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Mesomorphism Dependence on Terminal and Lateral Group in Azo Cinnamate Homologous Series

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ABSTRACT

A homologous series of 4-(4’-n-alkoxy-3’-methoxy cinnamoyloxy) phenyl azo-3’’-4’’-dimethyl benzene (A_1-A_{12}) has been synthesized and characterized. The primary objective of this study was to explore the correlation between molecular structure and mesomorphic behavior. All synthesized compounds, except for the shorter chain homologues (A_1, A_2 & A_3), exhibited a smectic mesophase upon cooling. The thermal transition temprature were determined using polarizing optical microscope, which yielded consistent results. The molecular structures of the compounds were confirmed through spectroscopic techniques, including ^{1}H NMR, ^{13}C NMR, FTIR, and mass spectrometry. This research provides valuable insights into the design and synthesis of novel liquid crystalline materials with tailored properties for potential applications in various fields, such as display technologies and optoelectronic devices.

Keywords: Smectic, Mesophase, Homologous, Mesomorphic, Liquid Crystal.

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1. INTRODUCTION

The Liquid crystals (LCs) have captivated researchers for decades due to their unique combination of fluid-like mobility [1] and anisotropic optical and electrical properties [2]. These fascinating materials exhibit phases intermediate between the isotropic liquid [3] and the three-dimensional [4] order of crystalline solids. The presence of both order and fluidity endows LCs with a diverse range of applications, including displays, sensors, and photonic devices [5].

The formation of liquid crystals in a material is contingent upon the precise molecular architecture and inherent properties of its constituent molecules. The newly identified molecules exhibit a distinctive structural motif. They are fundamentally constructed around a rigid core comprising three phenyl rings interconnected via -CH=CH-COO- and -N=N- linkages [6-9]. One terminus of the initial ring is appended with a flexible n-alkoxy chain (OR), while the third ring bears two methyl groups attached to the -N=N- moiety, further contributing to molecular flexibility [10-11]. This structural design strongly implies a direct correlation between the capacity of a molecule to engender liquid crystalline phases and its specific molecular architecture [12-13], specifically the interplay between rigid and flexible structural domains [14-16].

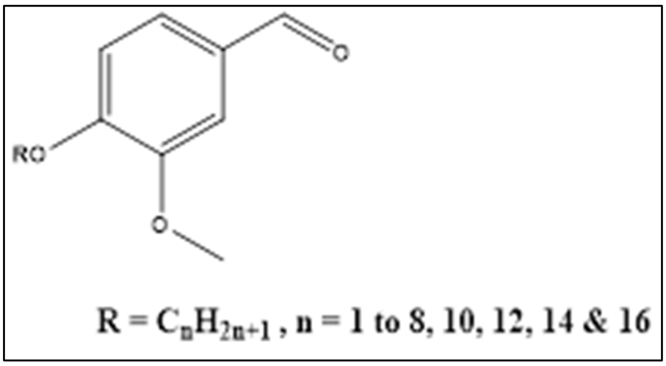
Cinnamate groups are well-known for their strong light-absorbing and photoisomerization [17] properties, offering potential for photoresponsive LC materials [18]. Alkoxy chains significantly impact mesophase stability and range [19-21], while the presence of methyl and methoxy terminal groups can further modulate molecular shape and intermolecular interactions.

This study aims to investigate the influence of these structural features on the LC phase behavior, thermal stability, and optical properties of the synthesized compounds.

2. EXPERIMENTAL

2.1. Synthesis

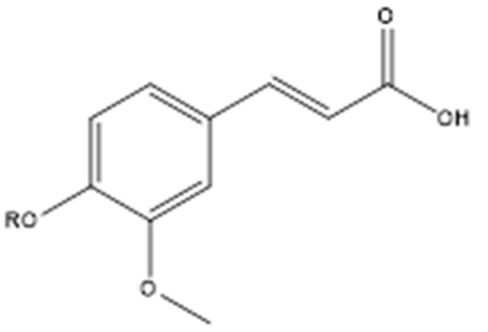
Preparation of 4-n-alkoxy-3-methoxy benzaldehyde



$R = C_nH_{2n+1}$, n = 1 to 8, 10, 12, 14 & 16

4-n-Alkoxy-3-methoxybenzaldehyde was synthesized via a Williamson ether synthesis. In this procedure, 4-hydroxy-3-methoxybenzaldehyde (1.0 equiv.) was refluxed with the appropriate n-alkyl bromide (1.5 equiv.) in the presence of potassium carbonate (1.2 equiv.) using acetone as the solvent [22]. The progress of the reaction was monitored by thin-layer chromatography (TLC). Upon completion, the reaction mixture was poured into ice-cold water, resulting in the precipitation of the desired product. The solid was collected by vacuum filtration and dried to afford the crude product, which was subsequently purified by recrystallization from ethanol for further use.

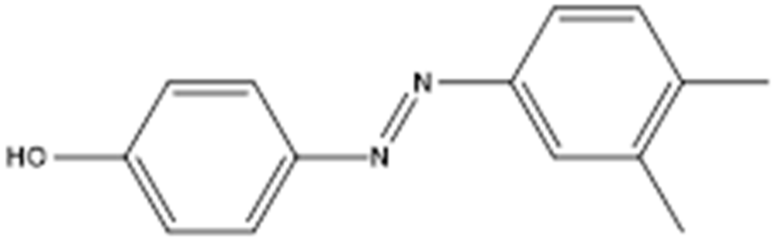
Preparation of 4-n-alkoxy-3-methoxy cinnamic acid [A-1]



$R = C_nH_{2n+1}$, n = 1 to 8, 10, 12, 14 & 16

The previously obtained 4-n-alkoxy-3-methoxybenzaldehyde (1.0 equiv.) was reacted with malonic acid (2.0 equiv.) in the presence of 1–2 drops of piperidine, using pyridine as the solvent [23]. The progress of the reaction was monitored by thin-layer chromatography (TLC). Upon completion, the reaction mixture was poured into acidic water (a mixture of distilled water and hydrochloric acid), leading to the precipitation of the product. The resulting solid was collected by vacuum filtration, and the crude product was recrystallized from ethanol to afford the purified compound for further use.

Preparation of 4-Hydroxy phenyl azo-3’,4’-dimethyl benzene [A-2]

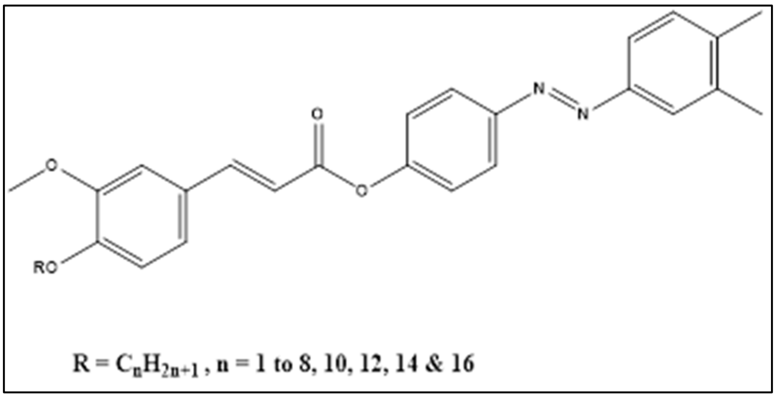


A-2

3,4-Dimethylaniline (1.0 equiv.) was placed in a 500 mL beaker, followed by the addition of a mixture of concentrated hydrochloric acid and distilled water. The resulting mixture was stirred in an ice bath using a magnetic stirrer until the solid completely dissolved, maintaining the temperature at 0 °C. A separately prepared aqueous solution of sodium nitrite was then added dropwise to the reaction mixture with caution, ensuring the temperature remained low. The reaction was stirred for an additional 1 hour to complete the diazotization [24].

In a separate vessel, phenol was dissolved in 30% aqueous sodium hydroxide solution and cooled. The cold alkaline phenol solution was then added slowly to the diazonium salt solution while maintaining a neutral pH. The reaction mixture was stirred for 2 hours at low temperature. Upon completion, orange-colored precipitates formed, indicating the formation of the azo compound. The solid product was collected by vacuum filtration and purified by recrystallization from ethanol. The final yield was 85%.

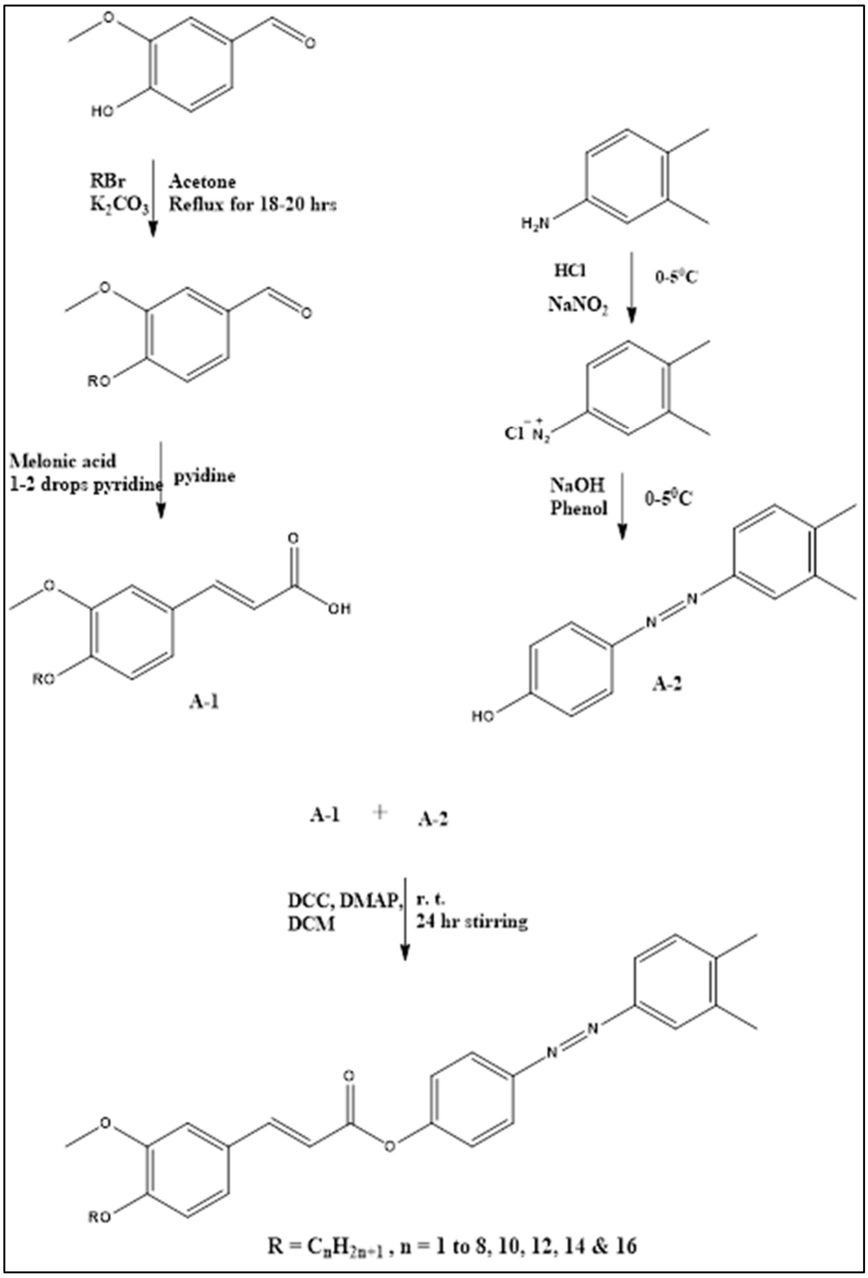
Preparation of final compound 4-(4’-n-Alkoxy-3’methoxy cinnamoyloxy) phenyl azo 3” 4” dimethyl benzene



$R = C_nH_{2n+1}$, n = 1 to 8, 10, 12, 14 & 16

4-n-Alkoxy-3-methoxycinnamic acid (A-1, 1.0 equiv.) was dissolved in an appropriate amount of dichloromethane (DCM) in a 250 mL single-neck round-bottom flask. To this solution, dicyclohexylcarbodiimide (DCC, 1.2 equiv.) and a catalytic amount of 4-dimethylaminopyridine (DMAP) were added. The mixture was stirred at room temperature for 1 hour. Subsequently, 4-hydroxy phenylazo 3’,4’-dimethylbenzene (A-2, 1.0 equiv.) was added, and the reaction mixture was stirred at room temperature for 24 hours [25].

The progress of the reaction was monitored by thin-layer chromatography (TLC). Upon completion, the reaction mixture was subjected to vacuum filtration to remove the precipitated dicyclohexylurea (DCU). The filtrate was concentrated using a rotary evaporator to remove the solvent, and the crude product was purified by recrystallization from ethanol.



Scheme 1. Synthetic route of the series.

2.2. Characterization

The few homologues of synthesized series were characterized by elemental analysis (Table 1), Infrared Spectroscopy, 1H NMR spectra, and Mass Spectroscopy. IR spectra were recorded on (Thermo scientific), 1H NMR spectra were recorded on Bruker spectrometer(400 Hz) using DMSO as a solvent, and mass spectra were recorded on Shimadzu GC-MS Model No. QP-2010.

Table 1. Elemental analysis of (1) butyloxy, (2) pentyloxy, and (3) hexayloxy derivatives.

Sr. No.	Molecular formula	Elements found %				Elements calculated %			
		C	H	O	N	C	H	O	N
1	$C_{28}H_{30}N_2O_4$	73.12	6.50	13.9	6.02	73.36	6.59	13.96	6.11
2	$C_{29}H_{32}N_2O_4$	73.65	6.73	13.49	5.78	73.71	6.83	13.54	5.98
3	$C_{30}H_{34}N_2O_4$	73.95	6.98	13.09	5.68	74.05	7.04	13.15	5.76

2.3. Analytical Data

Spectral Data

- ^{1}H NMR in ppm for octyloxy derivative (Figure 1)
  ^{1}H NMR (400 MHz, DMSO-D_6) δ 7.95(d, 2H, J = 8 Hz, middle phenyl ring) 7.84 (d, 1H, J = 16 Hz, last phenyl ring) 7.72 (s, 1H, last phenyl ring) 7.67 (d, 1H, J = 8, last phenyl ring)7.48-7.42 (m, 3H, -C$\underline{H}$=CH-COO- & 2H of middle phenyl ring) 7.39-7.33 (m, 2H, first phenyl ring) 7.02 (d, 1H, J = 8 Hz, first phenyl ring) 6.84 (d, 1H, J = 16 Hz, -CH=C$\underline{H}$-COO-) 4.01 (t, 2H, J = 6Hz, -CH_2 of -OC_8H_{17}) 3.84 (s, 3H, OCH_3) 2.34 (d, 6H, J = 8Hz, two CH_3 of last phenyl ring)1.75-1.71 (m, 2H, CH2 of -OC_8H_{17}) 1.41-1.34 (m, 10H, $(CH_2)_5$ of -OC_8H_{17}) 0.89 (t, 3H, J = 4Hz, CH_3 of -OC_8H_{17})

- ^{1}H NMR in ppm for decyloxy derivative (Figure 2)
  ^{1}H NMR (400 MHz, DMSO-D_6)δ 7.95 (d, J=8.8 Hz, 2H, middle phenyl ring near N=N) ,7.84 (d, J = 16Hz, 1H, last phenyl ring near N=N), 7.71 (s, 1H, last phenyl ring near N=N), 7.65 (d, J = 1.6 Hz, last phenyl ring near N=N) 7.47-7.42 (m, 3H, 2H of middle phenyl ring and 1H of -C$\underline{H}$=CH-COO-) 7.39-7.32 (m, 2H, first phenyl ring near -OR) 7.02 (d, 1H, J = 8.4 Hz, first phenyl ring) 6.84 (d, 1H, J = 16 Hz, -CH=C$\underline{H}$-COO-) 4.03 (t, 2H, J = 3.4 Hz, -OCH_2 of -$OC_{10}H_{21}$) 3.87 (s, 3H, -OCH_3) 2.35-2.26 (m, 6H, 2 -CH_3 of last phenyl ring) 1.76-1.69 (m, 2H, -CH_2 of -$OC_{10}H_{21}$) 1.51-1.23(m, 14H, -CH_2 of -$OC_{10}H_{21}$) 0.88-0.84(m,3H, -CH3 of -$OC_{10}H_{21}$)

- IR in cm^{-1} for propyloxy derivative (Figure 3)
  3023.6 (C-H str. of aromatic), 2936.3, 2878.6 (C-H str. of (-CH_2-) n group of - OC_7H_{15}), 1715.6 (>C=O str. of carbonyl carbon of ester group), 1627.0-1593.4 (>C=O str. of α, β unsaturated ketone), 1461.5(-C-H bending of -CH_2 alkane group), 1272.9, 1221.4 (>C=O str. of ester group), 972.6 & 689.5 (-C-H bending of alkene disubstituted). The IR data is reliable with the molecular structure.

- IR in cm^{-1} for pentyloxy derivative (Figure 4)
  3069.5 (C-H str. of aromatic), 2957.3, 2875.6 (C-H str. of (-CH_2-)n group of - OC_7H_{15}), 1716.4 (>C=O str. of carbonyl carbon of ester group), 1628.8-1593.4 (>C=O str. of α, β unsaturated ketone), 1464.8 (-C-H bending of -CH_2 alkane group), 1272.9, 1222.6 (>C=O str. of ester group), 985.3 & 689.5 (-C-H bending of alkene disubstituted). The IR data is reliable with the molecular structure.

- Mass spectra of otyloxy derivative (Figure 5)

m/z (rel.int%): 514 (M^+, 3%), 289 (100%), 225 (1%, ester linkage break), 177, 134, 105. The mass data is reliable with the molecular structure.

- Mass spectra of decyloxy derivative (Figure 6)

m/z (rel.int%): 542 (M^+, 3%), 317 (100%), 225 (1%, ester linkage break), 177, 134, 105. The mass data is reliable with the molecular structure.

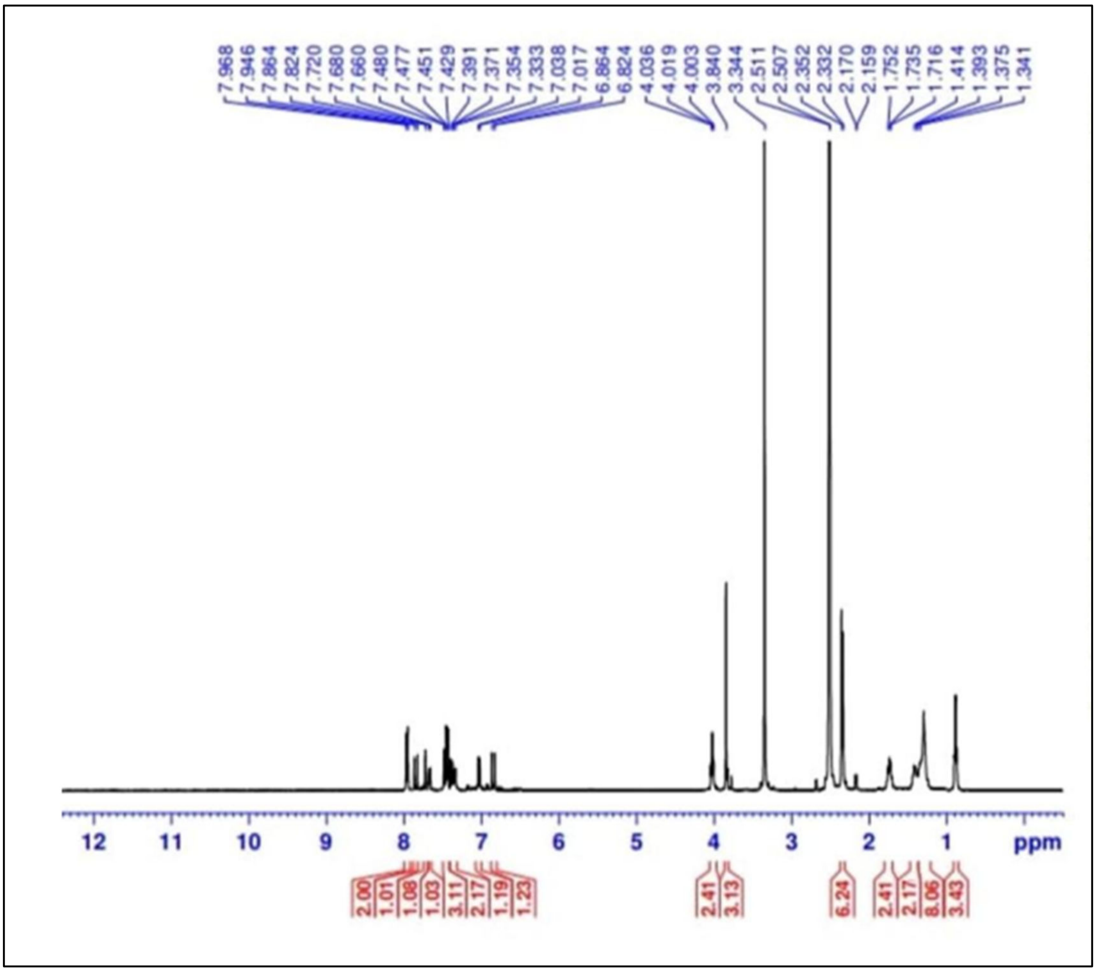


Figure 1. ^{1}H NMR in ppm for octyloxy derivative.

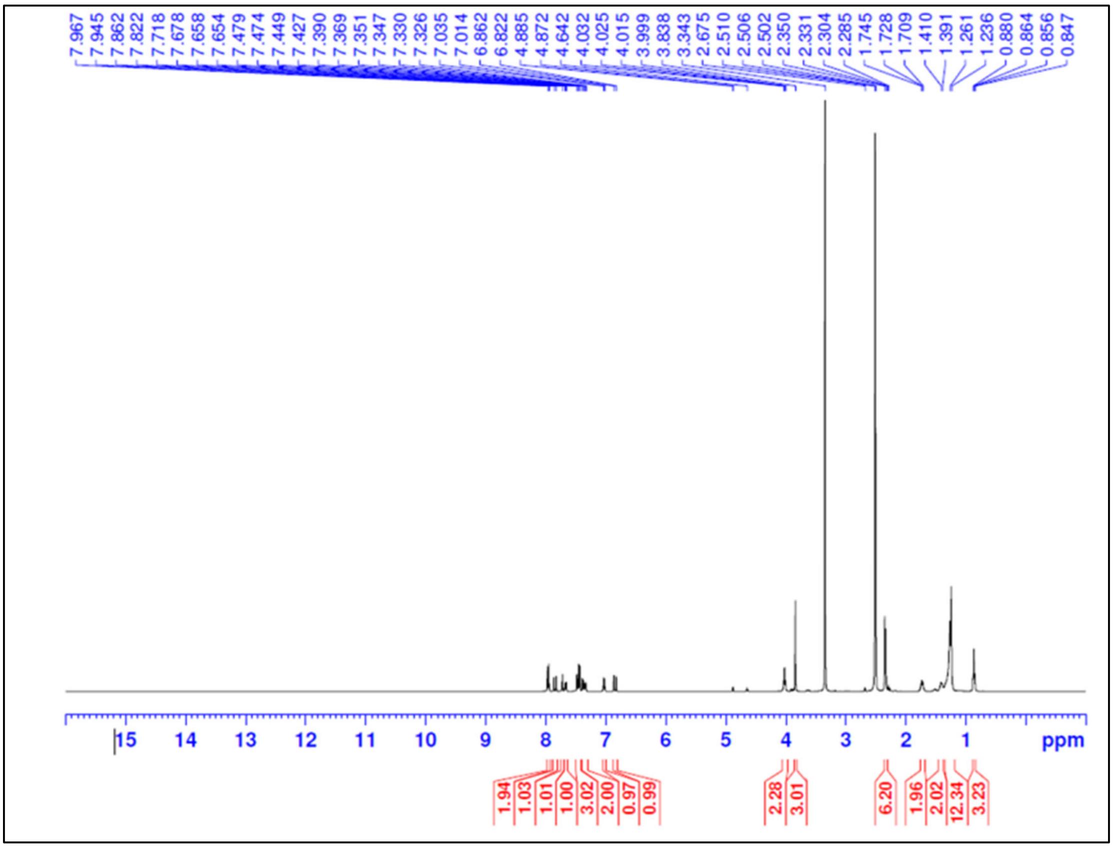


Figure 2. ^{1}H NMR in ppm for decyloxy derivative.

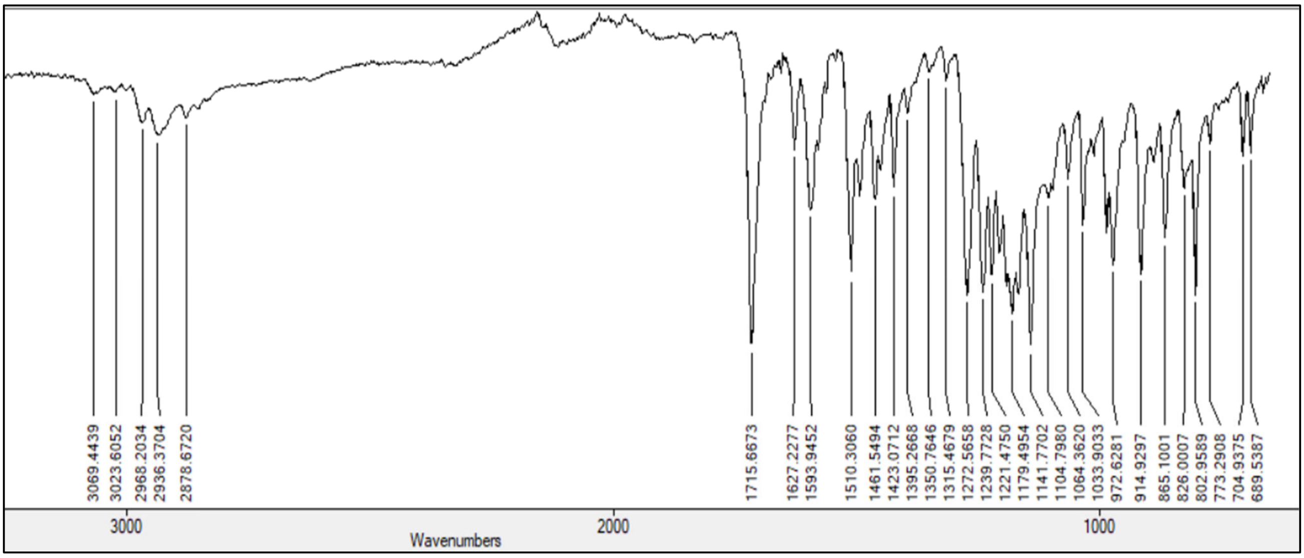


Figure 3. IR in cm^{-1} for propyloxy derivative.

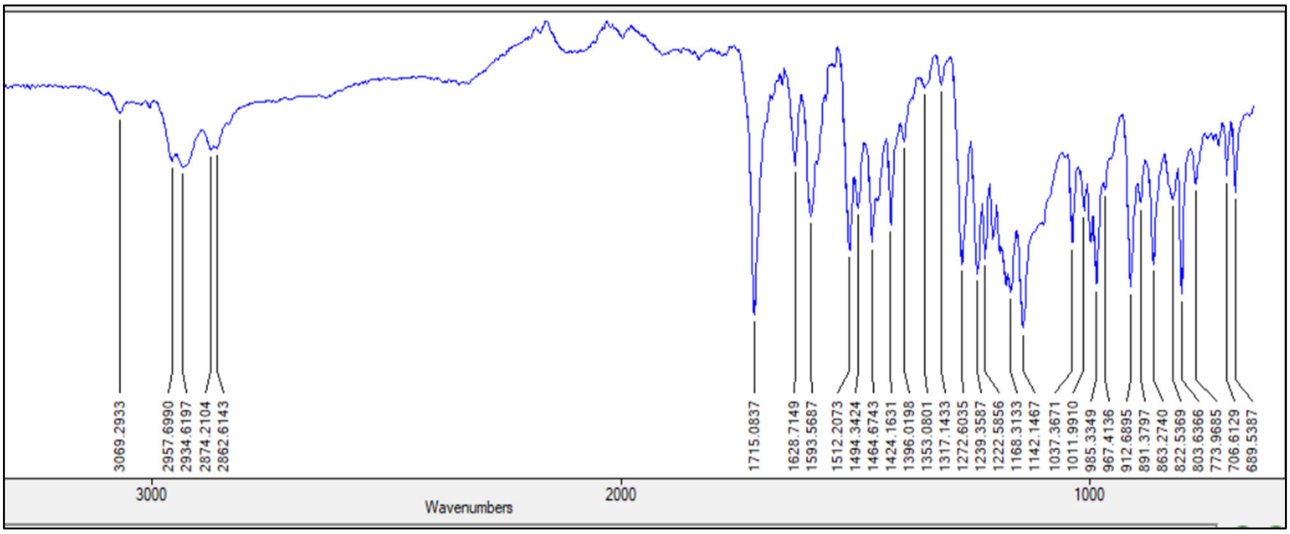


Figure 4. IR in cm^{-1} for pentyloxy derivative.

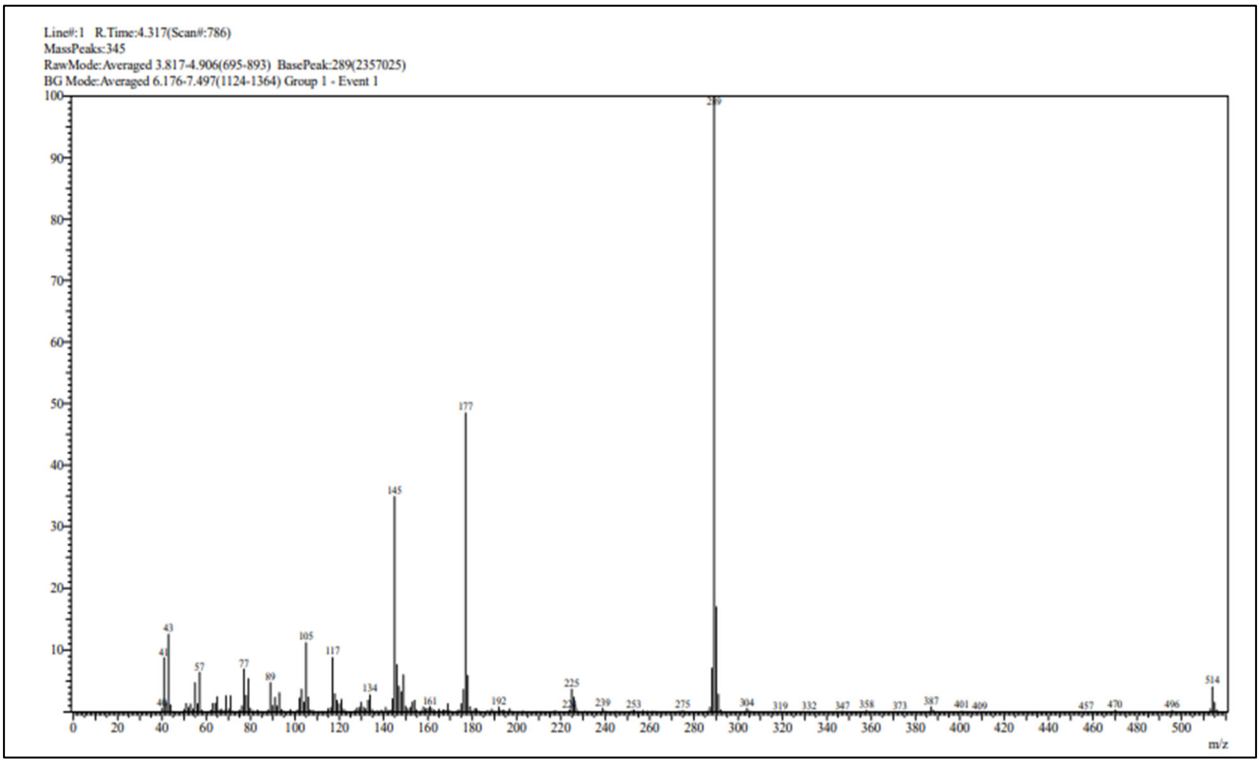


Figure 5. Mass for octyloxy derivative.

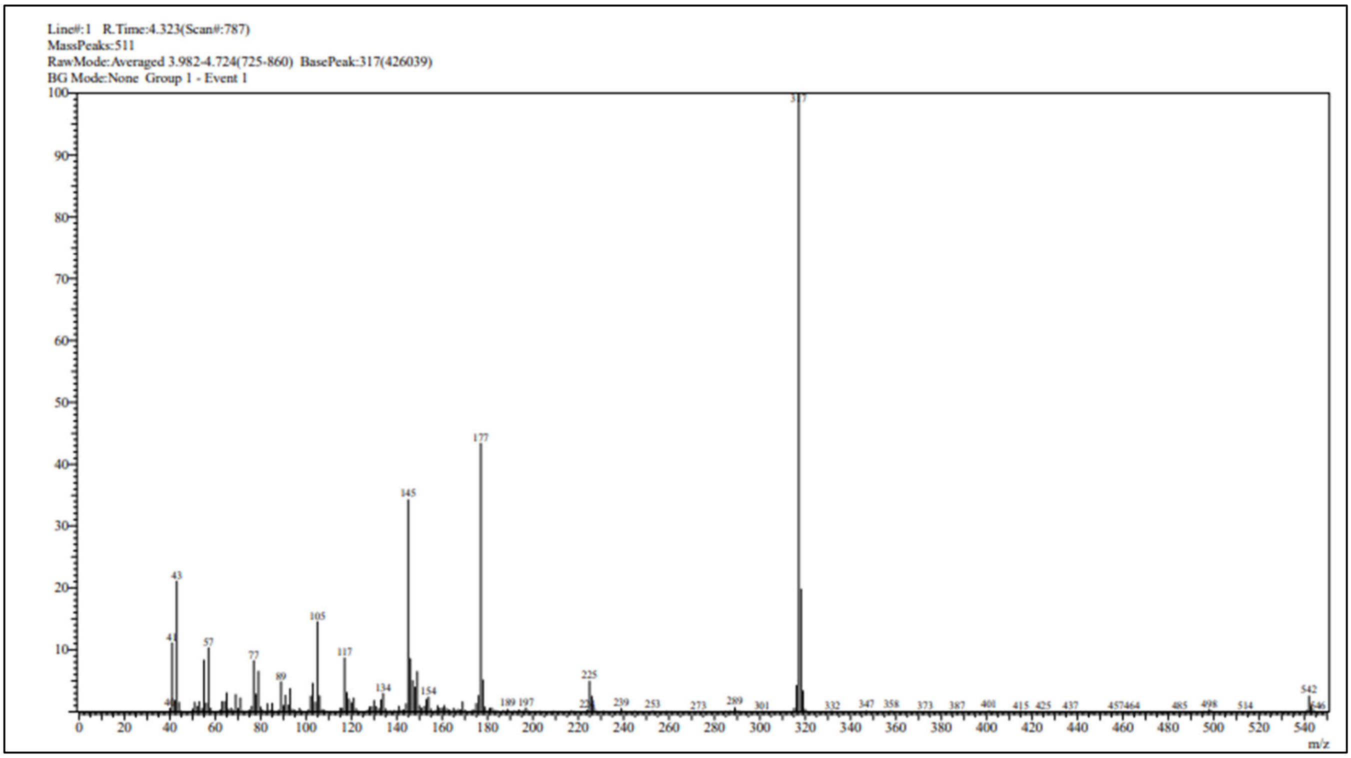


Figure 6. Mass for decyloxy derivative

3. RESULT AND DISCUSSION

A new homologous series of azo cinnamoyl esters was synthesized and found to exhibit thermotropic liquid crystalline behavior. The series comprises twelve members, designated A_1 to A_{12}. Mesophase formation begins with homologue A_4. Compounds A_4 to A_{12} exhibit monotropic smectogenic behavior exclusively.

The phase transition temperatures, determined using polarizing optical microscopy (Table 2), were plotted against the number of carbon atoms in the n-alkoxy chain at the terminal left end of the molecules (Figure 7). The resulting phase diagram displays the Cr–I and I–Sm transition curves, illustrating the mesophase behavior across the homologous series A.

The Cr–I transition curve exhibits a zig-zag pattern. With an overall decreasing trend. The I–Sm transition curve displays a relatively decreasing profile. Thus, the transition curves in the phase diagram follow typical and expected mesomorphic behavior.

The mesogenic properties vary across the homologous series depending on the number of carbon atoms in the n-alkyl chain. As a result, this new series is monotropically smectogenic upon cooling, except for homologues A_1, A_2 and A_3, which do not exhibit any mesophase. Notably, none of the compounds in the series display nematic behavior.

Table 2. Transition temperature of homologous Series A in ^{0}C.

Compound No.	R=n alkyl chain C_nH_{2n+1}	Transition temperature in ^{0}C		
		Sm	N	I
A_1	1	-	-	202.8
A_2	2	-	-	140.7
A_3	3	-	-	169.9
A_4	4	(131.2)	-	149.2
A_5	5	(100.0)	-	130.7
A_6	6	(105.4)	-	137.5
A_7	7	(97.1)	-	120.3
A_8	8	(101.4)	-	110.3
A_9	10	(97.1)	-	110.2
A_{10}	12	(93.3)	-	113.2
A_{11}	14	(93.1)	-	168.8
A_{12}	16	(88.6)	-	104.4

Where Sm = smectic, N = nematic, I = isotropic, () = monotropic

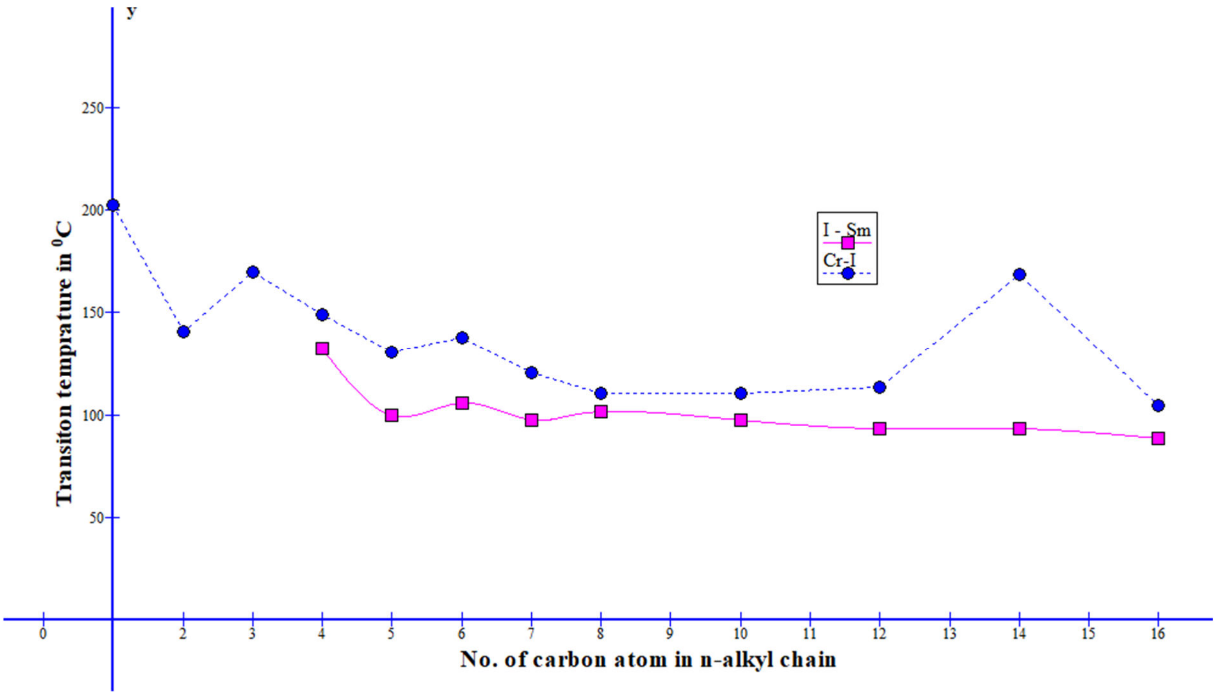


Figure 7. Phase behavior of homologous Series A.

The mesomorphic behavior observed in homologues A_4–A_{12} is attributed to the appropriate magnitude of anisotropic intermolecular forces, arising from favorable dipole–dipole interactions, molecular polarity and polarizability, molecular length, and dispersion forces. These interactions are influenced by the balance of molecular rigidity and flexibility, which in turn depend on the overall molecular geometry—such as molecular length, width, and size; the nature and position (ortho, meta, or para) of functional groups; the linearity and strength of central linkages; and the number and polarity of phenyl rings.

These structural factors collectively determine the strength of anisotropic intermolecular interactions necessary for mesophase formation. The smectogenic phase appears on cooling from the isotropic state, indicating monotropic behavior.

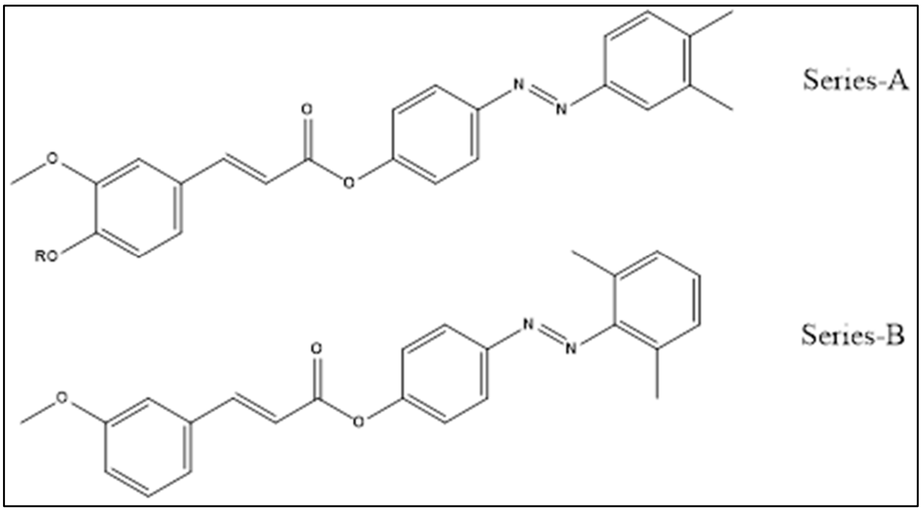


Figure 8. Structurally similar series.

The overall trend in the Cr–I transition curve is considered normal, with a gradual depression of isotropic transition temperatures from A_1 to A_{12}. This trend reflects typical behavior observed in homologous series as molecular length increases, particularly in the present series of newly synthesized azo esters.

The mesogenic properties of the newly investigated series A are compared with those of a structurally similar known analog, series B [26], as illustrated in Figure 8.

The newly synthesized homologous series A is structurally similar to series B, sharing common features such as the presence of three phenyl rings, a terminal n-alkoxy group on the left side, two methyl substituents, and central linking groups –N=N– and –CH=CH–COO– bridging the central and terminal phenyl rings. However, key structural differences exist between the two series. Series A features a lateral methoxy group on the first phenyl ring, which is absent in series B. Additionally, the positions of the two methyl groups on the third phenyl ring differ: in series A, they are located at the 3",4" positions, whereas in series B, they are substituted at the 2",6" positions.

These structural distinctions are responsible for the variations observed in the thermotropic mesogenic behavior between Series A and Series B. The differences in substitution patterns influence molecular rigidity and flexibility, which in turn affect key mesogenic properties such as the thermal stability of mesophases and the onset of mesomorphism. Consequently, the comparative mesophase behavior across corresponding homologues in Series A and Series B reflects the impact of these structural modifications.

Table 3. Relative thermal stability in ^{0}C.

Series	A	B
Smectic -Isotropic	-	127.0 B_1 - B_{13}
Commencement of smectic mesophase	A_4	B_1
Total mesophase length range (Sm)	-	6 ^{0}C (B_1) to 37 ^{0}C (B_3)

From Table 3 indicates that

- Series A exhibits smectic phases only upon cooling, whereas Series B shows smectic phases on both heating and cooling.
- The smectogenic thermal stability of the present Series A is lower than that of Series B when compared.
- The smectogenic mesophase begins at homologue A_4 in Series A, while it starts at homologue B_1 in Series B.
- The overall mesophase stability ranges, both upper and lower, for Series A are relatively lower than those observed in Series B.

The molecular structures of homologues within Series A and Series B, including the central bridges and number of phenyl rings, contribute equally to molecular rigidity. However, molecular flexibility is influenced by the structural differences between the two series. In Series A, the 3,4-dimethylphenyl ring and the methoxy group at the first phenyl ring introduce greater flexibility, while in Series B, the 2,6-dimethylphenyl ring and the absence of a methoxy group at the first phenyl ring result in differing molecular flexibility. These variations in molecular polarity and polarizability influence the anisotropic forces of intermolecular attractions, thereby affecting the formation of the liquid crystalline state.

Additionally, the positional differences of the two methyl groups on the third phenyl ring and the presence of the methoxy group at the first phenyl ring in Series A contribute to increased molecular rigidity. This enhanced rigidity leads to higher transition temperatures and, consequently, increased thermal stability in Series A. In contrast, the ortho-para substitution pattern of the methyl group in Series B disrupts the linearity of the molecule, reducing intermolecular cohesion and proximity. As a result, transition temperatures and the resistance to thermal vibrations are lower in Series B compared to Series A, even for homologues with similar structures.

Smectic mesophases begin at an earlier stage in Series B than in Series A. Additionally, the extent of molecular non-coplanarity varies between the two series for the same homologue due to differences in molecular flexibility.

A lamellar arrangement and the misalignment of molecules at an angle perpendicular to the surface are more favored in Series B compared to Series A. This indicates that the molecules in Series A are less rigid than those in Series B. Consequently, the differences in molecular rigidity, combined with their individual flexibility, lead to variations in the anisotropic forces of intermolecular attractions, both end-to-end and lateral. These variations affect the mesogenic properties, including thermal stability, the mesophase temperature range, and the commencement of smectic and/or nematic mesophases. These behaviors are observed to vary from series to series for the same homologue, as well as from homologue to homologue within the same series. Thus, the extent of molecular rigidity, planarity, or non-coplanarity influences the degrees of mesomorphism, which in turn determines the liquid crystalline (LC) properties of the compounds.

4. CONCLUSIONS

- The new homologous series A investigated in the present study is monotropically smectogenic, without exhibiting any nematic properties, and it exhibits a moderately ordered melting behavior.
- The incorporation of a lateral methoxy group at the first phenyl ring and 3,4-methyl groups at the third phenyl ring into the liquid crystal structures has led to a decrease in thermal stability across all derivatives, while still maintaining the presence of a smectic mesophase.
- The efficiency order of the groups for smectic formation is derived based on:

✓ Thermal stability

Smectic: Series B> Series A

✓ Commencement of mesophase

Smectic: Series B > Series A

✓ The total mesophase length (Sm)

Upper and lower:

Series B > Series A

- Further investigation into structural modifications and their effects could open the way for the development of more efficient and adaptable liquid crystalline materials.
- Mesomorphism and the degree of mesomorphism are highly sensitive to the corresponding molecular structure, as a result of the molecular rigidity and/or flexibility.

Acknowledgments

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