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# A Synthesis and Mesophase Behaviour of Homologous Series: 2-formyl-4-((E)-(4-((E)-Phenyldiazenyl)phenyl-4-alkyloxybenzoate

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## ABSTRACT

A new homologous series synthesis of 2-formyl-4-((E)-(4-((E)-Phenyldiazenyl)phenyl-4-alkyloxybenzoate is 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 exhibit a smectogenic mesophase. An odd–even effect is observed in the transition curve. The average thermal stability is 167.0°C and the nematogenic mesophase ranges from 10.0°C to 15,0°C. Thus, the series is of a middle ordered melting type with moderate nematogenic range. Analytical data confirm the structure of the compounds, and the mesomorphism was identified by optical microscopy. The mesogenic properties are compared with structurally similar compounds.

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

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## 1. INTRODUCTION

At the intriguing nexus between conventional liquids and solid crystals, liquid crystalline substances exhibit special qualities derived from their molecular makeup. These materials are characterised by intermediate states between liquids and solids, and their molecular structure has a significant impact on how they behave. Mesomorphic characteristics, which are essential for a variety of applications, can be significantly altered by even small changes in molecule geometry [1–3].

Novelly structured liquid crystal molecules have attracted a lot of attention as a result of developments in liquid crystal science and technology. A few examples are molecules having a multi-arm structure [4-5], as well as unusual shapes as hockey stick shapes [10-11], 'H' shapes [6], 'V' shapes [7], 'S' shapes [8], 'Y' shapes [9], and 'T' shapes [10]. These distinctive structures provide up new options for the behaviours and applications of liquid crystals.

In addition to polar phases like those found in bent-core liquid crystals (BCLCs), hockey-stick mesogens can also create conventional mesophases like those generated by rod-like molecules, like nematic, smectic A (SmA), or smectic C (SmC) phases [12–13].

Due to their distinct optical, thermal, and mechanical characteristics, these compounds' core structures containing azo and azomethine links have garnered a lot of interest [14–16]. These connections give the liquid crystals unique properties that make them more appropriate for a variety of uses in cutting-edge materials and technology [17].

The presence of a –N=N– bond inside their chemical structure is a characteristic of azo compounds. When exposed to light, this azo group enables reversible photoisomerization, in which the molecule alternates between various geometric forms [18–19]. For instance, when exposed to UV light, the planar trans-azobenzene structure can change into the bent cis-isomer [20–22]. This characteristic is particularly useful for creating photo-responsive materials, which have important uses in optical information processing, optical holography, and optical storage [23–26]. However, as reported for photoexcited azo-dye-induced torque in nematic liquid crystals, molecules containing the –N=N– group have been found to favour the trans conformation in the mesophase [27].

## 2. MATERIALS AND METHODS

4-hydroxybenzaldehyde, piperidine, pyridine, alkyl bromides, anhydrous $K_2CO_3$, alkyl bromides (R-Br), phenol, *p*-amino benzoic acid, HCl, NaOH, $NaNO_2$, DCC, DMAP and 4-hydroxy-3 methoxybenzaldehyde (vanillin) were purchased from Avra Chemical, India. Acetone and MDC solvents were purchased from Finar and further purified by usual established method. TLC plates (silica gel 60 F254 silica-aluminum plates) ware purchased from Merck. FT-IR spectra was carried out in KBr pellet method and further analysed in the range of 3800-560 $cm^{-1}$by Bruker TENSOR 27. $^1$H and spectra: The spectra were recorded on a Bruker Advance (400 MHz), in $CDCl_3$ Solvents where TMS is internal standard. The mesophase is identified by Polarizing Optical Microscope (Nikon Eclipse LV-100 POL) with temperature controlled heating stage.

## 3. RESULT AND DISCUSSIONS

### 3.1. Synthesis of 4-*n*-Alkox Benzoic Acid (Compound A)

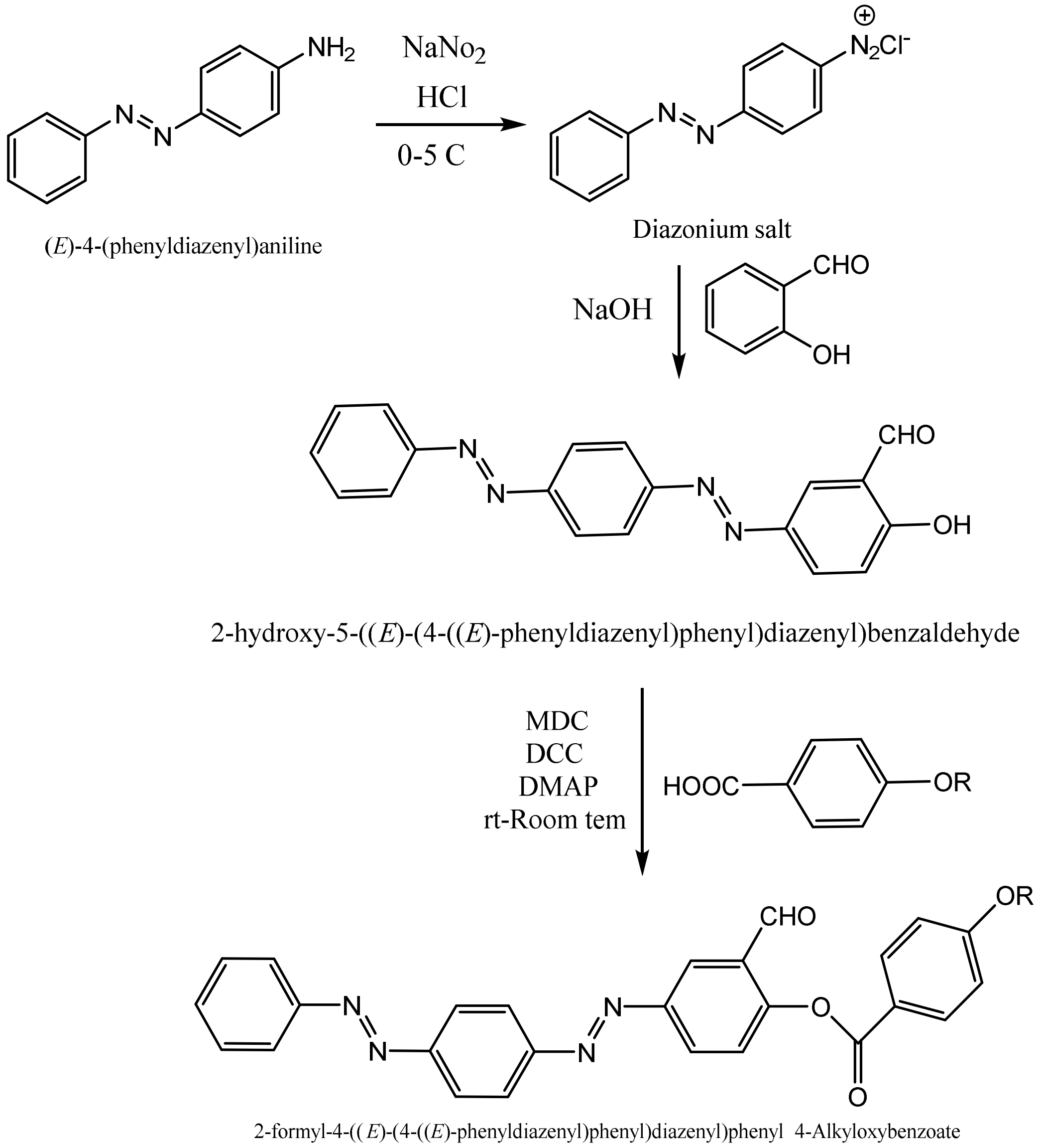
4-*n*-Alkoxybenzoic acid were synthesized by refluxing 4-hydroxybenzoic acid (1 equiv.) with corresponding *n*-alkyl bromides (1 equiv.) in the presence of anhydrous potassium carbonate (1.2 equiv.) using acetone as a solvent.[26] The resulting 4-*n*-alkoxy benzoic acid was purify by crystallization. (A).[27]

### 3.2. Synthesis of 2-hydroxy-5-((E)-(4-((E)-phenyldiazenyl)phenyl)diazinyl)benzaldehyde (Compound B)

2-hydroxy-5-((E)-(4-((E)-phenyl diazenyl) phenyl) diazinyl) benzaldehyde (B) was prepared by previously established method.[28] The diazotization reaction is performed on (E)-4-(phenyldiazenyl) aniline with salicylaldehyde by using $NaNO_2$, HCl, and NaOH in an ice bath. The mass volume of the reaction was vigorously mixed for 2 hours. At that point, the azo dye product (B) conformed by using starch iodide and congo red paper, showing the positive test by turning to blue.

### 3.3. Synthesis of 2-formyl-4-((E)-(4-((E)-Phenyldiazenyl)phenyl-4-alkyloxybenzoate (Compound $AB_1$-$AB_{12}$)

4-*n*-alkoxy benzoic acid were directly condensed with the 2-hydroxy-5-((E)-(4-((E)-phenyl diazenyl) phenyl) diazinyl) benzaldehyde by the reported method using DCC reagent and DMAP as catalyst in MDC solvent. The reaction was than stirred at room temperature for 12 hours.[29] The reaction compilation was confirmed by using thin layer chromatography. Further, the product was purified. The synthetic route to the series is mentioned below in **Scheme 1.**



**Spectral Data:** Analytical data of some selected representative homologues, viz; elemental analysis (Table 1), IR and $^1$H NMR data support the structure of molecules.

**Compound $AB_{10}$; $^1$H NMR δ (ppm):** 0.92 (t, 3H, $-CH_3$ of $–OC_8H_{17}$), 1.59 (m, 12H, $-CH_2-$ of alkyl chain), 1.86 (m, 2H, $-CH_2$ of alkyl chain), 2.20 (m, 2H, $-CH_2-$ of alkyl chain), 4.10 (t, 2H, $-OCH_2-$ of alkyl chain), 7.05 ( 2H, of Ar-H), 7.57 (dd, 4H, Ar-H), 8.00 (m, 2H, Ar-H), 8.13 (dd, 4H, Ar-H), 8.21 (m, 2H, Ar-H), 8.30 (m, 1H, Ar-H), 8.58 (s, 1H of CH=N-), 10.35 (s, 1H of -CHO). **IR in cm$^{-1}$:** 687 ( substitute aromatic ring ), 764 (aromatic ring), 852 poly($-CH_2-)_n$ group), 912 (*p*-di substituted benzene ring),, 1056-1091 (-C-H hydrocarbon), 1187 (C-N), 1224 (ether linkage), 1407 (-N=N-), 1476-1601 (C=C- of aromatic ring), 1691 (C=O of CHO), 1730 (-COO), 2855 ( doublet of CHO), 2923-3069 (-C-H str).

**Compound $AB_{12}$; $^1H$ NMR δ (ppm) :** 0.90 (t, 3H, $-CH_3$ of $–OC_8H_{17}$), 1.56 (m, 12H, $-CH_2-$ of alkyl chain), 1.86 (m, 2H, $-CH_2$ of alkyl chain), 2.20 (m, 2H, $-CH_2-$ of alkyl chain), 4.10 (t, 2H, $-OCH_2-$ of alkyl chain), 7.05 ( 2H, of Ar-H), 7.55 (dd, 4H, Ar-H), 8.09 (m, 2H, Ar-H), 8.12 (dd, 4H, Ar-H), 8.22 (m, 2H, Ar-H), 8.30 (m, 1H, Ar-H), 8.56 (s, 1H of CH=N-), 10.35 (s, 1H of -CHO). **IR in $cm^{-1}$:** 685 ( substitute aromatic ring ), 764 (aromatic ring), 847 poly($-CH_2-)_n$ group), 911 (*p*-di substituted benzene ring),, 1010-1056 (-C-H hydrocarbon), 1170 (C-N), 1229 (ether linkage), 1475 (-N=N-), 1509-1600 (-C=C- of aromatic ring), 1694 (C=O of CHO), 1740 (-COO), 2856 ( doublet of CHO), 2924-3062 (-C-H str).

**Table 1.** Elemental analysis for compound $AB_{11}$ and $AB_{12}$

| Sr. No. | Molecular Formula | Elements % Found (% Calculated) | | |
|---|---|---|---|---|
| | | C | H | N |
| 1. | $C_{36}H_{38}N_4O_4$ | 73.35% (68.48%) | 6.92% (5.90%) | 9.59% (6.97 %) |
| 2. | $C_{38}H_{42}N_4O_4$ | 73.93% (69.97%) | 6.59% (6.18%) | 9.61% (7.37 %) |

### 3.4. POM and DSC Investigation

Transition temperatures are observed through a polarizing microscope with heating stage as recorded in **Table 2**. 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.[30] In this series most compound showing only one type of phase. First compound converts solid to nematic phase than after compound goes in isotropic phase. The phase behaviour texture of $AB_8$ & $AB_{10}$ captured during POM investigation was showed in **Figure 1**. In the DSC analysis, of compounds $AB_{12}$ was selected for spectral analysis. DSC spectra showed only one peaks which indicates for nematic is convert into isotropic. This conversion indicate that mesophase present in molecules. Compound $AB_{12}$ showed peak at 138 °C for the conversion of crystal to nematic phase and no other peak observe at other tempreture indicate the smectic phase is not present **(Figure 2)**.

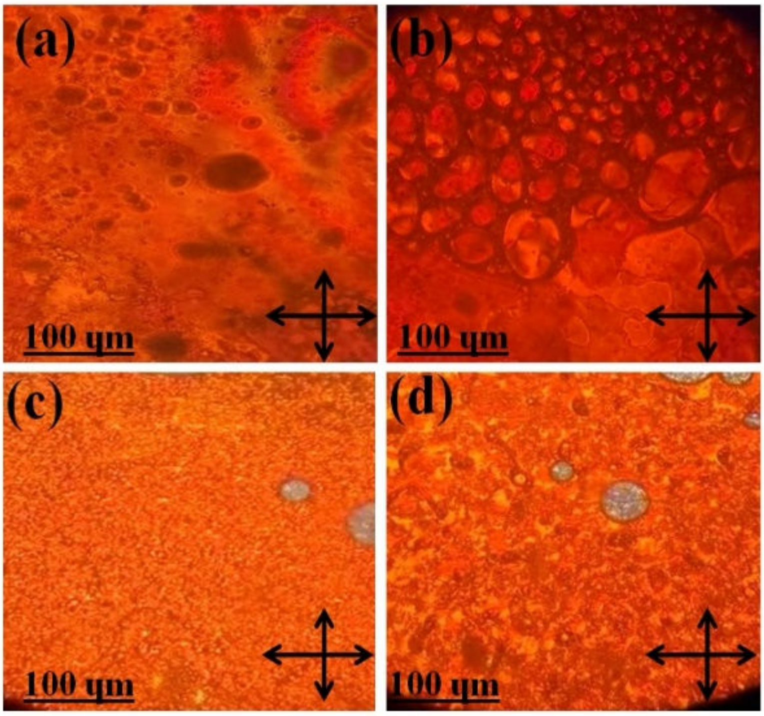
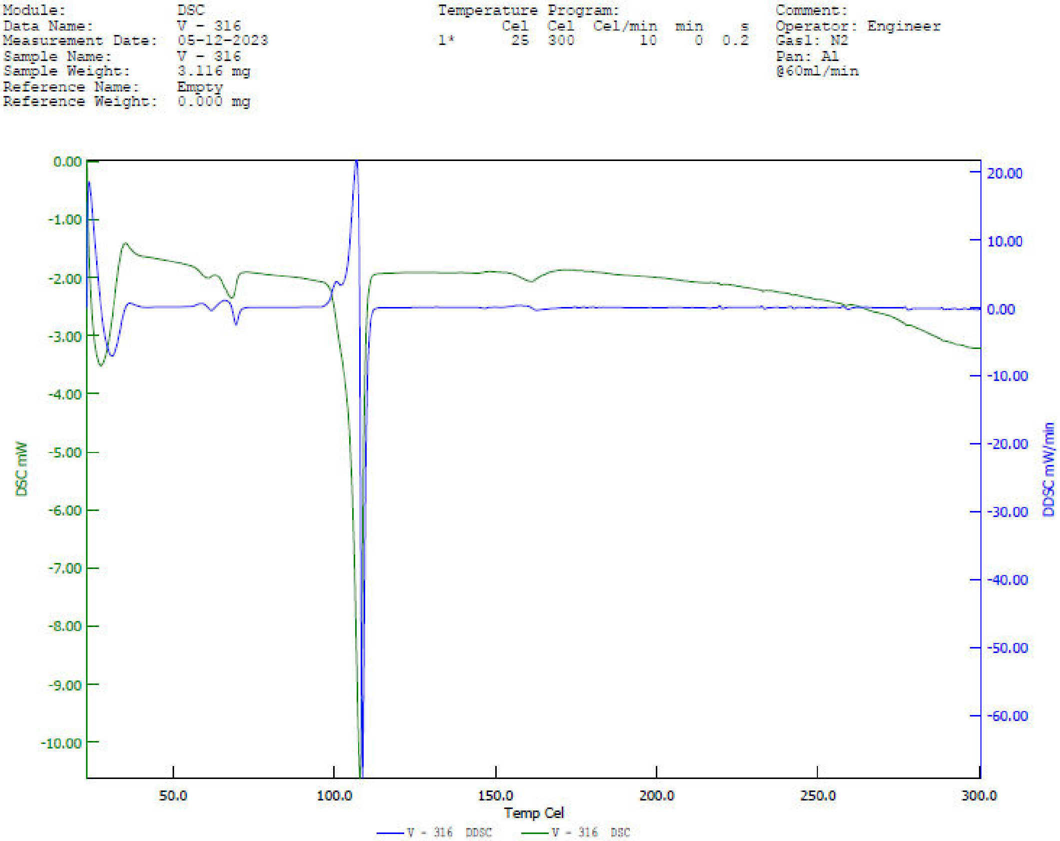


**Figure 1**. POM images of compound $AB_8$: (a), (b);and $AB_{10}$ (c),(d).



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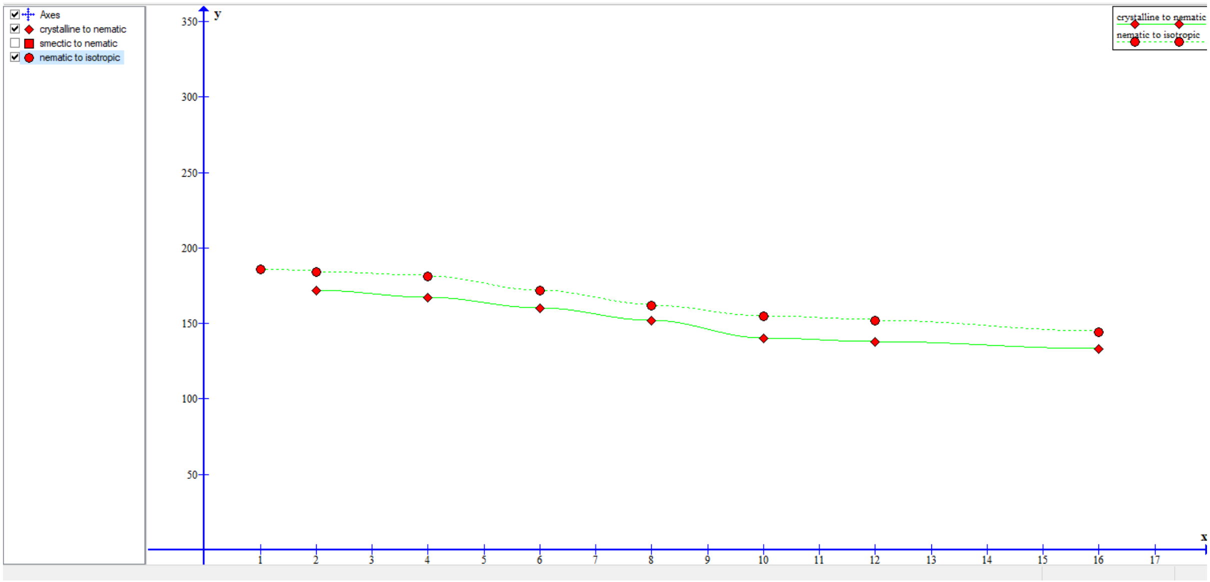
Module:              DSC                    Temperature Program:                    Comment:
Data Name:           V - 316                       Cel  Cel  Cel/min  min    s      Operator: Engineer
Measurement Date:    05-12-2023             1*      25  300       10    0  0.2      Gas1: N2
Sample Name:         V - 316                                                        Pan: Al
Sample Weight:       3.116 mg                                                       @60ml/min
Reference Name:      Empty
Reference Weight:    0.000 mg
```

**Figure 2.** DSC data of compound $AB_{12}$ (Dodecyloxy).

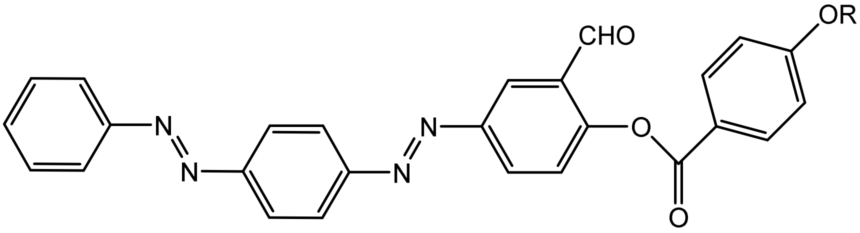
Homologous series 2-formyl-4-((E)-(4-((E)-phenyldiazenyl) phenyl-4-alkyloxybenzoate is entirely mesomorphic in character. All the members of the series display mesomorphism in enantiotropic manner with enough range of liquid crystallinity. All the homologues display mesomorphism of nematic type mesophase. 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 parallel path of rising and falling nature. The nematic –isotropic transition curve exhibits first rises and falling tendency. nematic isotropic transition curve behaves in a normal manner. Well known odd-even effect is not 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 144.0 °C and 186.0 °C with mesomorphic range varying from 10.0 °C at the octyl homologue to a maximum of 15.0 °C at the decyle homologue of the series. Thus, the present homologues series is considered as middle ordered melting type with wide range of liquid crystallinity. No terminal group is present in our compound. 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 to nematic transition curve rises and falls in parallel manner. Emergence of parallel effect in nematic-isotropic transition curve is observed due to presence of methylene units linked through oxygen atom as alkoxy group.

**Table 2.** Transition temperatures.

| **Sr. No.** | ***n*-Alkyl Group** | **Transition temperatures in °C** | | |
|---|---|---|---|---|
| | | **Smectic** | **Nematic** | **Isotropic** |
| 1 | Methyl | - | - | 186 |
| 2 | Ethyl | - | 172 | 184 |
| 3 | Propyl | - | - | - |
| 4 | Butyl | - | 167 | 181 |
| 5 | Pentyl | - | - | - |
| 6 | Hexyl | - | 160 | 172 |
| 7 | Heptyl | - | - | - |
| 8 | Octyl | - | 152 | 162 |
| 9 | Decyl | - | 140 | 155 |
| 10 | Dodecyl | - | 138 | 152 |
| 11 | Tetradecyl | - | - | - |
| 12 | Hexadecyl | - | 133 | 144 |

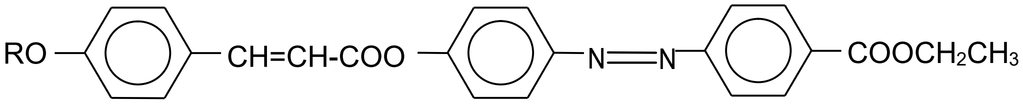


**Figures 3**. Phase behaviour of compound $AB_1$-$AB_{12}$: No of carbon in alkyl chain vs. Temperature.



Series – 1 2-formyl-4-((*E*)-(4-((*E*)-phenyldiazenyl)phenyl)diazenyl)phenyl 4-Alkyloxybenzoate

Series – A



Average thermal stability and stage of commencement of mesophase formation are recorded in **Table 3** as under.

The present homologues series (1) is compared with structurally other homologous series (A).[27] for molecular characteristics and thermal stabilities, which are shown in **Table 3.** The above homologous series (A) under discussion have the basic length due to three phenyl rings linked through central linking units –-COO- and –N=N-, left n-alkoxy group, and right terminal $–COOC_2H_5$ functional group at para position and other hand, series (1) have four phenyl ring linked through -N=N-,-N=N-,-COO- and left n-alkoxy group present. Hence, display of mesomorphic properties due to the molecular forces arising on account of these remains the same. The homologous series (1) and (A) mainly differ from number of phenyl ring and also differ from terminal group. No terminal group for series (1) while respect to –COOC2H5 for series (A).

However, the net effective resultant forces of intermolecular attractions are capable to resist thermal vibrations and maintain only statistically parallel orientations of molecules in floating condition, displaying nematogenic character with exhibition of smectic character. Thus, forces of attractions are weakening due to broadening a molecule. Thus, in case of series (1) as compared to series (A) melting and transition temperatures of titled homologous series (1) are relatively 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 then series (A).

**Table 3.** Average thermal stability.

| **Series** | **Series – 1** | **Series – A** |
|---|---|---|
| Nematic -isotropic<br>Commencement of nematic Phase | 167.0°C<br>$(C_1 - C_{16})$<br>$C_1$ | 293.66°C<br>$(C_1 - C_{16})$<br>$C_1$ |
| Total mesophase length in °C (Nm-Iso)<br>$Cn_1$ $Cn_2$ | 10,0°C – 15.0°C<br>$C_8$ - $C_{10}$ | 10 °C – 130°C<br>$C_{16}$ - $C_1$ |

## 4. CONCLUSION

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 of smectic phase. Present investigation support the earlier view 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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