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A Synthesis and Mesophase behaviour of Homologous Series: 4-(4′-n-alkoxy cinnamoyloxy) Azo 4″-bromo-3″-methyl benzene with Terminal Methyl and bromo group

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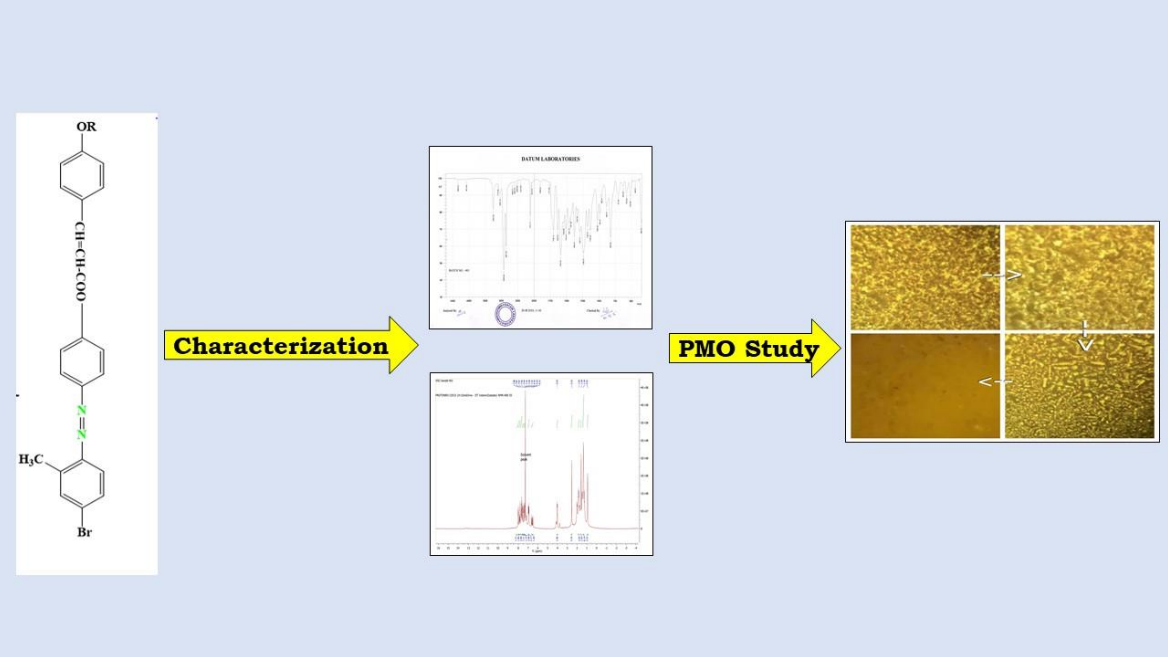

Abstract: A new homologous series 4-(4′-n-alkoxy cinnamoyloxy) Azo 4″-bromo-3″-methyl benzene was synthesized with a view to understand and establish the relation between mesogenic properties and structure of molecules. Ethyl to hexadecyl all twelve homologues are enantiotropically nematogenic. None of the homologues exhibited a smectogenic mesophase. No odd–even effect was observed in the transition curve. The average thermal stability was observed to be 127.9°C and the nematogenic mesophase ranged from 8.0°C to 16.0°C. Thus, the series was found to be of a low ordered melting type with moderate nematogenic range. Analytical data confirmed the structure of the compounds, and the mesomorphism was identified by optical microscopy. The mesogenic properties were compared with structurally similar compounds.

Keywords: Liquid crystal, smectogenic phase, nematic phase, mesophase.

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Introduction

Liquid crystals are substances that are distinguished by supramolecular, molecular mobility and organisation. Under particular conditions, this behaviour arises as the formation of phases whose unique order is somewhere between that of a three-dimensionally arranged solid and a fully disordered liquid. The following sequence is observed in simple compound: solid to liquid state whereas in liquid crystal compound: solid to liquid crystal state and liquid crystal state to liquid state. Liquid crystal phase arises by the temperature fluctuation on compound or treatment of solvent action. [1-10]

The term "thermotropic" refers to the mesophases that result from temperature change. The word "enantiotropic" refers to thermodynamically stable mesophases that emerge during both heating and cooling, whereas "monotropic" refers to thermotropic mesophases that appear only during cooling. Conversely, lyotropic liquid crystal (LC) phases are those that result from dissolving the molecule in the proper solvent at the right concentration and temperature. The compound exhibits LC state on both method (variation of temperature and solvent method) is called amphotropic liquid crystal. [11-17]

The LC materials can be categorised in a variety of ways, such as according to their molecular size, molecular shape, molecular structure and molecular type. The types of mesophase, transition temperature and mesophase length depends on above mention point (molecular size, molecular shape, molecular structure and molecular type etc.). Between the crystal and isotropic liquid states, we gets fourth stste of matters is know as liquid crystal. Polymesomorphism is the process by which materials exhibiting two or more LC phases are said to as polymesomorphic. Since there cannot be significant structural distinctions between these phases, various methods are often needed for accurate LC phase characterisation. The polarising optical microscope (POM), which displays the distinctive optical structure of a mesophase, is the tool most frequently used to determine the LC phases.

The left alkoxy (-OR) group of first rigid core and -COO- linkage present between first and second rigid is main feature for liquid crystal behaviour of present series. These types of feature present in all structures of the present series. The diazo (-N=N-) group also provides good tools for liquid crystal behaviour. The three phenyl rings in the now under investigation are connected by central bridges -COO- and -N=N- respectively and variable in the number of -CH2- units in the terminal alkyl chain.

The structure of present series was confirmed by different spectroscopic technique like Mass spectroscopy, 1H-NMR spectroscopy, IR spectroscopy. The mass spectroscopy gave data about molecular weight of corresponding compound. The 1H-NMR gave information about types of protons present in corresponding compound and functional group information was obtained by IR spectroscopy. The liquid crystal behaviour was confirmed by DSC thermal analysis and transition temperature, types of mesophase, enthalpy change and entropy change data obtain from DSC analysis. The structure of specific compound as well as DSC of specific compound of present both series was confirmed by above mention method.

Materials and methods

4-Hydroxycinnamic acid, n-alkyl bromides (CnH2n+1Br), KOH, dicyclohexylcarbodiimide (DCC), 4-dimethylamino pyridine (DMAP) were purchased from Spectrochem; 4-bromo-3-methylaniline, phenol from Merck; sodium nitrite, sodium hydroxide, pyridine, piperidine and other solvents used in synthesis from Sigma –Aldrich. 4-n-Alkoxybenzaldehyde, alkyl halides (R-X), methanol,4-Hydroxy cinnamic acid, malonic acid, K2CO3, DCC, DMAP, MDC, Acetone etc. required for synthesis were used as received except solvents which were dried and distilled prior to use. Purity of all these compounds was checked by TLC. It was accomplished on 0.2 mm pre-coated plates of silica gel G60 F254-Merck. The final compound of homologues series was characterized by elemental analysis performed on EuroEA Elemental Analyzer, mass spectroscopy was recorded on Shimadzu GC-MS Model No. QP-2010, fourier-transform infrared spectroscopy (IR) was recorded on Shimanzu FTIR-8400 and proton nuclear magnetic resonance (1H-NMR) was recorded on Brucker spectrometer using CDCl3. The thermal behaviour was recorded on Differential scanning calorimeter model no. Shimadzu, Singapore DSC-60 10.8.

Result and discussions

Synthesis of 4-n-Alkoxy Cinnamic acid (Compound A)

4-n-Alkoxybenzaldehydes were synthesized by refluxing 4-hydroxybenzaldehyde (5gm 0.036 mol) with corresponding n-alkyl bromides (1.2 Equiv) in the presence of anhydrous potassium carbonate (5.9gm 0.0432 mol) using 50ml of acetone as a solvent.[18]

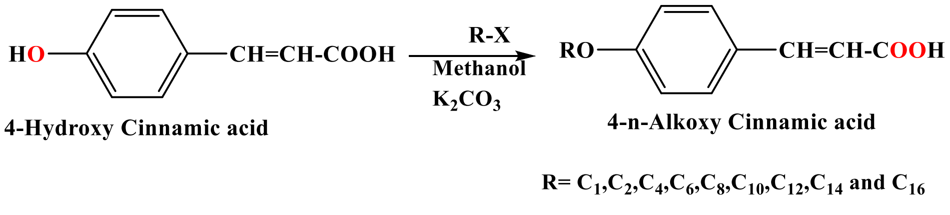
The resulting 4-n-alkoxy benzaldehydes were reacted with malonic acid (1.2 Equiv.) in the presence of 1–2 drops piperidine as catalyst and pyridine as solvent to yield 82-91% corresponding trans-p-n-alkoxy Cinnamic acids (A). [19]

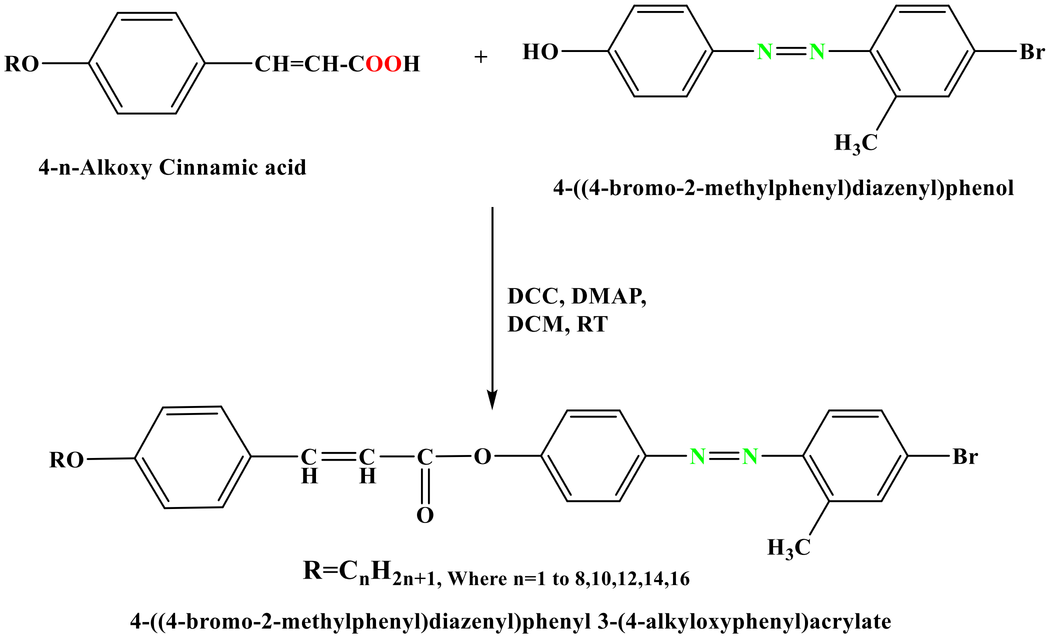
Synthesis of 4-Hydroxy phenyl azo-4″-bromo-3″-methyl benzene (Compound B)

4-bromo-3-methylaniline (4.092gm 0.022 mol) was weighed into a 500 mL beaker, and a mixture of 15 ml 50% HCl and water was added. The mixture was kept in an ice bath and stirred with a magnetic stirrer until solid dissolved. Then, NaNO2 (1.83gm 0.0265 mmol) was dissolved in water and added to the reaction mixture slowly with caution. The reaction mixture was stirred for an hour at 0 °C. Meanwhile a solution of phenol (2.046gm 0.022 mol) in 20%. NaOH was prepared and cooled at °C. Then 20ml phenol solution was added to the earlier reaction mixture at 0°C and the reaction mixture was further stirred for an hour at 0 °C. Yellow precipitate was collected at the end of the reaction, which was then filtered off using a vacuum filter and the crude product was utilized again after recrystallization in ethanol [20]. The yield was 86-90%.

Synthesis of 4-(4′-n-alkoxy cinnmyloxy) azo 4″-bromo-3″-methyl benzene (Compound AB$_1$-AB$_{12}$)

p-n-Alkoxy cinnamic acid (0.100gm 0.01mol) were directly condensed with the 4-((4-bromo-2-methylphenyl) diazenyl) phenol (0.01 mol) dissolved in 25ml DCM in portions with DCC & DMAP as catalyst by stirring reaction mixture [21]. Reaction mixture was collected filtered, washed, dried and purified, till the constant transition temperatures obtained. The yield was 72-90 %The synthetic route to the series is mentioned below in Schemes.





Scheme 1. Reagent and conditions: (1) R-Br, K_2CO_3, dry acetone, Reflux, 4-5 h; (2) malonic acid, piperidine, pyridine, reflux, 3-4 h; phenol, $NaNO_2$, HCl, NaOH, 0-5 °C; (3) DCC, DMAP, DCM, 12 hours, rt.

Spectral Data: The Synthesized compound are confirmed by using IR ^{13}C NMR and ^{1}HNMR data. The data are given below.

Compound AB$_7$

Yield 76%, brown yellow crystal, m.p. 140 °C IR,v,cm^{-1}: ~1138 & ~1180(-C-H hydrocarbon), ~1240 & ~1297 (C-H bending of alkene), ~1029(C-O str. of ether linkage), ~1437 (N=N), ~1461 ~1510 ~1546 ~1593 (aromatic ring), ~1771 & ~1705 (-COO, ester linkage), ~2922and ~2856 (-C-H str),C-Br~520, and ~707 (p-di substituted phenyl ring), and ~962 & ~825 poly(-CH_2-)n. ~3044 ~3110(CH=CH). ^{1}H NMR, ppm: δ 8.10 (s, 1H), 8.02 (dd, J = 17.8, 8.6 , 2H), 7.92 – 7.80 (m, 2H), 7.70 (d, J = 8.9, 2H), 7.64 (d, J = 8.9, 2H), 7.57 (d, J = 8.4 , 1H), 7.45 (d, J = 8.3, 1H), 7.36 (d, J = 8.5, 2H), 7.17 (d, J = 8.7, 1H), 6.93 (dd, J = 17.5, 8.3, 3H), 6.63 (d, J = 15.3, 1H), 6.53 (d, J = 15.9, 1H), 4.12 (t, J = 12.4, 1H), 4.02 (dt, J = 12.1, 6.6, 3H), 3.77 (d, J = 9.5, 1H), 2.54 (d, J = 6.1, 4H), 2.06 (d, J = 12.1, 0H), 2.00 (d, J = 13.2, 3H), 1.87 (d, J = 8.8, 2H), 1.84 (d, J = 7.1, 2H), 1.80 (d, J = 7.2, 1H), 1.77 (s, 1H), 1.76 – 1.70 (m, 1H), 1.47 (p, J = 9.1, 8.1, 3H), 1.34 (s, 9H), 1.23 (dd, J = 24.4, 12.1, 4H), 0.92 (s, 4H), 0.92 (d, J = 14.8, 1H), 0.53 (s, 8H). ^{13}C NMR, ppm: δ 164 (1C), 158 (1C), 151 (1C), 147 (1C), 145 (1C), 137 (1C),134 (1C), 131 (2C), 129 (1C), 128 (1C), 126 (2C), 124 (2C), 119 (1C), 115 (1C), 110 (2C), 108 (1C), 70 (1C), 35 (1C), 28 (1C), 26 (1C), 22 (1C), 19 (1C), 20 (1C). % Found: C 73.40; N 7.68; H 5.19. % Calculated: C 73.53; N 7.79; H 5.29.

Compound AB_{12}

Yield 79%, brown yellow crystal, m.p. 89 °C IR,V, cm^{-1}: ~1142(-C-H hydrocarbon), ~1213 & ~1257 (C-H bending of alkene), ~1030(C-O str. of ether linkage), ~1426 (N=N), ~1476 ~1505 ~1576 ~1611 (aromatic ring), ~1723 (-COO, ester linkage), ~2921and ~2854 (-C-H str),C-Br~513, and ~719&829 (p-di substituted phenyl ring), and ~996 & ~889 poly(-CH2-)n. ~3186(CH=CH). 1H NMR ,(ppm): 8.02 (dd, J = 17.6, 8.7, 2H), 7.92 – 7.80 (m, 3H), 7.72 (dd, J = 14.4, 8.5, 2H), 7.64 (d, J = 9.1, 2H), 7.57 (d, J = 8.5, 2H), 7.36 (d, J = 8.5, 2H), 7.19 (t, J = 13.0, 1H), 6.96 (d, J = 8.3, 2H), 6.53 (d, J = 15.9, 1H), 4.03 (t, J = 6.6, 2H), 2.54 (d, J = 5.9, 6H), 1.83 (d, J = 14.5, 1H), 1.48 (q, J = 7.4, 2H), 1.34 (s, 8H), 1.29 (d, J = 6.5, 9H), 0.91 (t, J = 6.7, 3H). ^{13}C NMR, ppm: δ 165 (1C), 159 (1C), 150 (1C), 148 (1C), 147 (1C), 145 (1C), 137 (1C), 132 (1C), 130 (3C), 127 (1C), 126 (1C), 124 (2C), 121 (2C), 117 (1C), 116 (1C), 114 (2C), 68 (1C), 31 (1C), 29-30 (4C), 28(3C), 26(1C), 22 (1C), 20 (1C), 14 (1C). % Found: C 74.30; N 7.39; H 5.21. % Calculated: C 74.39; N 7.54; H 5.66.

POM and DSC investigation

Transition temperatures were observed through a polarizing microscope with heating stage as recorded in Table 1. Textures of homologues are confirmed by miscibility method. The enthalpy change (ΔH), entropy change (ΔS) concept discussed qualitatively due to inadequate facility available to the source, otherwise quantitatively ΔH and ΔS values would have been determined from the peak value temperature of DSC scan.[22-26] In this series most of the compounds showg two type of phases. First compound converts solid to nematic phase than after nematic goes in isotropic phase. The phase behaviour texture of AB9 (decyloxy) captured during POM investigation was showed in Figure 1. In the DSC analysis, two compounds AB12 was selected for spectral analysis. DSC spectra showed one peaks which indicates nematic to isotropic conversion mesophase present in molecules. Compound AB12 showed peak at 133 oC for the conversion of crystal to nematic phase (Figure 2).

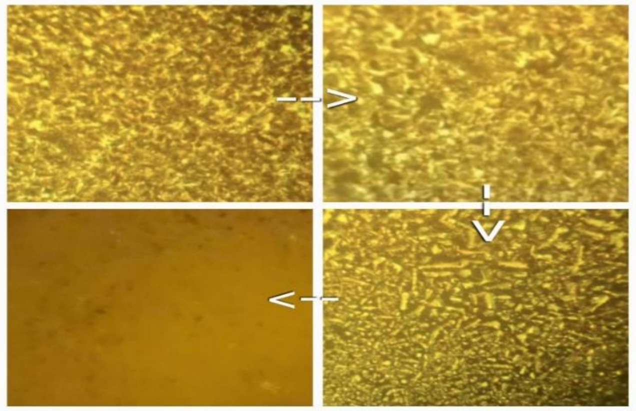


Fig.1. POM images of compound AB12: solid (a); Nematic phase (b); sematic phase (c).

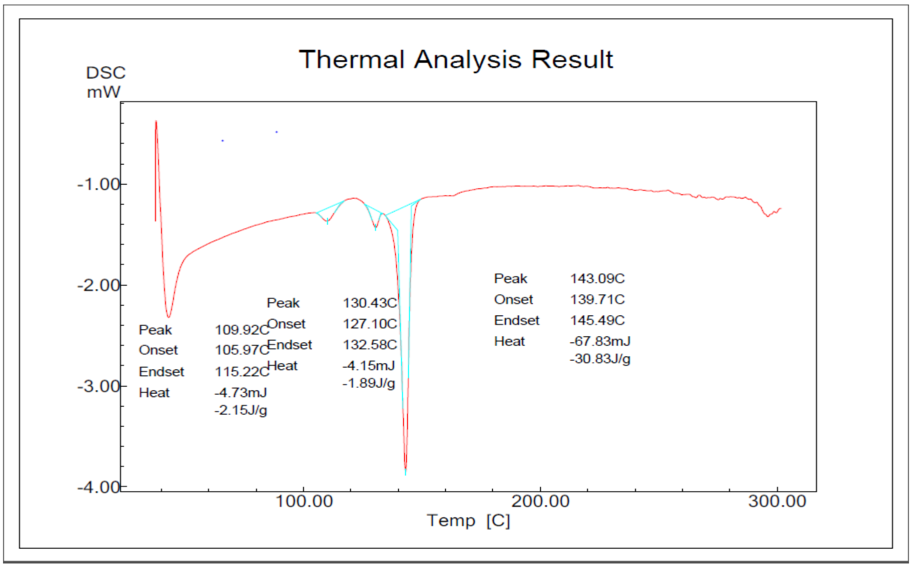
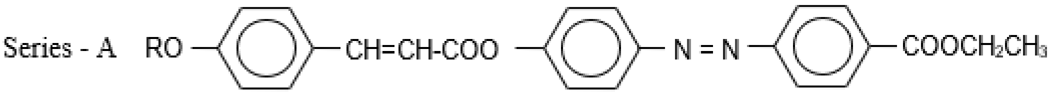


Fig.2. DSC data of compound AB7 (Heptyloxy)

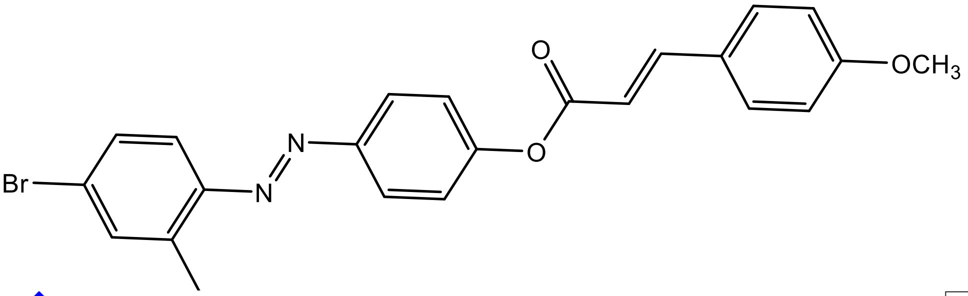
Table 1. Transition temperatures

Sr No.	n-Alkyl Group	Transition temperatures in ºC		
		Smectic	Nematic	Isotropic
1	Methyl	-	-	170 ºC
2	Ethyl	-	-	-
3	Propyl	-	-	-
4	Butyl	-	144 ºC	160 ºC
5	Pentyl	-	-	-
6	Hexyl	-	136 ºC	152 ºC
7	Heptyl	-	128 ºC	140 ºC
8	Octyl	-	121 ºC	132 ºC
9	Decyl	-	106 ºC	116 ºC
10	Dodecyl	-	89 ºC	100 ºC
11	Tetradecyl	-	81 ºC	92 ºC
12	Hexadecyl	-	81 ºC	89 ºC

Series – A



Series – 1



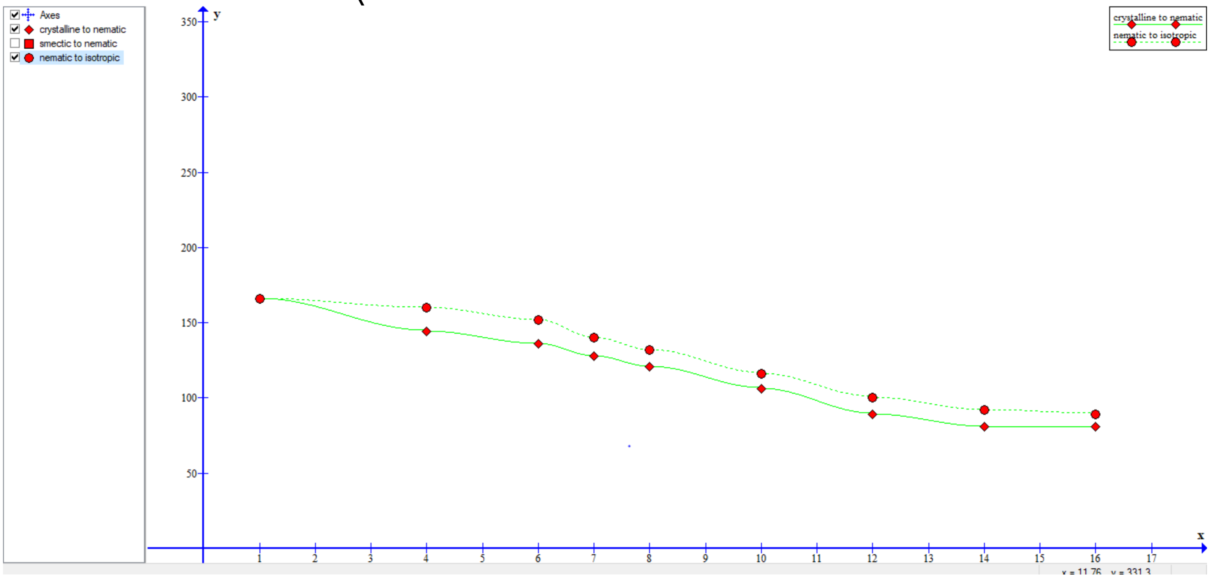


Fig.3. Phase behaviour of compound AB_1-AB_{12}: No of carbon in alkyl chain vs Temperature

Homologous series of 4-(4′-n-alkoxy cinnamoyloxy) azo 4″-bromo-3″-methylbenzene is entirely mesomorphic in character. All the members of the series display mesomorphism in enantiotropic manner with enough range of liquid crystallinity. All the homologues display mesomorphism of nematic type without exhibition of smectic mesophase. Transition temperatures of the homologues are plotted versus the number of carbon atoms in n- alkyl chain of left n-alkoxy terminal as given in figure-3. Smooth curves are drawn through like or related points. The solid-mesomorphic transition curve follows a linear path of rising and falling nature. The nematic –isotropic transition curve exhibits linear tendency as the series is ascended except at the hexadecyl homologue of the series in which nematic-isotropic transition curve abnormally behaves otherwise nematic isotropic transition curve behaves in a normal manner. Well known odd-even effect is observed in the nematic-isotropic transition curve with alternation of transition temperatures and merges into each other at the nineth homologue. The texture of nematic mesophase is threaded and drop late type as clearly judged from the field of view of hot stage polarizing microscope while observing the samples. The mesomorphic-isotropic transitions are between 89.0 oC and 170.0 oC with mesomorphic range varying from 8.0 oC at the hexadecyl homologue to a maximum of 16.0 oC at the methyl homologue of the series Table-2. Thus, the present homologues series is considered as low ordered melting type with wide range of liquid crystallinity. Formation of sliding layered arrangement of molecules does not occur in crystal structure which resulted in absence of smectic mesophase for all the homologue. Terminal –CH3&Br is less polar group which contributes to the weaker intermolecular end to end attractions. Thus, all the members of the series are enantiotropic nematic in character. Ester group is generally nematogenic and present homologous series is also entirely nematogenic. Solid nematic transition curve rises and falls in linear manner.

Average thermal stability and stage of commencement of mesophase formation are recorded in Table 2 as under.

Table 2. Average thermal stability

Series	**Series-1**	**Series-A**
Nematic - isotropic °C	293.66 °C	127.9 °C
Commencement of nematic	(C_1 – C_{16})	(C_1 – C_{16})
Phase	C_1	C_1
Total mesophase length in °C (Nm-Iso)	10°C - 130°C	8.0°C – 16.0°C
Cn_1 Cn_2	(C_{16} – C_1)	(C_{16} – CS_4)

The present homologues series (1) is compared with structurally similar other homologous series (A) [27]. for series 1 molecular characteristics and thermal stabilities which are shown in Table 2. The above homologous series (1) and (A) under discussion have the basic length due to three phenyl rings linked through central linking units –CH=CH-COO- and –N=N-, left n-alkoxy group and right terminal –Br functional group at para position and CH3 group in ortho position. Hence, display of mesomorphic properties due to the molecular forces arising on account of these remains the same. The homologous series (1) and (A) differ only at terminal grops part of the molecules. terminal part is -Br and -CH3 for series (1) while respect to –CH3CH2COO- for series (A). Hence, the variation in mesomorphic characteristics has direct relation with central bridges. The length to breadth ratio is diminished for series (1) as compared to series (A). Broadening of molecules increases intermolecular distance and hence it nearly the intermolecular forces of attractions on one hand while broadening of molecule increases polarizability of molecules and hence results in increase of intermolecular forces of attractions. In case of series (1) as compared to series (A) melting and transition temperatures of titled homologous series (1) are relatively nearly low than series (A). This is also reflected in relative thermal stability of series (1) and (A).

Therefore, nematic-isotropic thermal stability for series (1) is nearly low than series (A). Thus, nematic group efficiency order with respect to type of linking terminal i.e. last part positional substitution is as under on the basis of average thermal stability.

Conclusions

In summary, we have synthesized azo ester based homologous series (AB_1-AB_{12}) by veering twelve alkyl chains on terminal of moieties. All compound were prepared with good yield by esterification in final step. All derivative were confirmed by FTIR and NMR analysis. Liquid crystalline behaviour of compounds was examined by POM and confirmed by DSC analysis. Titled homologous series is entirely nematogenic with short range of liquid crystallinity exhibition phase. Present investigation supports the earlier view [27] and raises credibility to the established views derived earlier. Study suggested that this mesogens could be useful for further investigation and fabrication of LCs.

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