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Synthesis, Characterization and Liquid Crystalline Behaviour of 4-((*E*)-(((*E*)-4-(Decyloxy)Benzylidene)Hydrazineylidene) Methyl)Phenol

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The liquid crystal phase may be considered a disturbance of the liquid state. Basic mean-field theories can adequately describe some features of liquid crystal behavior by concentrating on liquid crystalline qualities. The modification of the organic chemical structure, such as the linking unit, core system, and terminal alkyl chain, was studied in order to produce new liquid crystalline derivatives. In this study, the preparation and characterization of a new hydrazine-based compound, 4-((*E*)-(((*E*)-4-(decyloxy)benzylidene) hydrazineylidene)methyl)phenol were conducted. The structural elucidation of this compound was characterized using Fourier transform infrared (FTIR) spectroscopy, nuclear magnetic resonance (NMR) spectroscopy, and CHN elemental analysis. The texture of liquid crystal properties was determined using polarized optical microscopy (POM), and their transition was further confirmed using differential scanning calorimetry (DSC). The compound was found to be mesogenic with smectic C and nematic phases. The mesogenicity of this compound was affected by the polarizability and molecular interaction stability between the Schiff base linkage and decyl terminal chain.

Keywords: Liquid Crystal; mesogenic; Schiff base; Smectic C; Nematic

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Liquid crystal is between the solid (crystalline) and liquid (isotropic) phases, commonly referred to as the "fourth state of matter" or "mesophase" [1-3]. These substances have both solid and liquid characteristics [4,5]. In a liquid crystalline material, anisotropic intermolecular interactions of the molecules, or mesogens, mean that the molecules have some orientational or positional order but with a lower degree of organization than in a crystalline solid. Liquid crystals might flow like a liquid, but their molecules can be oriented similarly to the crystal [6]. There are a few ways to classify the liquid. Calamitic (rod-like), discotic (disc-like), amphiphilic, nonamphiphilic, metal-containing, non-metal-containing, low molecular weight, or polymeric molecules can be found in the mesophases (mesogens) [7].

Schiff base is one of the most well-known linking units used in connecting the rigid core groups [8-10]. Schiff base, known as imine or azomethine, was named after Hugo Schiff. It is a nitrogen analog of an aldehyde or ketone in which the carbonyl group (C=O) is replaced by an imine group [11]. Schiff base contains a carbon-nitrogen double bond connected to an alkyl or aryl group but not the hydrogen. The Schiff base molecules provide a stepped core structure that can maintain the molecular linearity to provide high stability and enable mesophase formation [12,13]. Schiff bases are very useful in liquid crystal due to their low phase transition temperatures [14]. Schiff bases offer the possibility of controlling the alignment and orientation of their molecules to generate liquid crystal materials [15,16].

Hence, this research work focused on synthesizing thermotropic, rod-like, liquid-crystalline molecules. The new hydrazine-based compound having a Schiff base and a decyl terminal end was synthesized and characterized. The purpose of this study is to understand the relationship between the skeleton structure of these types of molecules and the liquid crystal mesophase. The presence of two Schiff base linkages in the hydrazine-based compound impacts the mesophase behavior. The molecules' length increased due to the Schiff base linking unit conjugating with the benzene rings, which improved the compounds' anisotropic polarizability and flexibility. As a result, the rigidity and linearity of its components were preserved, and mesomorphic materials were given numerous property modifications. The existence of the decyl terminal end influences the molecules' polarizability, allowing the formation of smectic phases. Thus, a new hydrazine-based compound with a Schiff base and a decyl terminal end was prepared to study the molecular interactions and their mesophase properties.

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EXPERIMENTAL

Characterization Method

The chemical progress of the reaction was monitored by thin layer chromatography, and the functional group of the compounds was determined using a Perkin Elmer FTIR Microscope Spotlight 200 spectrometer (PerkinElmer, Waltham, MA, USA). The chemical structure of these compounds was verified using a Bruker NMR 500 MHz Ultrashield spectrometer (Bruker, Coventry, UK), and the purity of the compounds was confirmed using a CHN analyzer, model Perkin Elmer II, 2400 (PerkinElmer, Waltham, MA, USA). Meanwhile, the POM instrument (Linkam, London, UK) was used to determine the mesophase transition, which was further clarified using the Pyris 1 Differential Scanning Calorimeter, PerkinElmer (PerkinElmer, Waltham, MA, USA).

Synthesis of Compounds 1-3

The overall schematic reaction of the preparation of compounds **1-3** is summarized in Scheme 1. All the compounds were synthesized following the published procedures, with some modifications [17-20].

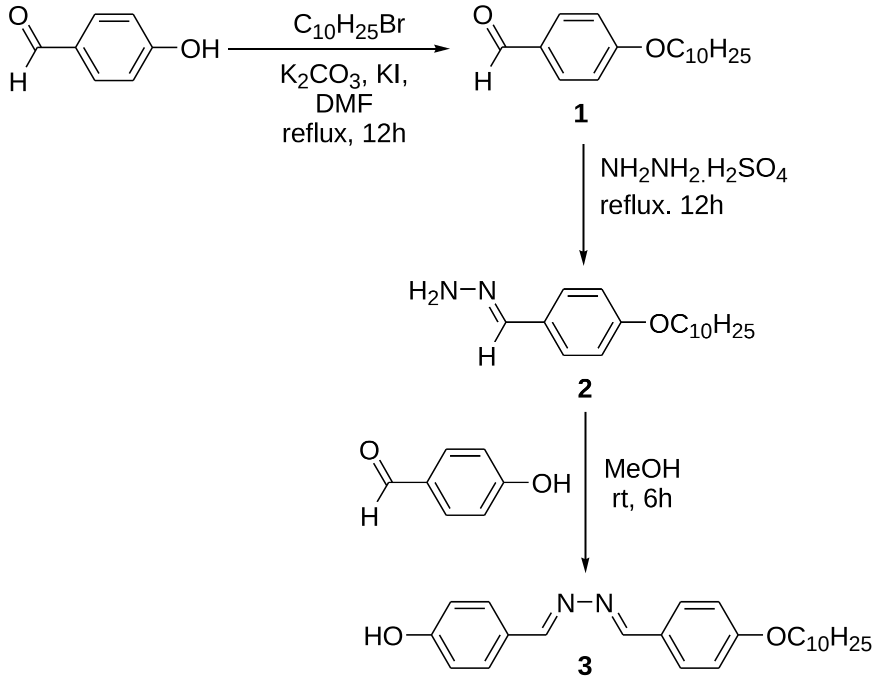
Synthesis of 4-decyloxybenzaldehyde, 1

4-hydroxybenzaldehyde (0.1 mol) was dissolved in *N*,*N*-dimethylformamide, DMF (20 mL) while 1-bromodecane (0.1 mol) was dissolved in DMF (20 mL), separately. Both solutions were mixed in a 250 mL round-bottom flask. Potassium carbonate (0.15 mol) and potassium iodide (0.01 mol) were added to the mixture and then refluxed for 12 hours. The reaction progress was monitored using TLC. Upon completion, the mixture was poured into 500 mL of cold water and extracted with dichloromethane. The organic layers were collected, dried in anhydrous sodium sulphate, filtered, and evaporated overnight.

Yield: 2.51 g (95.8%), yellow solution. FTIR (cm^{-1}): 2,923 and 2,854 (C*sp*^{3}-H stretching), 2,737 (CHO stretching), 1,510 (aromatic C=C stretching), 1,690 (C=O aldehyde), 1,253 (C-O stretching), ^{1}H NMR (500 MHz, $CDCl_3$) δ, ppm: 9.71 (s, 1H), 7.65 (d, *J* = 5.15 Hz, 2H), 6.82 (d, *J* = 5.15 Hz, 2H), 3.84 (t, *J* = 2.9 Hz, 2H), 1.64 (m, 2H), 0.75 (t, *J* = 3.45 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ**,** ppm: 190.21, 164.18, 131.82, 129.73, 114.65, 68.31, 31.90, 29.58, 29.35, 25.97, 22.68, 14.09.

Synthesis of 4-decylbenzylidene Hydrazine, 2

Finely powdered hydrazanium sulphate (0.008 mol) was suspended in a 250 mL beaker containing 20 mL of hot distilled water. Sodium acetate anhydrous (0.006 mol) was then added to the suspension with stirring. The reaction mixture was boiled for 5 minutes before it was cooled to 50 ºC. Hot ethanol (20 mL) was then added to the mixture and stirred for 5 minutes until a cloudy solution started to appear. The precipitate formed was removed via filtration, and the filtrate was added dropwise to an ethanolic solution of 4-decyloxybenzaldehyde, **1** (0.008 mol) in a 250 mL beaker. The reaction mixture was stirred for 12 hours at room temperature. The precipitate obtained was filtered and dried.



Scheme 1. Formation of compounds **1-3**.

Yield: 1.94 g (87.2%), melting point: 204.79 ºC, yellow powder. FTIR (cm^{-1}): 2,917 and 2,850 (Csp^3-H stretching), 1,605 and 1,508 (aromatic C=C stretching), 1,248 and 1,172 (C-O stretching). ^{1}H NMR (500 MHz, $CDCl_3$) δ, ppm: 8.58 (s, 2H), 7.74 (d, *J* = 8.5 Hz, 2H), 6.92 (d, *J* = 8.5 Hz, 2H), 3.99 (t, *J* = 13 Hz, 2H), 1.78 (m, 2H), 1.43(m, 2H), 1.30 (m, 2H), 1.27 (m, 14H), 0.87 (t, *J* = 7 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ, ppm: 161.64, 161.08, 130.10, 126.77, 114.77, 68.19, 31.87, 29.52, 29.38, 29.25, 29.19, 26.01, 22.66, 14.09. CHN elemental analysis for $C_{17}H_{30}N_2O$: C: 73.33%; H: 10.86%; N: 10.06%. Found: C: 73.33%; H: 10.18%; N: 10.10%.

Synthesis of 4-((*E*)-(((*E*)-4-(decyloxy)benzylidene) hydrazineylidene)methyl)phenol, 3

A mixture of 4-hydroxybenzaldehyde (0.01 mol) and intermediate **2** (0.01 mol) was dissolved in 15 mL methanol in a 100 mL round-bottom flask. The reaction was stirred at room temperature for 6 hours. The reaction progress was monitored by TLC. Upon completion, the mixture was cooled in ice water, and the precipitate that formed was filtered and dried.

Yield: 2.52 g (90.3%), Melting point: 129.3-141.1 ºC, yellow powder. FTIR (cm^{-1}): 3,495 (O-H stretching), 2,918 and 2,850 (Csp^3-H stretching), 1,622 (C=N stretching), 1,508 (C=C stretching), 1,250 (C-O stretching), 1,020 (C-N stretching). ^{1}H NMR (500 MHz, DMSO-d_6) δ, ppm: 8.69 (s, 1H), 8.49 (s, 2H), 7.81 (d, *J* = 10 Hz, 2H), 7.14 (d, *J* = 10 Hz, 2H), 7.02 (d, *J* = 5 Hz, 2H), 6.78 (d, *J* = 10 Hz, 2H), 4.02 (t, *J* = 15 Hz, 2H), 1.75 (m, 2H), 1.44 (m, 2H), 1.26 (m, 10H), 0.85 (t, *J* = 15 Hz, 3H). ^{13}C NMR (125 MHz, DMSO-d_6) δ, ppm: 165.01, 161.38, 157.05, 156.31, 143.47, 130.42, 129.68, 122.68, 116.12, 115.09, 68.15, 31.74, 29.41, 29.23, 29.11, 29.07, 25.94, 22.55, 14.41. CHN elemental analysis for $C_{24}H_{32}N_2O_2$: C: 75.75%; H: 8.48%; N: 7.36%; Found: C: 75.71%; H: 8.41%; N: 7.32%.

RESULTS AND DISCUSSION

FTIR Spectral Discussion

Figure 1 shows the FTIR spectrum of compound **3**. There is a strong peak that can be observed at 2,918 and 2,850 cm^{-1} that represents Csp^3-H stretching. The presence of the benzene ring was observed at 1,508 cm^{-1}, and the band 1,4-disubstituted C-H bending was assigned to 869 cm^{-1}. Besides, the C-O stretch occurred at the bands of 1,250 cm^{-1}. The existence of Schiff base can be detected at 1,622 cm^{-1}, and C-N stretching was detected at 1,025 cm^{-1}. Finally, the peak at 3,495 cm^{-1} shows the presence of O-H stretching for the hydroxyl terminal group. No amino group was present, indicating that the formation of two Schiff base linkages was successful.

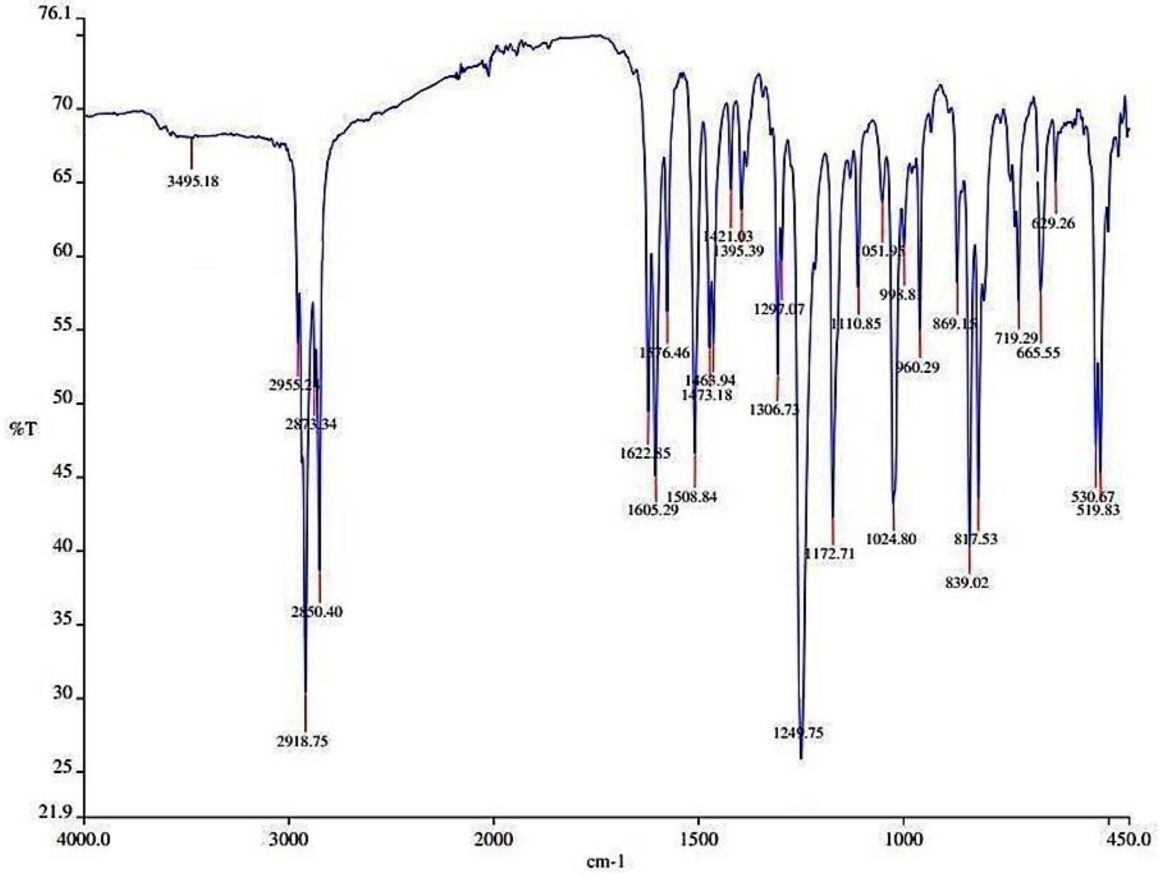


Figure 1. FTIR spectrum of 4-((*E*)-(((*E*)-4-(decyloxy)benzylidene)hydrazineylidene)methyl)phenol, **3**.

NMR Spectral Discussion

The chemical structure of compound **3** with complete atomic numbering is shown in Figure 2. Based on the ^{1}H NMR spectrum (Figure 3), two azomethine protons appear at δ 8.69 and 8.49 ppm, which were assigned to H5 and H6, respectively. The doublets of the aromatic ring were resonated at δ 7.81, 7.14, 7.02, and 6.78 ppm, which were labelled as H2/H2', H3/H3', H8/H8', and H9/H9', respectively. The H2/H2' were deshielded compared to H3/H3', H8/H8', and H9/H9' due to the electron withdrawing group at the hydroxyl terminal end. The electron donation effect from the oxymethylene (-OCH_2-) increases the electron density around the proton, causing them to appear at slightly more shielded region [21,22]. The signal at δ 4.02 ppm was assigned to H11, which is bonded directly to the oxygen atom. The chemical shifts of δ 1.26-1.75 ppm were assigned to decyl chains (H12-H19). Another signal (H20) was observed at δ 0.87 ppm with a triplet splitting pattern that represents the methyl group.

Based on Figure 4, the peaks at δ 165.0 (C5) and 161.4 (C6) ppm denote the presence of azomethine carbons, which confirmed the formation of a Schiff base linkage. C1 and C10 show the peak at δ 157.1 and 156.3 ppm, respectively. C1 experiences more downfield due to the electronegativity of the hydroxyl group. The quaternary carbons C4 and C7 resonated at δ 143.5 and 129.7 ppm, respectively. The aromatic carbons were assigned as C2/C2', C3/C3', C8/C8', and C9/C9', which resonated at δ 130.4, 122.7, 116.1, and 115.1 ppm. The methylene carbon of C11 was located at δ 68.2 ppm. Moreover, it is observed that many signals are present in the region of δ 22.6-31.7 ppm (C12-C19), which are attributed to the decyl terminal chain. The methyl group of these is found to resonate at δ 14.4 ppm (C20).

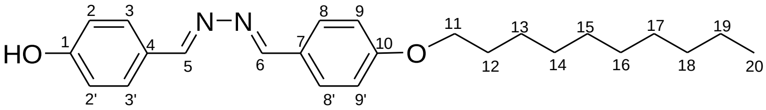


Figure 2. Chemical structure of compound **3** with complete atomic numbering.

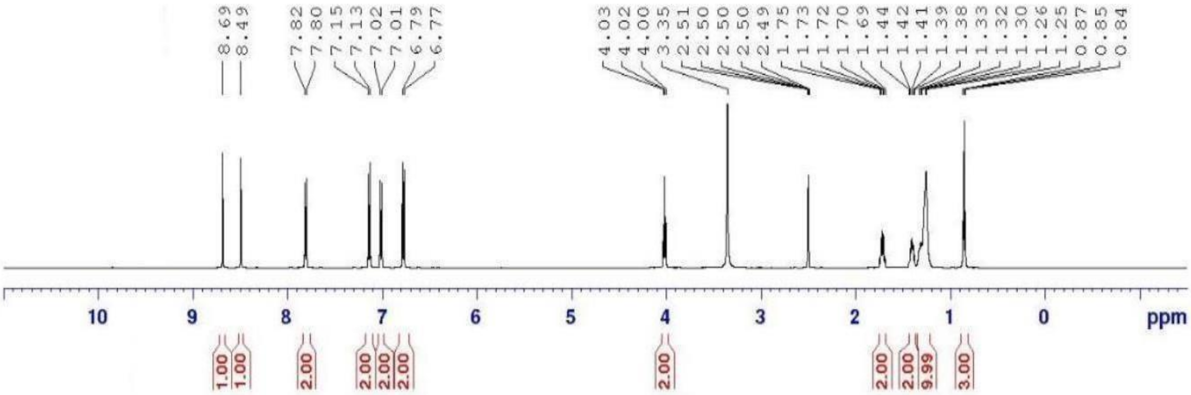


Figure 3. The ^{1}H NMR spectrum (DMSO-d_6, 500 MHz) of compound **3**.

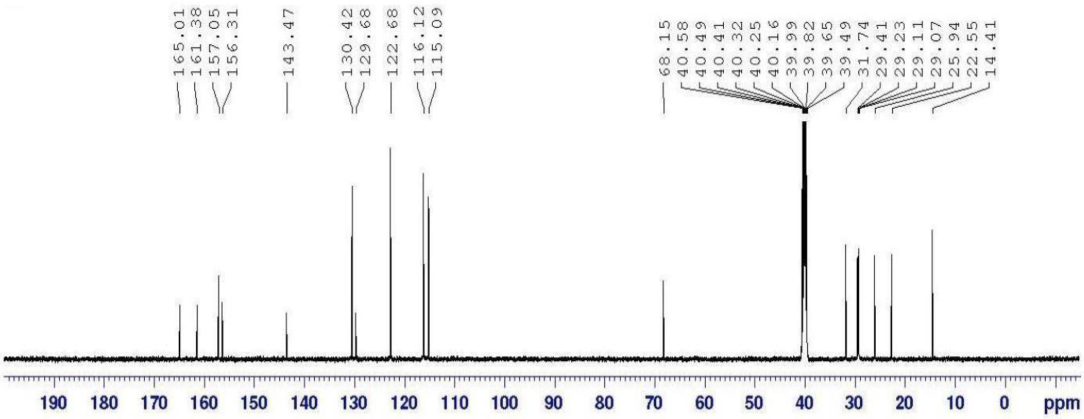


Figure 4. The ^{13}C NMR spectrum (DMSO-d_6, 125 MHz) of compound **3**.

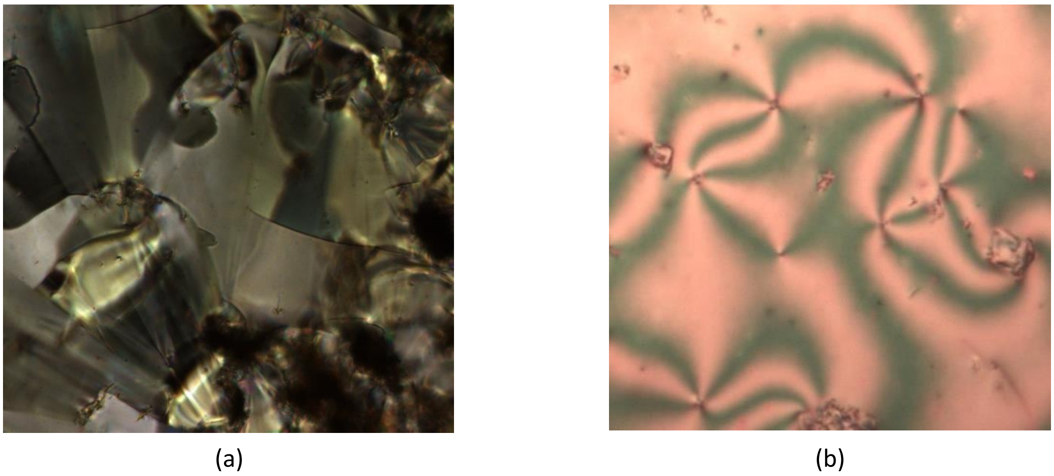


Figure 5. The POM photomicrographs of compound **3** (upon cooling): (a) broken fan-shaped texture of SmC at 130.2 ºC and (b) thread-like open brush nematic phase at 136.8 ºC.

Determination of Liquid Crystal Properties

The determination of the mesophase(s) behavior of the compounds was done using a polarized optical microscope (POM). Observations under POM showed that compounds **1** and **2** are non-mesogenic, while compound **3** was found to exhibit liquid crystal mesophase (mesogenic) properties.

Compound **3** was found to exhibit SmC phases at 129.3 ºC before it turns to nematic at 133.1 ºC in the heating cycle. Meanwhile, the SmC phase appeared at 140.5 ºC from nematic phases and became the crystal phase at 128.4 ºC upon cooling. Figure 5 shows the broken-fan shaped texture of SmC upon cooling (a) at 130.2 ºC and the nematic texture of a thread-like four point brush upon cooling cycle (b) at 136.8 ºC.

The presence of SmC and nematic phases in both cycles were further confirmed using differential scanning calorimetry (DSC). The DSC thermogram of compound **3** (Figure 6) showed three endotherms in the cooling and heating cycles. In the heating cycle, the crystal transition to the smectic C phase occurred at 129.3 ºC, while it turned to the nematic phase at 133.1 ºC. Further heating caused the nematic texture to change to an isotropic phase at 141.1 ºC. The same trends were observed in cooling cycles, but in reverse order. The transition phase upon the heating and cooling cycle of the final compound is shown in Table 1. The dots (•) in Table 1 represent the phases that occurred in the cycles.

The liquid crystal phase may be thought of as a disturbance of the liquid state, and basic mean field theories can adequately describe some features of liquid crystal behavior by concentrating just on liquid crystalline qualities**.** The melting point absorbs the majority of the crystal stabilization energy, and the enthalpy changes associated with crystal/liquid crystal or liquid crystal/isotropic liquid phase transitions are minor [23,24]. Liquid crystal phases are fluid because disorder in molecular locations is dynamic, yet viscosities and other qualities are dependent on the degree of translational order [25,26]. The nematic liquid crystal phase features somewhat ordered molecular orientations but no long-range positional organization. Smectic phases are layered structures with translational order in one direction, whereas columnar liquid crystal phases are layered structures with translational order in two directions and molecular positions disordered in the third [27-29]. Based on this experiment, POM was used to investigate the behavior of some liquid crystalline mesophases.

Table 1. The transition phase of heating and cooling cycles of compound 3.

Mode	**Transition temperature (ºC)**						
Heating	**Cr**		**SmC**		**N**		**I**
	•	129.3	•	133.1	•	141.1	•
Cooling	**I**		**N**		**SmC**		**Cr**
	•	140.5	•	132.1	•	128.4	•

Note: **Cr** = Crystal, **SmC** = Smectic C, **N** = Nematic, **I** = Isotropic

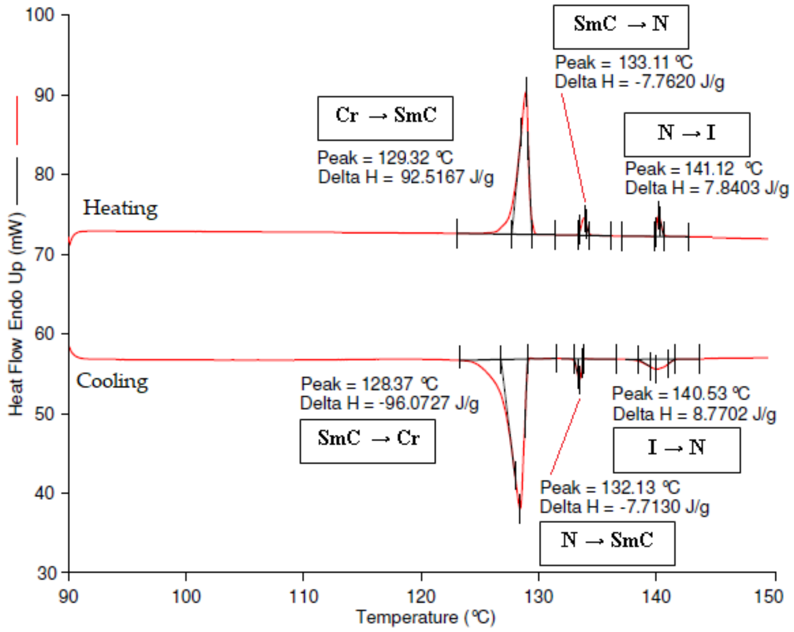


Figure 6. DSC thermogram of compound **3**.

In order to achieve a further illustration of the liquid crystal behavior, POM observations were realized in cooling and heating cycles for compound **3**. Based on this technique, it illustrated liquid crystal properties. Under crossed polarizations, compounds **1** and **2** are not capable of inducing any liquid crystalline behavior; they present only crystal phases. Both compounds only consist of a single benzene ring without any linkages. A connecting unit in a liquid crystal molecule joins two cores or links the terminal chain to the core. This study used the Schiff base as a linking unit in synthesized compound **3**. The Schiff base was able to maintain the linearity of the molecules. Due to its low phase transition temperature, the Schiff base is an example of a connecting unit that is highly valuable in liquid crystal research [30,31]. Based on the POM observation, compound **3** exhibited smectic C and nematic phases. The orientational order will be lost first in the LC mesophase as the temperature rises, but the translational order will be preserved. However, the nematic phase is the only mesophase without translational order at higher temperatures. As a result, the stable nematic phase held for a shorter time than the stabilized smectic phase [32]. Compound **3** showed the smectic phase as the molecule favors a lamellar packing and consists of higher cohesive forces between the alkoxy chain and the core of the compound. Other than that, when compared to shorter homologues, materials with long terminal alkoxy substituents have much higher mesogenicity [33]. Alkoxy groups are common terminal moieties with liquid crystal characteristics. The presence of the decyl terminal group in compound **3** caused high Van der Waals interactions and intertwining possibilities in the terminal chains. Furthermore, the decyl terminal chains can add some flexibility to the rigid core and stabilize molecular interactions for the formation of mesophase. The hydrazine bridge of the Schiff base unit was able to enhance the dipole moment of the compound. Polar linkages with high dipole moments have the capacity to promote mesomorphic characteristics [34]. The enhanced dipole moment improves lattice stability and melting temperatures. The molecules tend to align in a parallel pattern as the length of the terminal substituent grows [35,36]. As a result, the compounds have high polarizability, which generates mesophase transitions.

CONCLUSION

In conclusion, all the compounds were successfully synthesized and characterized. All the compounds were characterized using Fourier transform infrared (FTIR) spectroscopy, nuclear magnetic resonance (NMR) spectroscopy, and CHN elemental analysis. The absence of an aldehydic signal indicated the formation of Schiff base linkages in compound **3** was a success. The determination of liquid crystal mesophase was confirmed by using a polarized optical microscope (POM). The observation under POM showed that the phase transition of compound **3** exhibits SmC and nematic phases and proves that the compound is mesogenic in the DSC thermogram. Compound **3** consists of the hydrazine bridge of the Schiff base unit, which increases the molecules' polarizability. Incorporating the decyl terminal chain stabilized the molecular interaction between the core unit and terminal group. Hence, the effect of the Schiff base linkage and the decyl terminal end in the compound can induce the liquid crystal behavior.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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