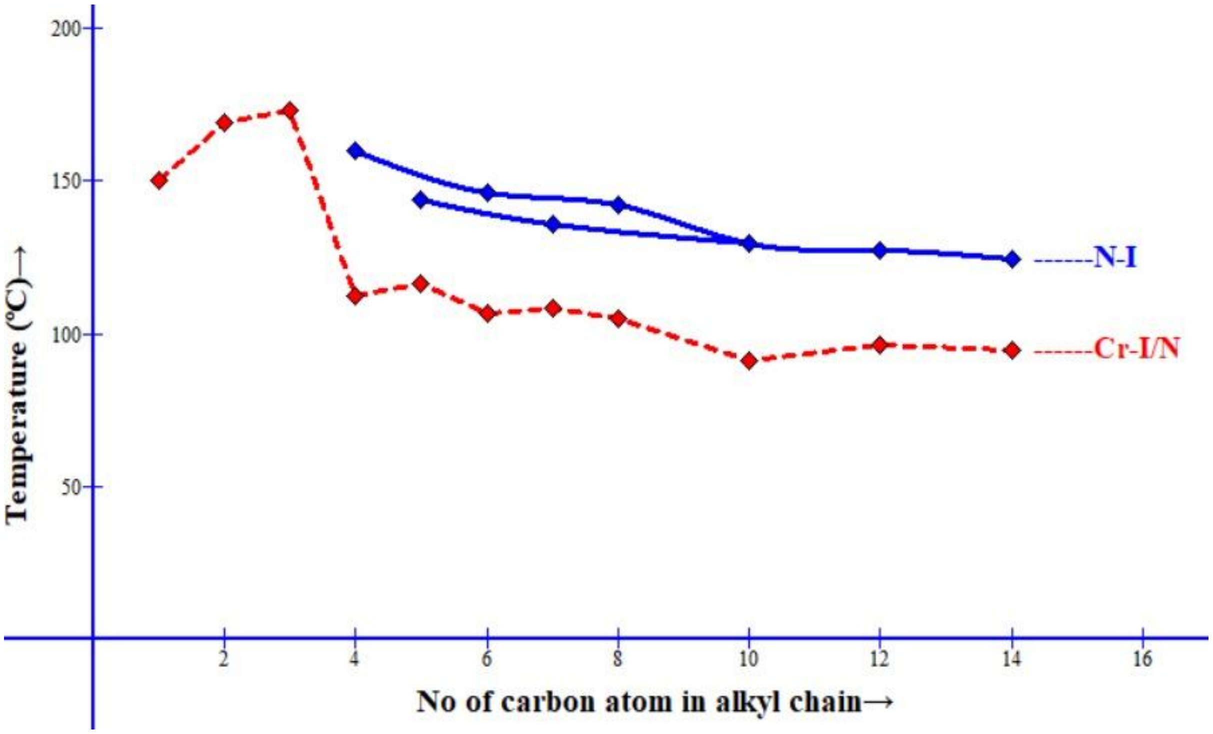


Figure 7. Phase transition behaviour of series A.

The disclosure of nematic phase suggests that, to promote liquid crystalline behaviour, the minimum alkyl chain length is necessary. From the graph we can say that the nematic-isotropic transition temperature (blue curve) moderately decreases with an increase in alkyl chain length from 160 °C for A_4 to about 124 °C for A_{14}; nematic stability weakens as molecular flexibility increases. Conversely, the Cr-N transition represented by the red curve drops sharply from A_4 to A_6, after which it remains stable at 90 to 100 °C for higher homologues, this specifies that as the alkyl chain length increases, the crystal arrangement becomes more secure. The intermediate chain length refines nematic phase stability, while widen chain length encourage structural disorder, which lowers the transition temperature. [33]

The Cr-I/N transition temperature curve display a characteristic zigzag pattern, especially in lower homologues (A_1-A_{10}). The transition temperature rises from C_1 to C_3 followed by sharp decline at C_4, a slight rise at C_5 and then a gradual decrease at around C_6-C_{10}, after which shows a slight variation at higher homologues. The overall zigzag path shows a descending trend. The N-I transition curve shows the odd-even effect very clearly with increasing alkyl chain length. Even-numbered homologues exhibit high transition temperature due to their greater molecular symmetry and molecular packing; conversely, odd numbered homologues have low transition temperatures because of molecular distortion and less efficient packing. The odd-even effect mearge at C10 homologues. At this point, transition temperature combines into a smooth and continuous curve.

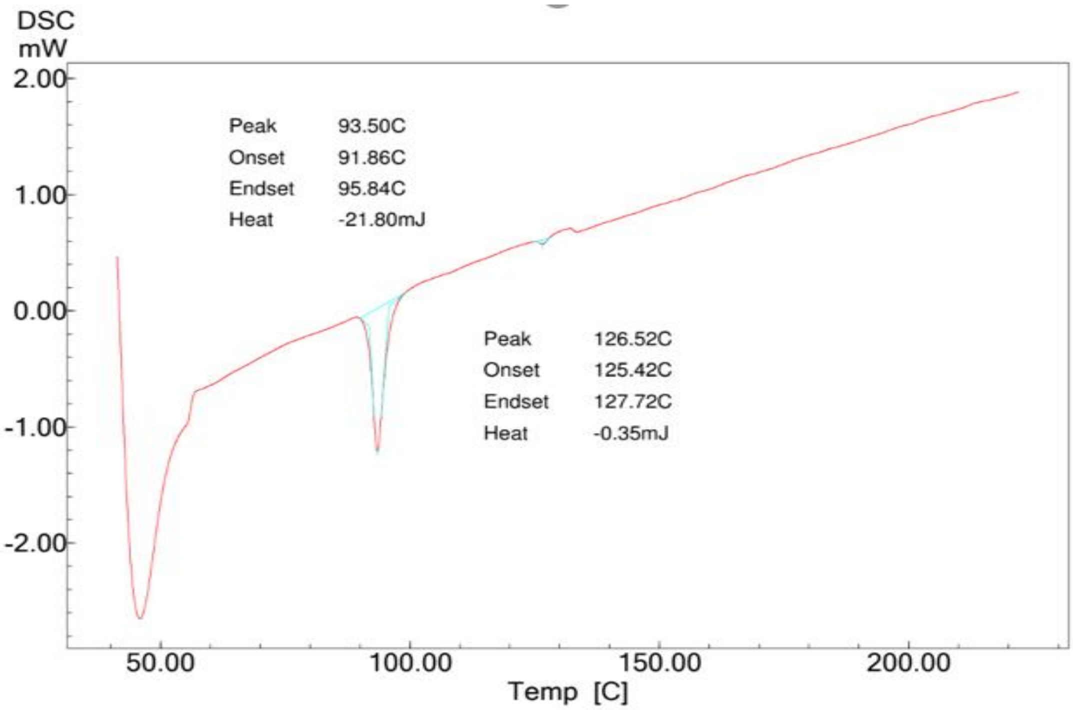


Figure 8. DSC thermogram of A_{12} homologue.

Differential scanning calorimetry (DSC) analysis of homologue A_{12} revealed specific thermal transition points (Fig. 3). The thermogram of A_{12} shows two endothermic peaks: first transition at 93.5 °C, representing the melting from a crystalline state into mesophase (Cr-N), and a narrower peak appears at 126.5 °C, representing the transition from mesophase to isotropic liquid (N-I). During this second transition, the enthalpy change is very minimal, resulting from a lower molecular orientation, matching the anticipated properties of the nematic mesophase.

Comparison Study

Comparative study of series A (bearing *tert*-butyl group) with previously reported structurally similar series X (bearing *sec*-butyl group) [31] shows a significant difference in mesomorphic behaviour, driven by structural variations in the terminal group architecture (Fig. 9).

In series A, the attachment of tert- butyl terminal group to the aromatic core led to achiral and highly symmetric molecular end and do not favour any chiral mesophase. While in series X there is a sec-butyl terminal group with stereo genic centre making the molecule asymmetric and chiral. Which improves the thermal stability of nematic phase and encourages the formation of chiral (N^*) nematic phase.

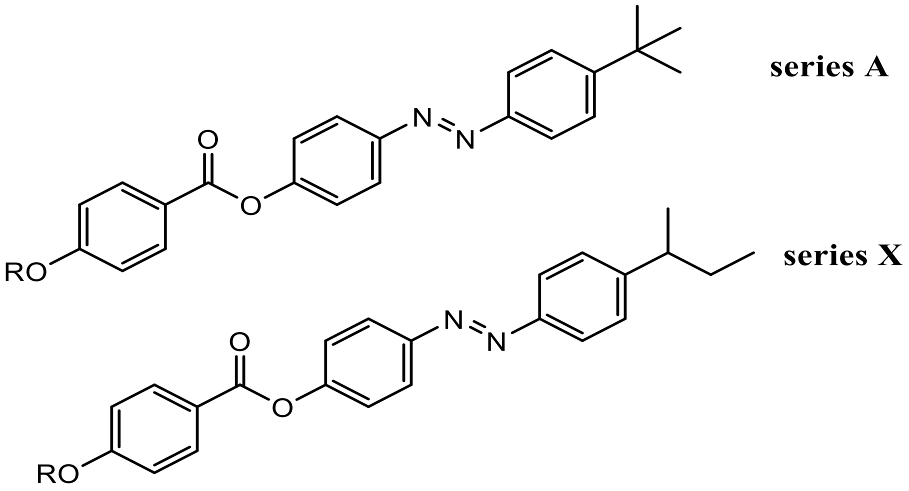


Figure 9. Structurally analogous series.

As shown in Table 2, series A demonstrate a consistence and distinct nematic phase across the A_4 to A_{14} homologous range, without development of any smectic phase. A phase transitions are sharp and precisely defined. In contrast, series X displays both chiral nematic (N^*) plus smectic (SmA and SmC) phases, with smectic behaviour becomes more prominent in higher homologous. Although appearance of smectic phase reflects enhanced molecular ordering, the exclusive nematic behaviour of series A is highly beneficial it ensuring the high fluidity, homogenous alignment and rapid response time required for high performance application. The phase behaviour is guide by terminal substituent a bulky tert-butyl substituent creates steric hindrance and introduce nonlinear geometry, which prevents molecular close packing essential for smectic phase however, sec-butyl promote layered packing.

Table 2. Average thermal stabilities in °C.

Series →	A	X
Smectic-Nematic	-	105.7 °C
Commencement of smectic mesophase	-	C_1
Nematic-Isotropic	138.7 °C	137.7 °C
Commencement of Nematic mesophase	A_4	C_{10}
Total Mesophase length range (Sm+N)	27.0 °C (C_7) to 47.7 °C (C_4)	7 °C (C_{10}) to 86 °C (C_9)

In terms of thermal behaviour, we can say that Series X exhibits enhanced thermal stability in its lower homologues (1 to 4) due to stronger molecular interactions, while Series A shows gradual decrease in transition temperature. Steric bulk introduced by tert-butyl substituent is responsible for reduction in N-I clearing temperature of Series A. for crystalline to mesophase transition (Cr-N), series A possess lower melting points (90 to 110 °C) suggests that bulky tert-butyl group remarkably disrupts lattice energy. However, Series X maintains more stable crystalline transition indicating that sec-butyl group facilitate sturdier intermolecular packing. In Series A commencement of nematic mesophase started from $A_{4.}$ [32-33]

4. CONCLUSION

A new homologous series of eleven homologues (A_1-A_{14}) were synthesized to study the influence of sterically hindered tert-butyl terminal group.

Series A shows n enantiotropic nematic mesophase, whereas Series X exhibit both enantiotropic nematic as well as smectic mesophase.

The relative effectiveness of different groups in promoting smectic and nematic phases was evaluated on the basis of:

- Thermal stability;
  Smectic: Series X > Series A
  Nematic: Series A > Series X
- Commencement of mesophase
  Smectic: Series X > Series A
  Nematic: Series A > Series X
- The total mesophase length
  Series X > Series A

The liquid crystal behaviour of present series is driven by molecular structure, with the balance of rigidity and flexibility and the effect of tert-butyl group, which remarkably influence mesophase property and thermal stability.

The Series A shows enantiotropic nematic mesophase (A_4 toA_{14}) without the formation of smectic phase throughout the series because of steric hindrance disrupting the layered packing.

The variation in transition temperature shows that these compounds are highly sensitive to chain length. This tuneable property makes them favourable candidates for electro-optical technologies and other advance material.

Acknowledgement

The authors express their sincere gratitude to the Department of Chemistry, Saurashtra University, Rajkot, for providing chemicals and lab facility. The authors also extend their appreciation to the Birla Institute of Technology and Science (BITS), Pilani, for analytical support.

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