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A Synthesis and Mesophase Behavior of Homologous Series: 4-((E)-((2,6 Dibromo-4-((E)-phenyl diazinyl) phenyl) diazinyl) phenyl) amino) methyl) phenol

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ABSTRACT

Ten homologues of 4-((E)-((2,6 Dibromo-4-((E)-phenyl diazinyl) phenyl) diazinyl) phenyl) amino)methyl) phenol have been synthesized to create a new homologous series of liquid crystals. This series shows both nematic and smectic mesophases and a middle-order melting behaviour. With alternating increases and reductions, the transition curve between solid, isotropic, and mesomorphic states does not exhibit a zigzag pattern. The nematic phase exhibits a droplet-nematic texture. This series' mesomorphic characteristics and thermal stability are contrasted with those of structurally related substances. An optical hot-stage polarising microscope is used to measure transition temperatures, and analytical results validate the compounds' molecular structures.

Keywords: Nematic, Smectic, Liquid crystals, Azo compounds, Ester linkage, Mesomorphism, Thermal analysis.

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INTRODUCTION

Because of their many uses and adaptable functioning, liquid crystalline (LC) materials have attracted more attention recently [1–5]. The addition of long-chain alkoxy groups from acid derivatives has been thoroughly investigated among the attempts to produce novel mesogenic cores, especially in order to comprehend the structure-property correlations. These investigations concentrate on how important characteristics, including melting point, transition temperatures, and mesophase shape, are impacted by variations in flexibility and rigidity. Such studies demonstrate that mesomorphic features can be significantly altered by very small changes in molecular structure [6–9]. In this situation, the stiff core groups are connected by the Schiff base functionality (-CH=N-) [10–14].

Liquid crystalline (LC) materials have been investigated by several researchers for a range of uses. A thorough analysis of the most recent developments in LC materials in the biomedical sector was given by Shang et al. [15–18]. The basic principles of LC materials and their functionalised derivatives are covered in their study, which emphasises their potential for uses in medication research, artificial implants, illness detection, and health monitoring. Important insights for the advancement of LC-based technology in various fields are also presented in the study. Rabbi et al. provided a thorough analysis of the characteristics and potential uses of thermotropic and lyotropic LC materials in optoelectronics, concentrating on the characterisation methods for these materials [16].

In a different study, Wang et al. explored the synthetic methods available to manipulate molecule orientation and create macroscopic changes in characteristics while discussing the state of 4D printing of liquid crystalline elastomers. Their study explores how programmable topological characteristics of printed materials interact with programmable LC behaviours and external stimuli [19].
Liquid crystalline (LC) materials are becoming more and more important in the field of materials research, as evidenced by the studies that have been published thus far. Our goal in this work is to examine the mesomorphic behaviour of a novel homologous series of liquid crystals (LC) made up of derivatives of n-alkoxybenzaldehyde with different alkyl chain lengths. Ambroxol and the Schiff base of an aromatic aldehyde were combined to create the LC materials. Several experimental approaches were used to characterise the resulting homologous series, which had alkyl chain lengths ranging from C1 to C16.

EXPERIMENTAL

MATERIALS & MATHODS

To carry out our experimental work, all other chemicals and reagents used were of analytical grade. 4-hydroxybenzaldehyde, alkyl bromides of C_1 to C_{16}*n*-alkyl chains, and Ambroxol were procured from Avra Synthesis Private Ltd. Hyderabad, India. Double distilled water was used for each experimental work. TLC plates (silica gel 60 F254 silica-aluminum plates) were purchased from Merck, India.

Synthesis of Liquid Crystal

Firstly, to synthesize 4-*n*-Alkoxybenzaldehydes was refluxed with corresponding *n*-alkyl bromides in the presence of potassium carbonate (1 equiv.) and acetone as a solvent by using reported method [20].Then after, the prepared n-alkoxybenzaldehyde reacted with 4-((2-amino-3,5-dibromobenzyl)amino)cyclohexan-1-ol in the presence of ethanol reflux condition 5-6hrs.

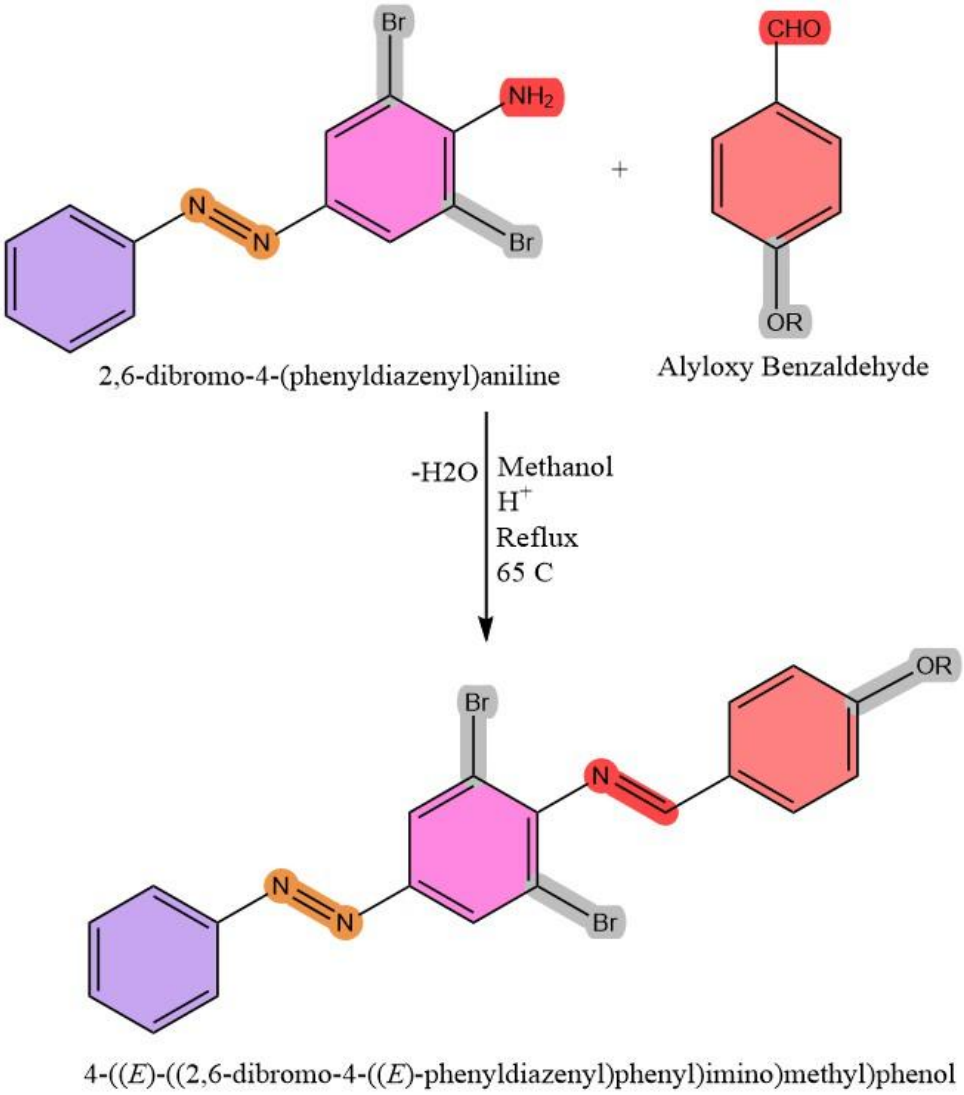

Figure 1. Synthetic scheme for n-alkoxybenzaldehyde derivatives.

CHARACTERIZATION

Thermal analysis was conducted using differential scanning calorimetry (DSC) on a Perkin Elmer Thermal Analyzer, with a heating rate of 10 °C/min. Fourier-transform infrared (FT-IR) spectra were obtained using the KBr pellet method and analyzed in the range of 3500–500 cm^{-1} with a Bruker TENSOR 27 instrument. Proton Nuclear Magnetic Resonance (^{1}H-NMR) spectra were recorded on a Bruker Avance (400 MHz) spectrometer in $CDCl_3$ solvent, with tetramethylsilane (TMS) as the internal standard. The mesophase was identified using a polarizing optical microscope (POM), employing a Nikon Eclipse LV-100 POL with a temperature-controlled heating stage.

Spectral Data:

FT-IR (KBr): ν_{max}/cm^{-1}

Compound-C14

IR Spectrum: 2957-2849 (SP^3 C-H str), 1606 (CH=N str), 1569-1467 (aromatic SP^2 C-H stretching), 1306 (stretchingof C-O) , 1251-1167 (ether linkage), 1106-1019 (C-C str), 882 (str of poly CH_2 Group), 855-813 (C-H oop bending, substituted benzene ring), 765-722 (pera di substituted benzene ring), 685 (C-Br str) cm^{-1}

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.42 (s, 1H, CH of CH=N), 7.90 (m, 5H, Ar-H of mono substituted Ar-ring), 7.51 (m, 2H, Ar-H of br2 substituted ring), 7.25 (dd, 2H, Ar-H of benzaldehyde), 6.97 (dd, 2H, Ar-H of benzaldehyde), 4.02 (t, 2H, CH_2 of OCH_2), 1.83 (m, 2H, CH_2 of OCH_2 CH_2), 1.55 (m, 2H, CH_2 of OCH_2 CH_2), 1.25 (m, 20H, CH2 of alkyl chain), 0.87 (t, 3H of alkyl chain CH_3).

Compound-C10

IR Spectrum: 2955-2851 (SP^3 C-H str), 1605 (CH=N str), 1568-1420 (aromatic SP^2 C-H stretching), 1306 (stretchingof C-O) , 1250-1166 (ether linkage), 1106-1021 (C-C str), 881 (str of poly CH_2 Group), 855-815 (C-H oop bending, Substituted benzene ring), 765-719 (pera di substituted benzene ring), 685-619 (C-Br str) cm^{-1}

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.42 (s, 1H, CH of CH=N), 7.90 (m, 5H, Ar-H of mono substituted Ar-ring), 7.51 (m, 2H, Ar-H of br2 substituted ring), 7.31 (dd, 2H, Ar-H of benzaldehyde), 6.99 (dd, 2H, Ar-H of benzaldehyde), 4.02 (t, 2H, CH_2 of OCH_2), 1.81 (m, 2H, CH_2 of OCH_2 CH_2), 1.47 (m, 2H, CH_2 of OCH_2 CH_2), 1.27 (m, 20H, CH2 of alkyl chain), 0.88 (t, 3H of alkyl chain CH_3).

Results & Discussion

The prepared LC samples of (E)-4-((3,5-dibromo-2-((4n-alkoxybenzylidene)amino)benzyl)amino) cyclohexan-1-ol derivatives have been analyzed for elemental (CHN) analysis. The obtained data are found to be well matched with theoretical values of corresponding samples. (**Table 1**)

Table 1. Elemental analysis for compound AB_1 and AB_2.

Sr. No.	Molecular Formula	Elements % Calculated (% Found)		
		C	H	N
1.	C20H15Br2N3O	50.73% (50.56%)	3.17% (3.12%)	8.87% (8.39 %)
2.	C21H17Br2N3O	51.74% (51.68%)	3.49% (3.25%)	8.62% (8.37 %)

POM Texture Analysis

POM analysis was performed using a glass slide, where the samples were sandwiched and covered with a cover slip to identify the phase transition temperatures of the synthesized LC derivatives. In this study, microphotographs of the textures observed for the C10 and C12 samples are shown in **Figure 2.** The image of sample C10 clearly demonstrates a nematic phase at 123 °C under heating conditions. In same way, sample C12 exhibits a droplet-type texture characteristic of the nematic phase at 117 °C and 130 °C upon heating.
The prepared LC samples were further examined for their phase behavior, with phase transition temperatures recorded using a polarizing microscope with a heating stage (Table 2). The focus of this study was to investigate the effect of varying aliphatic alkyl side chains substituted in the n-alkoxy group on the mesomorphic properties. To visualize this, a plot of transition temperature versus the number of carbon atoms in the alkyl chain was created, as shown in **Figure 3.**The results indicate that among the samples in this series, C1, C2, C3, C5, and C7 do not exhibit liquid crystalline behavior due to their high crystallinity. This behavior is attributed to the presence of weak dispersion forces and limited dipole-dipole interactions, which result in insufficient anisotropic intermolecular forces. The adverse effects of both molecular rigidity and flexibility contribute to these samples' inability to form mesophases.

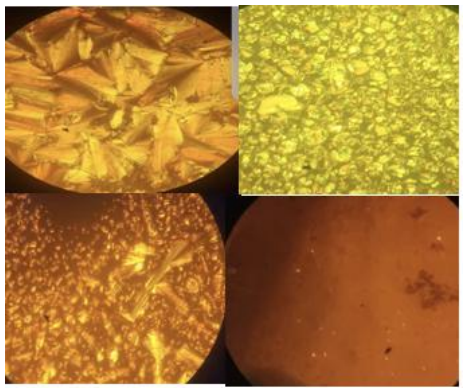

Figure 2. POM images for the texture of Samples C_{10}(A) Nematic marble texture and C_{12} (B) Nematic marble texture.

Table 2. Phase transition temperatures (°C) of the synthesized LC samples (heating scan).

Samples	**Smectic**	**Nematic**	**Isotropic**
C_1	--	-	165
C_2	--	-	161
C_4	--	-	157
C_5	--	-	141
C_6	--	-	136
C_8	--	-	135
C_{10}	-	123	133
C_{12}	-	117	130
C_{14}	-	111	119

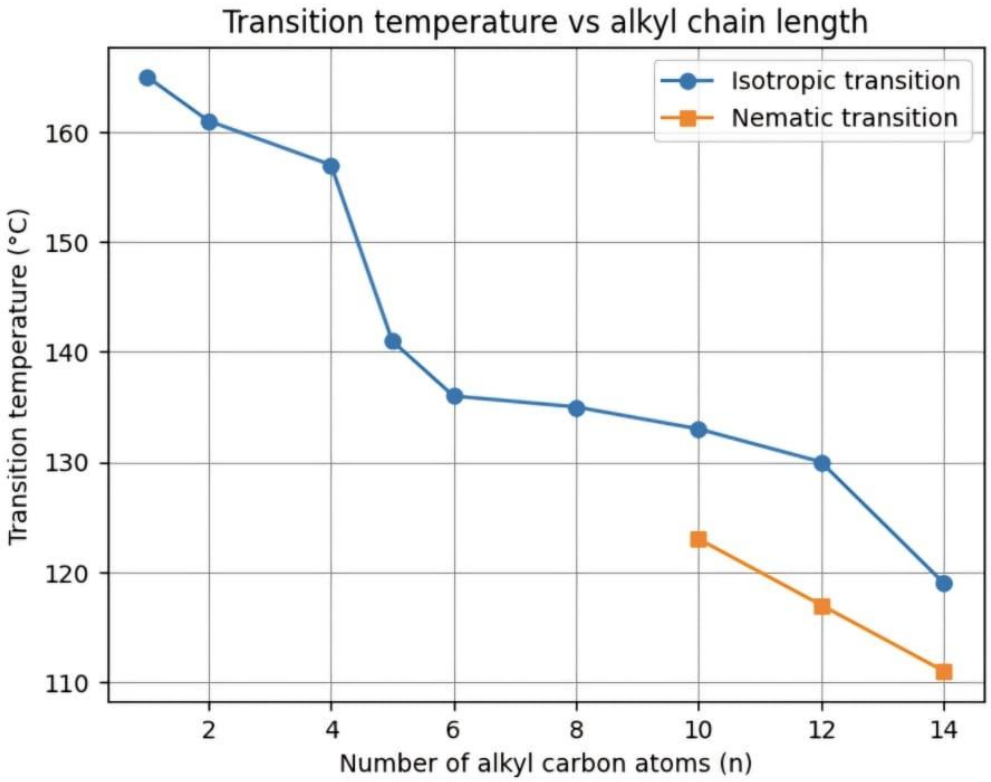

Figure 3. Phase diagram for the synthesized samples.

CONCLUSIONS

In this work, we synthesized a series of n-alkoxybenzaldehyde derivatives by varying the alkyl chains at the terminal moieties as alkoxy groups. The structural identification of the synthesized compounds was confirmed through spectral and thermal analyses. The series was found to exhibit nematogenic behavior with a short-range liquid crystallinity, displaying a smectic phase. The observed mesogenic properties and transition temperatures were influenced by the size of the alkyl chains, indicating that mesomorphism is sensitive to the molecular structure. For practical applications, the smectogenic and nematogenic homologues with transition temperatures below 80°C could be useful in device applications.

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Conflict of interest: None

The authors declare no conflict of interest.

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