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Mesomorphism Dependence on Chalconyl Derivative with Heterocyclic Tail and Methoxy Lateral Group

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ABSTRACT

A new homologous series of liquid crystal (LC) materials incorporating a heterocyclic ring was synthesized to investigate and establish the relationship between molecular structure and liquid crystal properties. The series comprises 12 members, labeled A_1 to A_{12}. Among them, A_8 to A_{12} exhibit monotropic nematic phases. Comparative studies between mesomorphic and non-mesomorphic members were conducted, leading to several important conclusions. Transition temperatures were measured using a polarizing optical microscope equipped with a heating stage. Analytical and spectral analyses confirmed the molecular structures of the homologues. This series is classified as having middle-ordered melting characteristics. The LC properties of this new series were also compared with those of structurally related known series.

Keywords: Mesomorphic, Homologues, Nematic, Monotropic.

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1. INTRODUCTION

Liquid crystals have both the optical characteristics of solids and the mobility of liquids. The synthesis of usual thermotropic liquid crystals was previously dependent upon the presence of isometric rod-like (calamitic) or disc-like (discotic) molecules, as steric packing considerations are significant in this intriguing soft matter state.

A thermotropic LC molecule is made up of two parts: the tail, which is made up of either straight alkyl or alkoxy chains to provide flexibility and stabilize the molecular alignment, and the core, which consists of a linkers rod-like structure to link aromatic rings by different linker[1–3]. As the core units, heterocycle are of great importance thermotropic liquid crystals due to their ability to impart changes in lateral and longitudinal dipoles combined with changes in the molecular shape [4–7].

A molecule's mesomorphic behavior can be changed by even slight changes to its structure. Heteroatoms (S, O, and N) can change the polarity, polarisability, and even the geometric properties of the molecule's, which in turn effects the mesogens' phase transition temperatures and other properties[8–11]. Oxygen-containing heterocyclic compound benzofuran has been explored on liquid crystal molecules [12,13].

The correlation between the properties and structures demonstrates that linear molecules are fascinating to create stable mesophases with wide range of mesomorphic temperature, while in the bent shape molecules there is possibilities to disappearance of mesophase. We went back to creating a single heteroatom-containing LC material, concentrating on benzofurans because they were often used in optoelectronics and photovoltaics but rare in LCs.The investigation also showed that the mesomorphic characteristics are significantly influenced by the length of the alkyl chain. A beneficial charge transfer axis results from π-bond conjugation with chalcones (α, β-unsaturated ketone), and aromatic rings serve as a tool for both donors and acceptors [14–17].

One important area of study in the molecular design of nonlinear optical materials is the impact of donor–acceptor strength on the electronic characteristics of conjugated molecules. Chalconyl derivatives exhibit thermotropic liquid crystal (LC) properties combined with bioactivity properties, which could be used for the good of mankind in the production of LC devices and pharmaceutical preparations.

2. MATERIALS

Vanillin, n-alkyl bromides (R-Br), Potassium carbonate (K_2CO_3), thiophene-2-aldehyde, 4-hydroxy acetophenone, MDC, Ethanol, Piperidine, Pyridine, Malonic acid and other required reagents were procured from Spectrochem, Merck, and Sigma-Aldrich. All chemicals and solvents were used without any further purification. The purity of the synthesized compounds was confirmed using thin-layer chromatography (TLC), performed on 0.2 mm pre-coated silica gel G60 F254 plates (Merck). Visualization of the spots was carried out under UV light at wavelengths of 254 and 365 nm.

3. EXPERIMENTAL

3.1. Synthesis

Synthesis of 4-n-alkoxy-3-methoxy benzaldehyde

4-n-Alkoxy-3-methoxy benzaldehyde was synthesized by reacting 4-hydroxy-3-methoxy benzaldehyde (1.0 eq.) with n-alkyl bromide (1.0 eq.) in the presence of potassium carbonate (1.0 eq.) using DMF as the solvent[18]. The reaction mixture was refluxed at 80 °C for 8 hours. Completion of reaction monitored by TLC.

Synthesis of 4-n-alkoxy-3-methoxy cinnamic acid

4-n-Alkoxy-3-methoxy benzaldehyde (1.0 eq.) was reacted with malonic acid (1.2 eq.) using piperidine as a catalyst and pyridine as the solvent. The reaction mixture was refluxed for 6 hours[19]. The completion of reaction was confirmed by TLC using 3:7 Ethyl acetate: Hexane as mobile phase. After completion of reaction, the reaction mass was neutralized with 2M HCl solution. The 4-n-alkoxy-3-methoxy cinnamic acid was found as a white solid product.

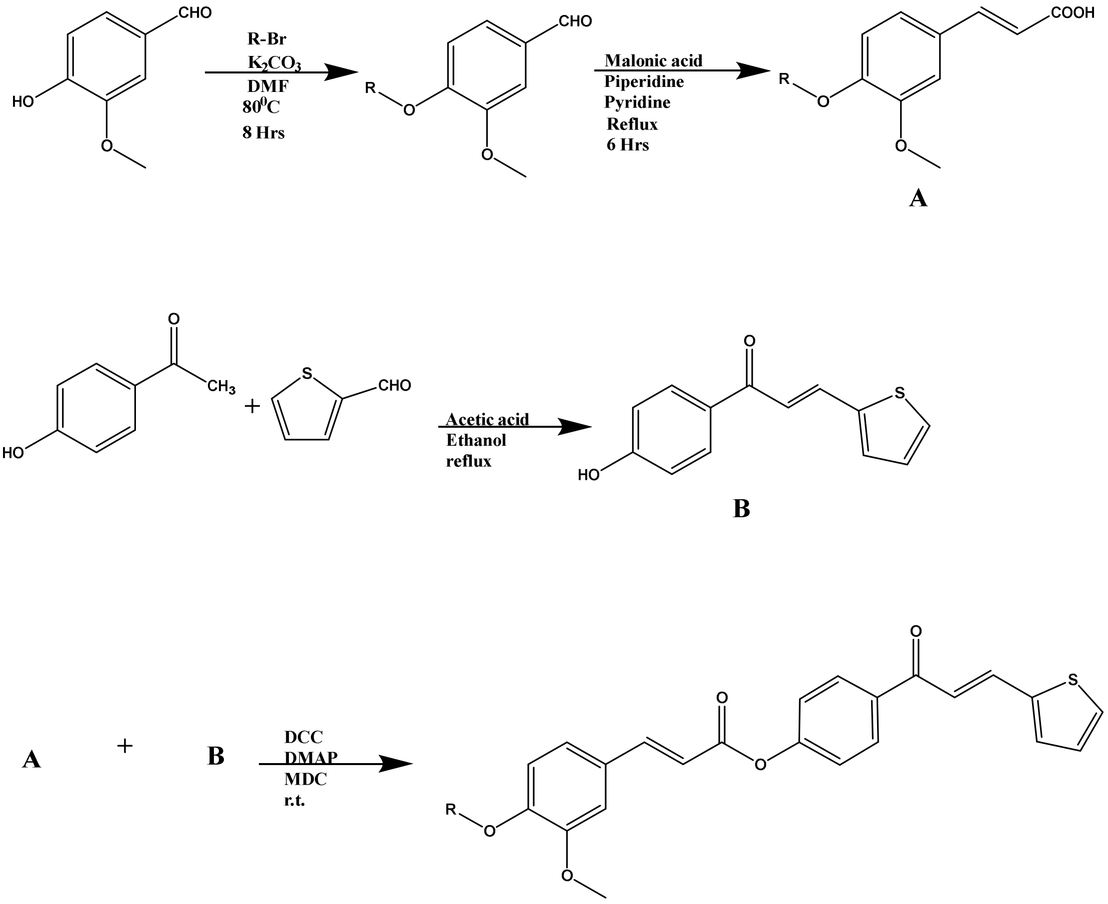
Synthesis of α-4-Hydroxy Benzoyl-β-(2’-thiophenyl) ethylene

4-Hydroxy acetophenone (1.0 eq.) was reacted with thiophene-2-aldehyde (1.0 eq) using KOH and methanol as solvent[20]. the reaction mixture was refluxed for 2 hours. The completion of reaction was confirmed by TLC using 3:7 Ethyl acetate: Hexane as mobile phase.

Synthesis of α-4(4’-n-alkoxy 3’-methoxy cinnamoyloxy) benzoyl-β-(2’-thiophenyl) ethylene

4-n-alkoxy-3-methoxybenzaldehyde was reacted with α-4-Hydroxy benzoyl-β-(2’-thiophenyl) ethylene via staglich esterification using DCC,DMAP as reagent and MDC as solvent[21]. The completion of reaction was confirmed by TLC. Obtained product is recrystallized using ethanol.

4. SYNTHETIC ROUTE



α-4(4' -n-alkoxy 3'-methoxy cinnamoyloxy) benzoyl-β-(2-thiophenyl) ethylene

Where, R=CnH2n+1, n=1 to 8,10,12,14 & 16

Scheme 1. Synthetic route of the series A.

5. CHARECTARIZATION

Representative members of the novel series were characterized by elemental analysis (Table 1), Infrared spectroscopy, ^{1}HNMR spectroscopy, and mass spectroscopy. Microanalysis was performed on Perkin-Elmer PE 2400 CHN analyzer. IR spectra were recorded on Shimadzu FTIR-8400, ^{1}H NMR spectra were recorded on Bruker spectrometer using $CDCl_3$ as solvent and mass spectra were recorded on Shimadzu GC-MS Model No. QP-2010. The mesogenic behavior of the homologue derivatives were observed through polarizing microscopy.

Table 1. Elemental analysis for (1) propyloxy (2) butyloxy (3) heptyloxy derivatives.

Sr. no	molecular formula	Elements % found				Elements % calculated			
		C	H	O	S	C	H	O	S
1	$C_{26}H_{24}O_5S$	69.56	5.35	17.86	7.23	69.62	5.39	17.83	7.15
2	$C_{27}H_{26}O_5S$	70.13	5.65	17.24	6.98	70.11	5.67	17.29	6.93
3	$C_{30}H_{32}O_5S$	71.36	6.32	15.87	6.45	71.40	6.39	15.85	6.35

6. ANALYTICAL DATA

IR in cm^{-1} for hexyloxy derivative (Figure 1)

3070.78(C-H str. of disubstituted alkene), 2924 & 2854(C-H str. of alkyl chain), 1728(C=O str. of ester), 1651 (C=O str. of unsaturated ketone), 1627 & 1589(C=C str. of alkene), 1589& 1512(C=C str. of aromatic ring), 1303 & 1273(C–H bending of alkene disubstituted), 1211(C–O str. of ether linkage), 1126(C–O str. of ester group), 964, 817 & 702(C–H bending of alkene).

IR in cm^{-1} for decyloxy derivative (Figure 2)

3070(C-H str. of disubstituted alkene), 2924 & 2854(C-H str. of alkyl chain),1728(C=O str. of ester), 1651 (C=O str. of unsaturated ketone), 1627 & 1597(C=C str. of alkene), 1512(C=C str. Of aromatic ring), 1303 & 1273(C–H bending of alkene disubstituted), 1211(C–O str. of ether linkage), 1126(C–O str. of ester group), 956,848 & 817(C–H bending of alkene).

Mass spectra of pentyloxy derivative (Figure 3)

m/z (rel.int%):477.15,191.15, 163.10

Mass spectra of octyloxy derivative (Figure 4)

m/z (rel.int%):518, 289, 177, 145

^{1}H NMR in ppm of dodecyloxy derivative (Figure 5)

^{1}H NMR (500 MHz, DMSO-*D*$_6$) δ 8.19 – 8.13 (m, 2H,middle phenyl ring), 7.90 (d, *J* = 15.2 Hz, 1H, -CO-CH=C$\underline{H}$-),7.81(s,1H, C$\underline{H}$=CH–COO–), 7.78 – 7.75 (m, 1H,thiophene ring), 7.68 (d, *J* = 3.6 Hz, 1H,thiophene ring), 7.56 (d, *J* = 15.3 Hz, 1H, -CO-C$\underline{H}$=CH-), 7.43 (d, *J* = 2.0 Hz, 1H,phenyl ring near –OCH_3), 7.40 – 7.33 (m, 2H,middle phenyl ring), 7.29 (dd, *J* = 8.4, 2.0 Hz, 1H,phenyl ring near –$OC_{12}H_{25}$ group), 7.17 (dd, *J* = 5.0, 3.7 Hz, 1H,thiophene ring), 6.98 (d, *J* = 8.3 Hz, 1H,phenyl ring near –$OC_{12}H_{25}$ group), 6.79 (d, *J* = 15.9 Hz, 1H, –CH=C$\underline{H}$–COO–), 3.97 (t, *J* = 6.5 Hz, 2H, -CH_2 of –$OC_{12}H_{25}$ group), 3.79 (s, 3H, -CH_3 of –OCH_3 group), 1.68 (p, *J* = 6.7 Hz, 2H, -CH_2 of –$OC_{12}H_{25}$ group), 1.36 (p, *J* = 6.9 Hz, 2H, -CH_2 of –$OC_{12}H_{25}$ group), 1.27 (d, *J* = 7.5 Hz, 2H, CH_2 of –$OC_{12}H_{25}$ group), 1.20 (t, *J* = 6.1 Hz, 16H,-$(CH_2)_8$of –$OC_{12}H_{25}$ group), 0.85 – 0.78 (t, 3H, -CH_3 of –$OC_{12}H_{25}$ group).

^{1}H NMR in ppm of octyloxy derivative (Figure 6)

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.10 – 8.06 (m, 2H, middle phenyl ring), 7.96 (d, *J* = 5.4 Hz, -CO-CH=C$\underline{\text{H}}$- 1H), 7.84 (d, *J* = 15.5 Hz, C$\underline{\text{H}}$=CH–COO– 1H),7.43 (d, *J* = 5.4 Hz, 1H,thiophene ring, 7.39 (t, *J* = 7.4 Hz, 1H,thiophene ring), 7.35 – 7.31 (m, 2H,middle phenyl ring), 7.16 (t, *J* = 7.1 Hz, 1H, , -CO-C$\underline{\text{H}}$=CH-), 7.11 (d, *J* = 9.3 Hz, 1H, phenyl ring near $–OCH_3$), 6.90 (d, *J* = 8.2 Hz, 1H, thiophene ring), 6.50 (d, *J* = 16.0 Hz, 2H, ,phenyl ring near of $–OC_8H_{17}$ group), 5.03 (s, 1H, –CH=C$\underline{\text{H}}$–COO–),4.07(t, *J* =6.9,2H,$-CH_2$ of of $–OC_8H_{17}$ group),3.92(s,3H,$-CH_3$ of $–OCH_3$ group), 2.35 (t, *J* = 9.9,2H, $-CH_2$ of $–OC_8H_{17}$ group), 1.88-1.84(m,2H, $-CH_2$ of $–OC_8H_{17}$ group) ,1.36 – 1.25 (m, 8H, $-(CH_2)_4$of $–OC_8H_{17}$ group),0.89(t, *J* = 6.3 Hz, 3H, $-CH_3$ of $–OC_8H_{17}$ group).

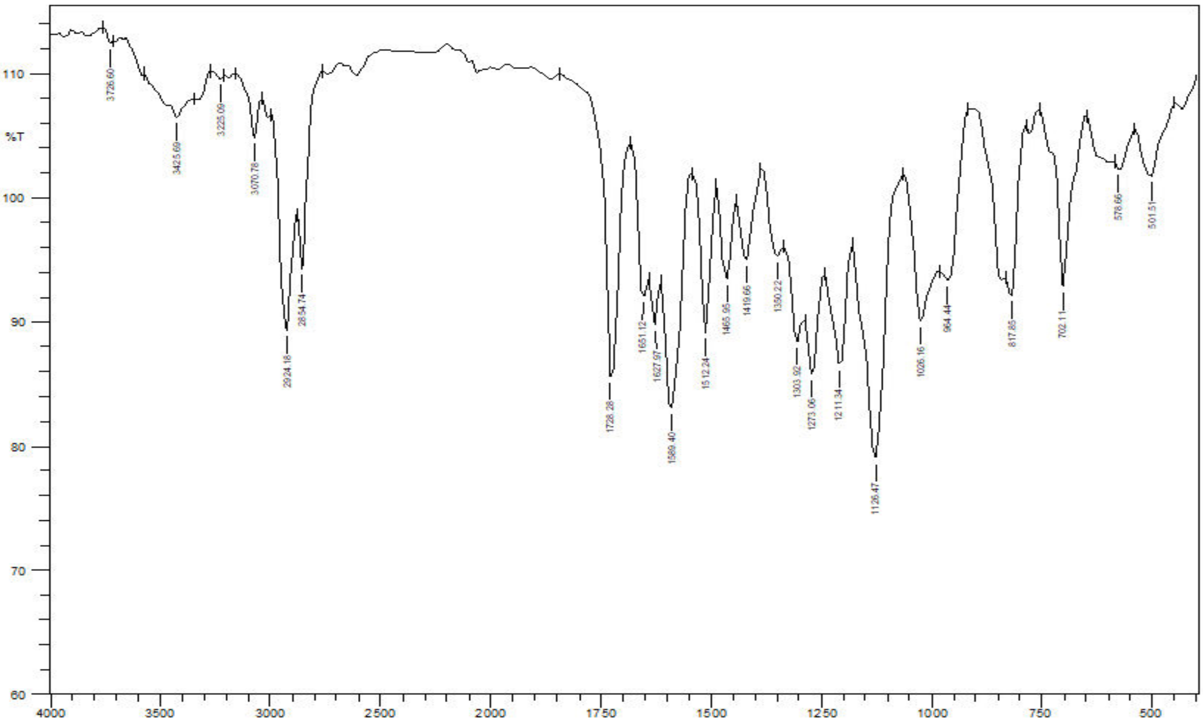


Figure 1. IR in cm^{-1} for hexyloxy derivative.

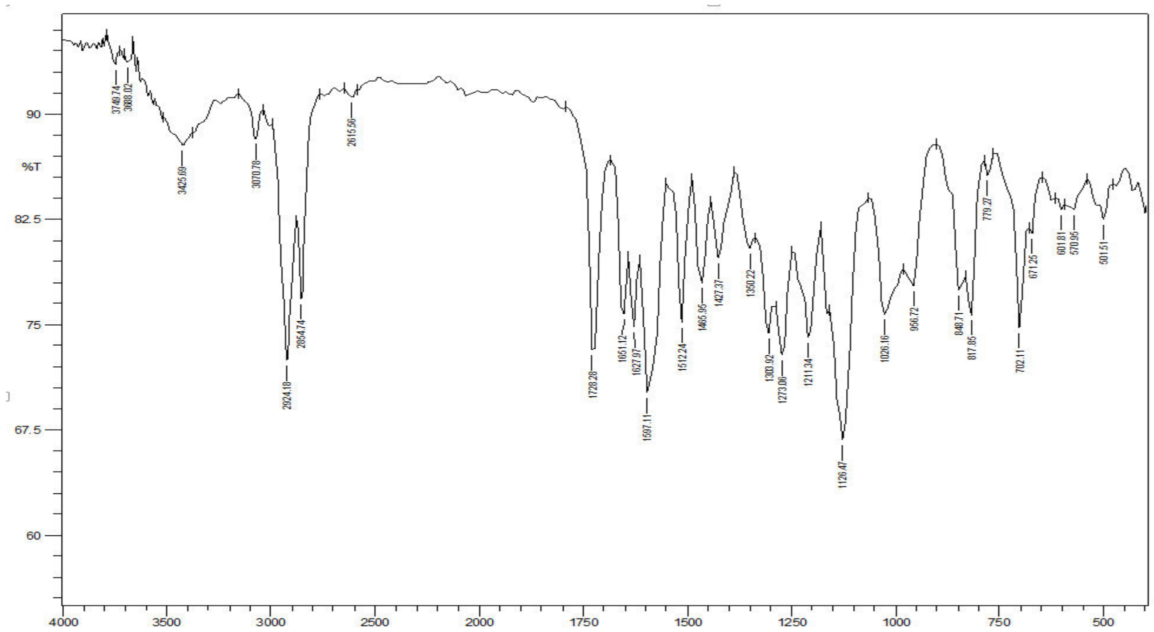


Figure 2. IR in cm^{-1} for decyloxy derivative.

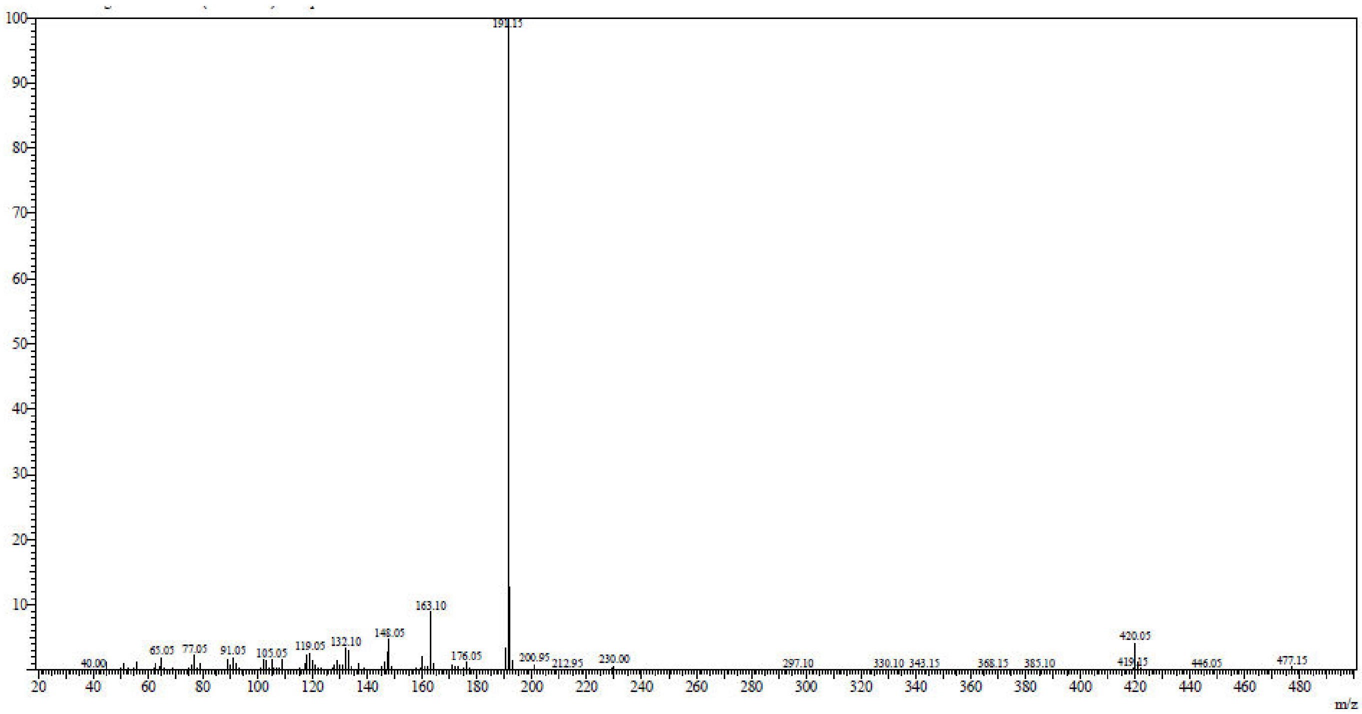


Figure 3. Mass spectrum of pentyloxy derivative.

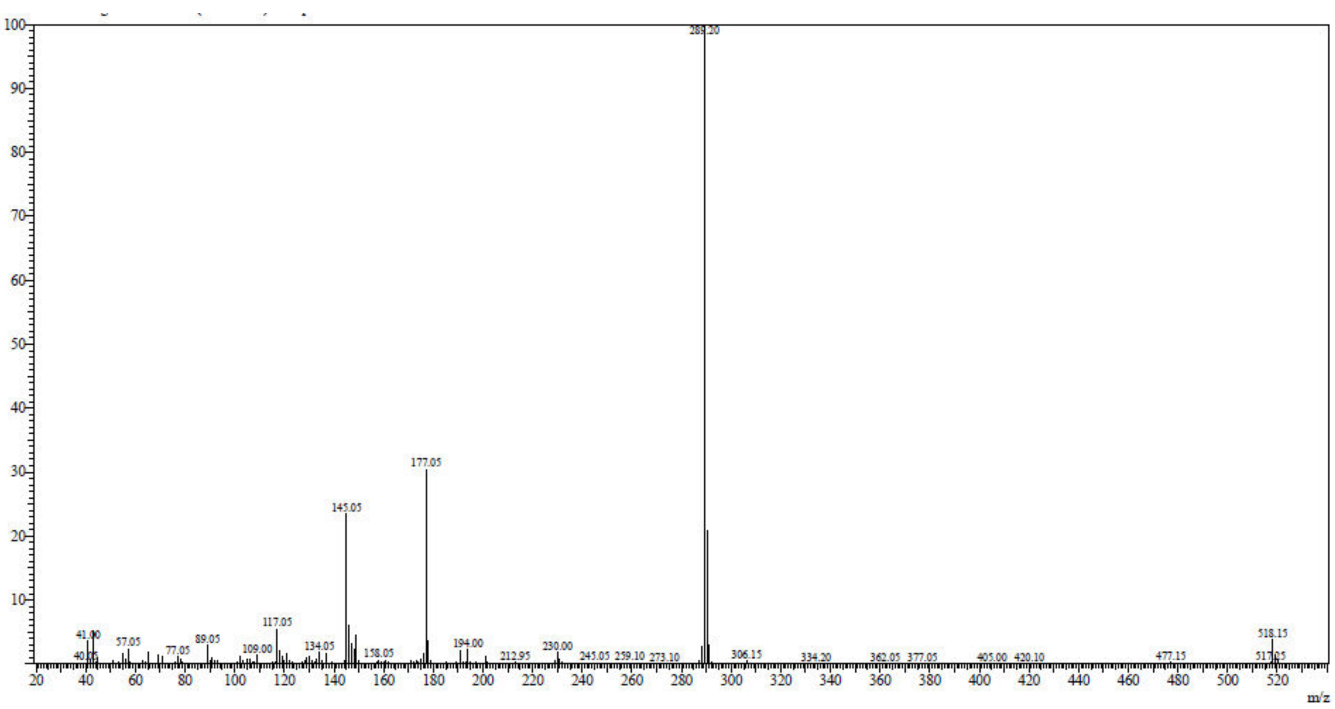


Figure 4. Mass spectrum of octyloxy derivative.

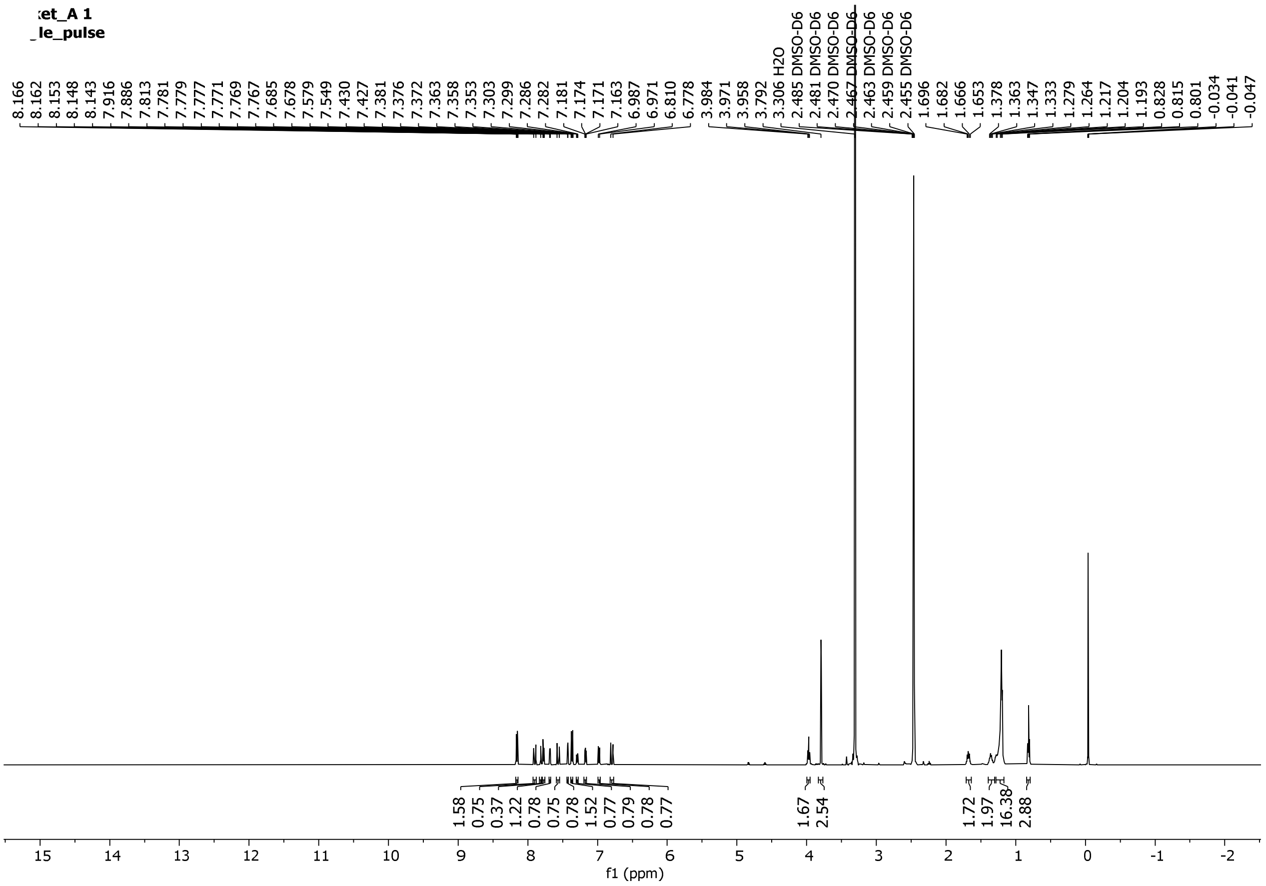


Figure 5. ^{1}H NMR in ppm of dodecyloxy derivative.

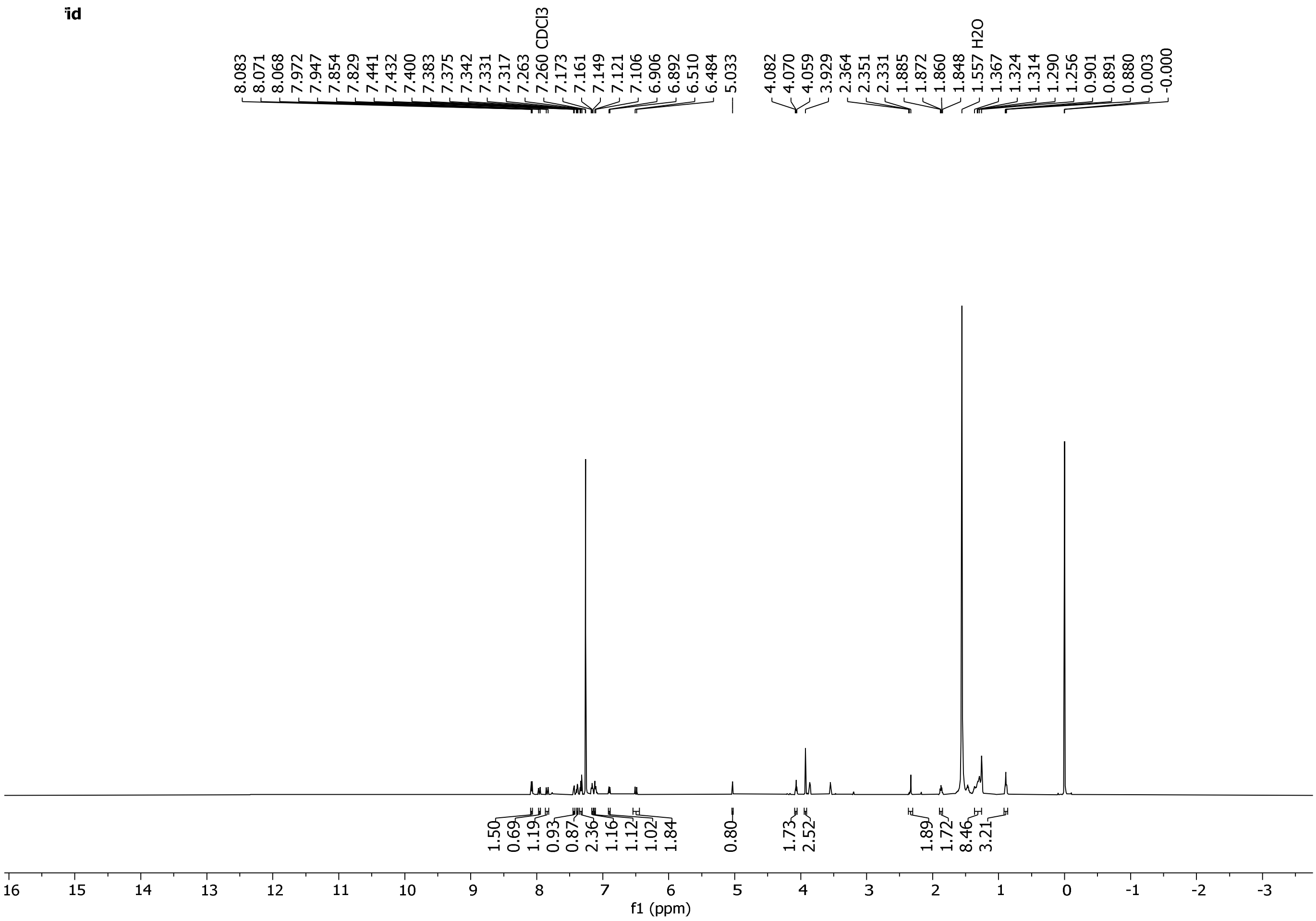


Figure 6. ^{1}H NMR in ppm of octyloxy derivative.

7. RESULT AND DISCUSSION

Mesomorphic homologues were synthesized by linking trans-4-n-alkoxy-3-methoxycinnamic acid with a 4-hydroxychalcone derivative containing a thiophene heterocyclic ring. The nematic mesophase appears starting from the A_8 homologue, with a total of five members in this new series exhibiting mesomorphic behavior. Transition temperatures (Table 2), determined using a polarizing optical microscope equipped with a heating stage, were plotted against the number of carbon atoms in the n-alkyl chain of the flexible -OR unit. Related data points were connected to produce Cr-I and I-N transition curves, illustrating the phase behavior of the new series in a phase diagram (Fig. 7). Analytical and spectral studies confirmed the molecular structures of the novel compounds. The Cr-I transition curve displays an zig-zag type from A_1 to A_6, The Cr-I curve shows an overall downward trend, dipping until A_8 and then leveling off through to A_{12}. The I-N transition curve remains flat from A_8 to A_{16}. Overall, the Cr-I transition curves display typical behavior, characterized by a general downward trend in isotropic transition temperatures.

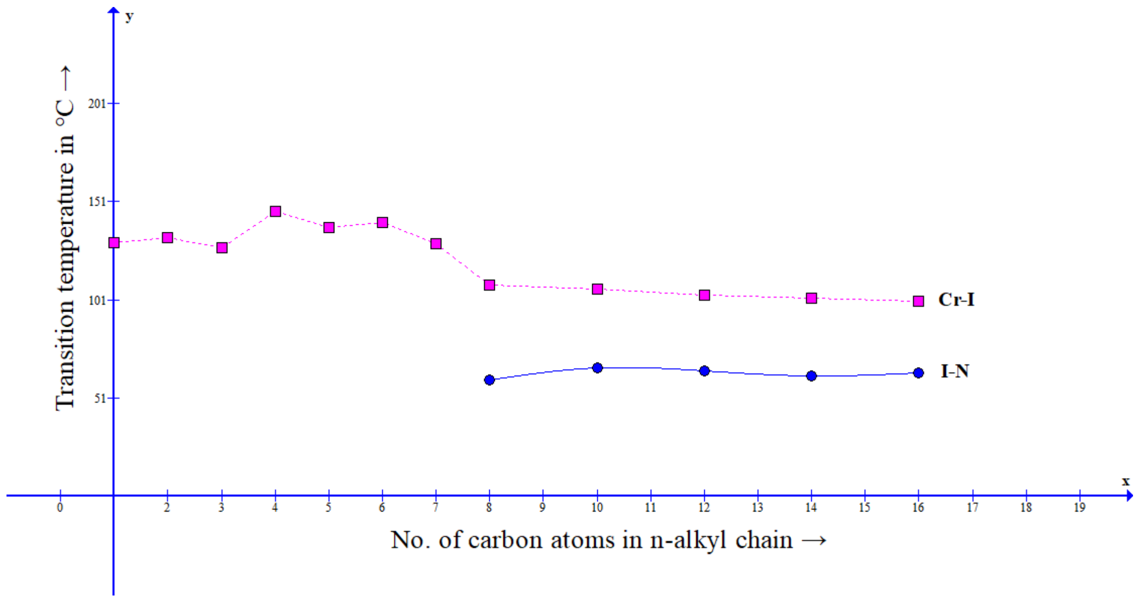


Figure 7. Phase transition behaviour of homologous series A.

Table 2. Transition temperature of homologous series A.

Sr NO.	No of carbon in n-alkyl chain (C_nH_{2n+1})	N	Isotropic
A_1	1	-	130.2
A_2	2	-	132.5
A_3	3	-	127.3
A_4	4	-	146.2
A_5	5	-	137.8
A_6	6	-	140.5
A_7	7	-	129.5
A_8	8	(60.1)	108.7
A_9	10	(66.2)	106.2
A_{10}	12	(64.7)	103.3
A_{11}	14	(62.3)	101.9
A_{12}	16	(63.9)	99.2

The current series is partially monotropically nematogenic in behaviour, with transition or melting temperatures ranging from 101.9 °C to 146.2 °C. The liquid crystalline properties vary across homologues within the series, showing alternating transition temperatures. As the series progresses—from one homologue to the next—through the sequential addition of methylene units, several molecular characteristics change, including molecular length, length-to-breadth ratio, flexibility, intermolecular end-to-end and lateral interactions, dipole moment along the molecular axis, dipole–dipole and electron–electron interactions, dispersion forces, polarity, and polarizability.

These changes occur due to increasing molecular flexibility, while the rigid core remains constant. These progressive alterations lead to conditions where the anisotropic forces are optimized to support the formation of the nematic mesophase. Consequently, homologues A_8 through A_{12} display mesomorphic behavior, each with distinct degrees of mesomorphism and varying transition temperatures.

The alignment of the homologues' molecules occurs at angles of 90° or less relative to the surface plane, enabling them to resist thermal agitation within a specific temperature range. This thermal resistance is determined by the unique balance of rigidity and flexibility in each molecule, which reflects the energy stored as enthalpy (ΔH), facilitating and stabilizing the formation of the nematic mesophase over a defined temperature range. Accordingly, this series is characterized as monotropic and exhibits a medium-order melting behavior.

Furthermore, the mesomorphic properties of the newly synthesized series-A were compared with those of a structurally similar series-X [22], as mentioned in Fig. 8. Both series share key structural features, including two phenyl rings, two central linkers (–CH=CH–COO– and –CO–CH=CH–), a left-side n-alkoxy terminal group, and a thiophene ring at the opposite end. The main distinction lies in the lateral substituent on the first phenyl ring, specifically the -OCH_3 group. Variations in mesomorphic behavior and the degree of mesophase stability are attributed to the combined effects of molecular rigidity and flexibility introduced by the structural differences between series-A and series-X.

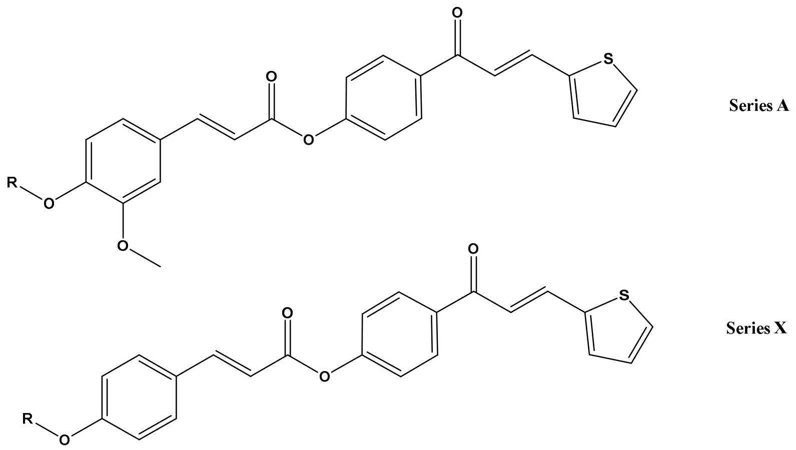


Figure 8. Structurally similar series.

Table 3. Average thermal stabilities in °C.

Series →	A	X
Smectic-Isotropic or Smectic-Nematic	–	132.86(C_6-C_{18})
Commencement of smectic mesophase	–	C_4

Nematic-Isotropic	_	152.78(C_1-C_{18})
Commencement of Nematic mesophase	A_8	C_1
Total Mesophase length range (N)/(Sm+N)	-	4.4(C_3) to 26.0(C_8)

Following Table 3 represents some mesomorphic properties of Series A and X under comparison.

Homologous Series A is monotropically namatogenic while Series X is enantiotropically smectogenic in addition to nematogenic in character. Monotropically nematic mesophase commences lately from A_8 to A_{12} in Series A.

- Smecti and nematic properties of Series X commences earlier from C_4 and C_1 homologues, respectively.
- Smectic thermal stability of Series X is 132.86°c while Series A not having smectogenic property.

The nematic liquid crystal phase forms when the balance between the rigid and flexible parts of a molecule creates the right kind of attractive forces between molecules—either end-to-end or side-by-side. This balance comes from the molecule's structure. Because of this, molecules in the same or different series may align differently on the surface when heated, depending on how much they are affected by thermal energy. Different molecules resist thermal motion to different degrees. This resistance helps maintain either a loosely aligned (nematic) arrangement or layered structures, depending on the temperature. The ability of molecules to show liquid crystal behavior (mesomorphism) depends on the polarity and polarizability of both the molecule and its end groups. In Series A, the nematic phase starts from molecule A_8, while in Series X it starts earlier, from molecule C_1. This is because molecules in the two series have different levels of flexibility, which changes how flat (coplanar) or bent (non-coplanar) they are. The energy each molecule holds (ΔH) also changes from one to another, affecting how well it resists heat. As a result, the temperatures at which phase changes happen, the type of liquid crystal phase formed, and how stable it is all depend on the molecule's structure and energy behavior.

8. CONCLUSION

- A novel ester homologous series of twelve members (A_1 to A_{12}) are synthesized to understand the effect of heterocyclic tail on mesomorphism.
- Series A is partly monotropically nematogenic, while Series X is partly smectogenic as well as fully nematogenic in nature.
- The group efficiency order derived for smectic and nematic on the basis of
    - Thermal stability
      Series X > Series A.
    - Commencement of mesophase:
      Series X < Series A.
    - The total mesophase length:
      Series X > Series A.

- Liquid crystal properties and the degree of mesomorphism are very sensitive and susceptible to molecular structure as a consequence of molecular rigidity and flexibility.
- LC material of present investigation may be useful for dermatological treatment if studied properly by researchers working in medical or pharmaceutical fields.

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