Wasit Journal for Pure Science



Journal Homepage: https://wjps.uowasit.edu.iq/index.php/wjps/index
e-ISSN: 2790-5241 p-ISSN: 2790-5233

Synthesis, Characterization and Study of Liquid Crystal Behavior of Linear-Shaped Mesogens Containing Cyano and Alkyl as a Terminal Groups

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DOI: https://doi.org/10.31185/wjps.845

Received 03 April 2025; Accepted 02 June 2000; Available online 30 December 2025

ABSTRACT: A novel series of liquid crystalline compounds containing 2,4-thiazolidinedione units with varying terminal alkyl chain lengths was successfully synthesized and characterized. The chemical structures of the synthesized compounds were confirmed by FT-IR, ¹H-NMR, and mass spectrometry. The mesomorphic behavior was investigated using polarized optical microscopy (POM) and differential scanning calorimetry (DSC). Compounds [V]₄, [V]₅, and [V]₆ exhibited enantiotropic nematic phases, while compound [V]₈ displayed a smectic A (SmA) phase. No liquid crystalline behavior was observed for compound [V]₃. The liquid crystalline properties were found to depend on the terminal-to-lateral chain length ratio, molecular geometry, and the nature of the linking groups. Additionally, an odd—even effect was observed in the crystal—nematic and nematic—isotropic transition temperatures for certain members of the series. The results highlight the crucial role of terminal chain length and cyano substitution in stabilizing mesophases and tuning the thermal behavior of these 2,4-thiazolidinedione-based liquid crystals.

Keywords: liquid crystal, heterocyclic, Nematic phase, Smectic phase



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1. INTRODUCTION

The liquid crystalline (LC) phase represents a unique state of matter that combines the anisotropic molecular order of crystalline solids with the fluidity of isotropic liquids [1]. This dual nature arises from the partial alignment of mesogenic units along a preferred director axis, allowing the material to respond to external stimuli such as electric fields, temperature, and light. Over the past few decades, liquid crystals have revolutionized display technologies (e.g., LCDs) owing to their tunable electro-optical properties [2].

A critical factor in LC design is the incorporation of heterocyclic motifs [3–9], which enhance thermal stability, polarizability, and mesophase diversity. The polar characteristics of the cyano group and the high polarizability of the aromatic core confer to cyanophenyl its characteristic large dipole moments, while cyano group termination significantly improves liquid-crystal stability [10]. Recent studies have investigated the optical properties, electronic transitions in solvent media, and dipole moments of nematic liquid crystals containing cyano groups [11]. This unique combination enables LCs to exhibit a wide range of anisotropic physical properties, including direction-dependent optical and mechanical characteristics, with applications in optical modulators and resilient liquid-crystal-based spider silk fibers. Furthermore, LCs are readily distorted and respond dynamically to external influences such as electric fields or shear stresses [12].

In comparing the synthesized compound in this study with a previously reported compound [6], it was found that the compound prepared here, containing both a cyano group and an alkyl chain (n = 8), exhibited smectic A and nematic phases. In contrast, the compound reported in the literature [6], which contained only a cyano group without an alkyl

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substituent, showed exclusively a nematic phase. Therefore, the aim of this work is the synthesis of a novel series of linear-shaped mesogens incorporating thiazolidine-2,4-dione moieties with cyano and variable alkyl chains.

2. EXPERIMENTAL

Materials: All chemicals were purchased from Loba Chemie, HiMedia, Alpha Chemika, and Merck.

Techniques: Fourier-transform infrared (FTIR) spectra were recorded using a SHIMADZU IRSpirit-X series spectrometer (400–4000 cm⁻¹, Japan). ¹H-NMR spectra were obtained on a Bruker 400 MHz spectrometer. Mass spectra were measured with an Agilent 5975C spectrometer. Melting points were determined using a Stuart SMP10 melting point apparatus. Polarized optical microscopy (POM) was performed with a PW-BK 5000PR microscope. Differential scanning calorimetry (DSC) measurements were carried out on a STA500 instrument at a heating/cooling rate of 20 °C/min.

2.1 Synthesis

The novel compounds were synthesized using the synthetic pathway defined in Scheme 1.

$$\begin{array}{c} \text{HO} \\ \text{4-hydroxybenzylaldehyde} \\ \text{4-hydroxybenzylaldehyde} \\ \text{Pipridine} \\ \text{CI} \\ \text{DMF} \\ \text{CI} \\ \text{CI} \\ \text{DMF} \\ \text{CI} \\ \text{CI} \\ \text{DMF} \\ \text{CI} \\ \text{CI} \\ \text{CI} \\ \text{CI} \\ \text{CI} \\ \text{OH} \\ \text{Reflux 8-10 ln} \\ \text{H2N} \\ \text{NH2} \\ \text{NH2N} \\ \text$$

n= 3,4,5,6 and 8

Scheme 1: Synthesis routes of compounds [I-Vn]

Synthesis of 4-formylphenyl 2-chloroacetate [I]

4-hydroxybenzylaldehyde (1mmol,0.122g) was dissolved in a mixture of 2 mL of pyridine and 2 mL of DMF. (1mmol,0.113g) chloroacetyl chloride* was added dropwise with stirring while maintaining an ice bath for 4 hours [13].

After this, a few drops of 10% hydrochloric acid were added to neutralize the excess base. The product was then isolated by extraction with ethyl acetate and dried. and recrystallized from ethanol The yield was 87%, mp= 71-72°C. FT-IR (v/cm^{-1}): 3070 (C-H Ar.), 2984,2865(C-H aliph.), 2750(C-H aldehyde), 1759(carbonyl ester) 1668 (carbonyl aldehyde), 1604(C=C), 754 (C-Cl). ¹H-NMR show signals in δ (ppm): 9.80(s,1H, aldehyde), 7.99-6.75(dd,4H, ArH), 4.29(s,2H, CH₂). m/z =197,169,121(base peak),105,93,78,65,64,51and 49.

Synthesis 4-formylphenyl 2-(4-cyanophenoxy) acetate [II]

Compound [I] (1mmol,0.198g) and 4-cyanophenol (0.119 g, 0.001mol) were combined with CH3COONa (0.082 g, 0.001mol) in 5mL of EtOH in a round bottom flask. And it was reflux for 4 hours, The extraction method was used to isolate the product with ethyl acetate and leave it to dry and recrystallized from ethanol [14].

The molecular formula: C₁₆H₁₁NO₄, Color: dark brown, melting point= 88-91°C, Yield 84 %

FT-IR(ν / cm^{-1}): 3024(C-H Ar.), 2957,2813 (C-H Alph.), 2213 (C \equiv N), 1743(C=O ester), 1658(C=O aldehyde), 1600 (C=C), 1218 (C-O). H-NMR show signals at δ (ppm): 9.80(s,1H, CHO), 7.79-6.89(m,8H, Ar-H), 1.92(s,2H, CH₂).

Synthesis of the Thiazolidine-2, 4-dione [III]

Thiourea (4.56 g, 60 mmol) and chloroacetic acid (5.64 g, 60 mmol) were added to a 50 mL round-bottom flask. The mixture was allowed to stand until a precipitate formed, then stirred at 100 °C for 15 min. Concentrated HCl (6 mL) was added dropwise via a dropping funnel, and the reaction mixture was refluxed for 10 h. Upon cooling, crystals were obtained, washed with ice water to remove residual HCl, and recrystallized from cold ethanol to yield thiazolidine-2,4-dione [III] as a crystalline solid [15].

yield 82% melting point= 124-127 °C, The FTIR(v/cm⁻¹): 3135 (N-H), 3047 (C-H Ar), 2947,2823 (C-H aliph.), 1735-1654 (carbonyl groups), 617(C-S).

Synthesis of (Z)-4-((2,4-dioxothiazolidin-5-ylidene) methyl) phenyl 2-(4-cyanophenoxy) acetate [IV]

A mixture of compound [II] (1mmol,0.281g) and compound [III] (1mmol,0.117g) was add in 5mL of Et-OH with 0.5 mL of piperidine as a base. posteriorly heated and stirred the mixture for 3-4 hr. The extraction method was used to isolate the product with ethyl acetate and leave it to dry and recrystallized from ethanol [16].

Molecular formula: $C_{19}H_{12}N_2O_5S$, Color reddish-brown, mp= 289-291°C Yield 71 %, FT-IR(ν /cm⁻¹): 3212 (N-H), 3076 (C-H Ar.), 2971,2858(C-H aliph.), 2212(C≡N), 1763-1668(C=O groups), 1581 (C=C Ar.), 1235(C-O). ¹H-NMR show signals at δ (ppm): 9.79(s,1H, N-H), 7.78-6.90(m,8H, Ar-H), 5.88(s,1H, CH=), 1.92(s,2H, CH₂).

Synthesis of N-alkyl compounds [V]n

Compound [IV] (1 mmol, 0.380 g) and potassium carbonate (1 mmol, 0.138 g) were dissolved in acetone (10 mL). The appropriate n-alkyl bromide (1 mmol) was then added, and the reaction mixture was refluxed for 20 h [17]. The crude product was isolated by extraction with ethyl acetate and recrystallized from ethanol to afford compound [V] as the corresponding n-alkyl derivative.

(Z)-4-((2,4-dioxo-3-propylthiazolidin-5-ylidene) methyl) phenyl 2-(4-cyano phenoxy) acetate [V]3

Compound [IV] (1 mmol, 0.380 g) and potassium carbonate (1 mmol, 0.138 g) were dissolved in acetone (10 mL). The appropriate n-alkyl bromide (1 mmol) was added, and the reaction mixture was refluxed for 20 h [17]. The product was isolated by extraction with ethyl acetate and recrystallized from ethanol to yield compound [V] as a reddish-brown solid.

Molecular formula: C22H18N2O5S

Yield: 68% Mp: 279–281 °C Color: Reddish-brown

FT-IR (v, cm $^{-1}$): 3040 (C–H, Ar), 2967, 2803 (C–H, aliph.), 2224 (C \equiv N), 1723–1673 (C=O), 1601 (C=C), 1212 (C–O).

¹H NMR (δ, ppm): 7.88–6.81 (m, 8H, Ar–H), 4.04 (s, 1H, –CH=), 3.98–3.61 (t, 2H, –CH₂– of –CH₂CH₂CH₃), 1.92–1.00 (m, 2H, –CH₂– of –CH₂CH₃), 0.98–0.83 (t, 3H, –CH₃).

(Z)-4-((3-butyl-2,4-dioxothiazolidin-5-ylidene) methyl) phenyl 2-(4-cyano phenoxy) acetate [V]4

Molecular formula: $C_{23}H_{20}N_2O_5S$, Color reddish brown, mp = 257-259°C, Yield 70 % FT-IR(ν /cm⁻¹): 3070(C-H Ar), 2958,2871 (C-H aliph.), 2225(Cyano), 1724-1673(C=O), 1601(C=C), 1215(C-O).

¹HNMR show signals at δ(ppm): 7.84-6.89(m,8H, Ar-H), 4.08(s,1H, CH=), 3.67-3.62(t,2H, CH₃(CH₂)₂CH₂), 2.30-1.24(m,4H, (CH₂)₂), 0.96-0.85(t,3H, CH₃).

(Z)-4-((2,4-dioxo-3-pentylthiazolidin-5-ylidene) methyl)phenyl 2-(4-cyano phenoxy)acetate [V]5

Molecular formula: $C_{24}H_{22}N_2O_5S$, Yield 81 %, Color reddish brown, m.p.= 283-285°C FT-IR(ν /cm⁻¹): 3007(C-H Ar), 2954,2858 (C-H aliph.), 2225(Cyano), 1721-1676(C=O), 1602(C=C), 1215(C-O).

¹HNMR show signals at δ(ppm): 8.11-6.90(m,8H, Ar-H), 4.07(s,1H, CH=), 3.66-3.36(t,2H, CH₃(CH₂)₃CH₂), 2.00-1.18(m,6H, (<u>CH</u>₂)₃), 0.92-0.83(t,3H, CH₃).

(Z)-4-((3-hexyl-2,4-dioxothiazolidin-5-ylidene) methyl)phenyl 2-(4-cyano phenoxy)acetate [V]6

The Molecular formula: $C_{25}H_{24}N_2O_5S$, Yield 81 %, Color reddish brown, m.p.= 269-271°C FT-IR (v/cm⁻¹): 3003(C-H Ar), 2950,2858 (C-H aliph.), 2225(Cyano), 1722-1673(C=O), 1574(C=C), 1212(C-O).

¹HNMR show signals at δ (ppm): 7.88-6.90(m,8H, Ar-H), 4.08(s,1H, CH=), 3.93-3.61(t,2H, CH₃(CH₂)₄CH₂), 2.32-1.24(m,6H, (CH₂)₄), 0.91-0.84(t,3H, CH₃)).

$(Z)-4-((3-octyl-2,4-dioxothiazolidin-5-ylidene)\ methyl) phenyl\ 2-(4-cyano\ phenoxy) acetate\ [V] 8$

Molecular formula: C₂₇H₂₈N₂O₅S, Yield 76 %, Color reddish brown, m.p.= 216-218°C

FTIR (v/cm^{-1}): 3079(C-H Ar), 2922,2801 (C-H aliph.), 2229(Cyano), 1729-1680(C=O), 1607(C=C), 1221(C-O). ¹H-NMR show signals at δ (ppm): 8.11-6.89(m,8H, Ar-H), 4.14(s,1H, CH=), 3.59-3.55(t,2H, CH₃(CH₂)₆CH₂), 2.49-1.60(m,6H, (CH₂)₆), 1.49-0.82(t,3H, CH₃).

3. RESULTS AND DISCUSSION

All synthesized compounds exhibited spectral data consistent with their proposed structures, as confirmed by FT-IR, 1 H-NMR, and mass spectrometry. Compound [I] was obtained by reacting one mole of 4-hydroxybenzylaldehyde with one mole of chloroacetyl chloride in DMF with pyridine. The FT-IR spectrum of compound [I] showed the disappearance of absorption bands corresponding to the starting materials and the appearance of characteristic bands for the C=O group of the aldehyde and the C=O stretch. The 1 H-NMR spectrum (Figure 1) displayed signals at δ (ppm): 9.80 (s, 1H, -CHO), 7.99–6.75 (dd, 4H, Ar-H), and 4.29 (s, 2H, -CH₂-). The mass spectrum of compound [I] (Figure 2) showed peaks at m/z = 197, 169, 121 (base peak), 105, 93, 78, 65, 64, 51, and 49, with the major fragmentation pathways illustrated in Scheme 2.

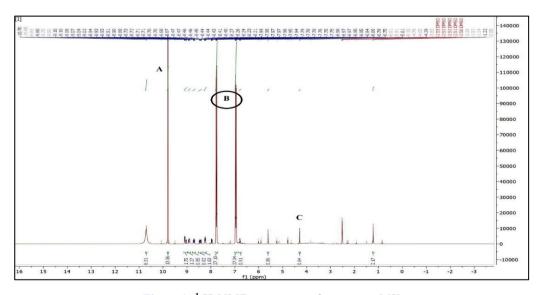


Figure1: 1 H-NMR spectrum of compound [I]

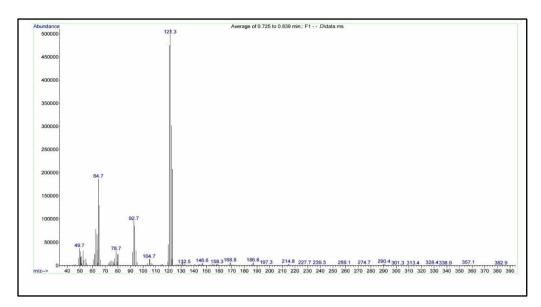
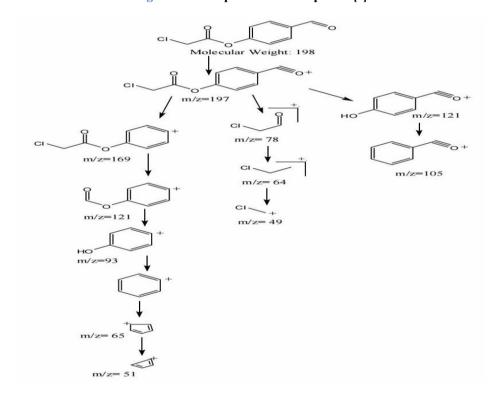


Figure 2: Mass spectrum of compound [I]



Scheme2: Mass fragments for compound [I]

Compound [II] was synthesized by the reaction between compound [I] and 4-cyanophenol in the presence of sodium acetate using ethanol as the solvent. Equimolar amounts of α -chloroacetic acid and thiourea were reacted to afford thiazolidine-2,4-dione [III]. A subsequent Knoevenagel condensation between compound [II] and thiazolidine-2,4-dione [III] in piperidine yielded compound [IV], with the proposed mechanism shown in Scheme 3. The FT-IR spectrum of compound [IV] revealed the disappearance of the aldehyde C=O absorption band present in compound [II] and the appearance of new bands corresponding to the –CH= group and the C=O groups of the thiazolidine-2,4-dione moiety (1721–1674 cm⁻¹).

Scheme3: Mechanism of the Knoevenagel condensation reaction for compound [IV]

The series of n-alkyl derivatives, compounds $[V]_n$, was synthesized by reacting compound [IV] with one mole of the appropriate n-alkyl bromide in the presence of potassium carbonate in acetone. The FT-IR spectra of compounds $[V]_n$ showed the disappearance of the N–H stretching band present in compound [IV] and the appearance of a new C–N stretching band in the range $1061-1109~cm^{-1}$; the FT-IR spectrum of compound $[V]_4$ is shown in Figure 3. The 1 H-NMR spectra of compounds $[V]_n$ exhibited characteristic multiplets at 6.34-8.51~ppm, corresponding to aromatic phenyl protons, and signals in the range 3.91-0.58~ppm, assigned to the terminal alkyl groups (C_nH_{2n+1}) ; the 1 H-NMR spectrum of compound $[V]_5$ is shown in Figure 4.

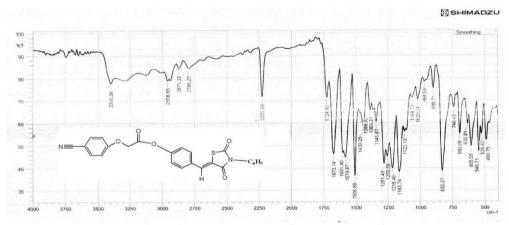


Figure3: FTIR spectrum of compound [V]4

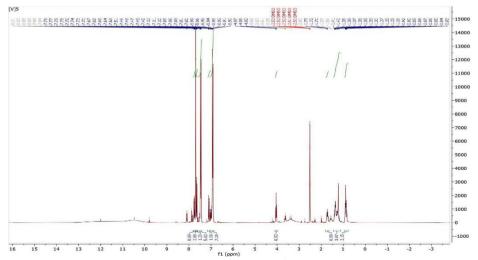


Figure4: ¹H-NMR spectrum of [V]5 compound

3.1 Mesomorphic behavior

The mesophase behavior of the synthesized compounds was investigated using polarized optical microscopy (POM) equipped with a heating stage, complemented by differential scanning calorimetry (DSC) to determine the phase transition temperatures. The observed transition temperatures are summarized in Table 1. Mesophase characterization was performed based on microscopic analysis using established classification systems [18,19].

Compound No.	Phase transition
[V]3	Cr <u>305</u> I
[V]4	$Cr = 193 \qquad N \qquad 288 \qquad I$
[V]5	Cr 88 N 151 I
[V]6	Cr = 97 $N = 200$ I
[V]8	Cr = 77 $SmA = 135$ $N = 198$ I

Table 1: Compounds phase transition temperatures [V]n

Cr: Crystalline / SmA: Smectic A / N: Nematic / I: Iso-tropic liquid

All compounds [V] 4,5,6,8 exhibit liquid crystal properties as Figure 5, while, compound [V]₃ didn't show mesomorphic behavior, and only crystal to isotropic liquid transition was observed.

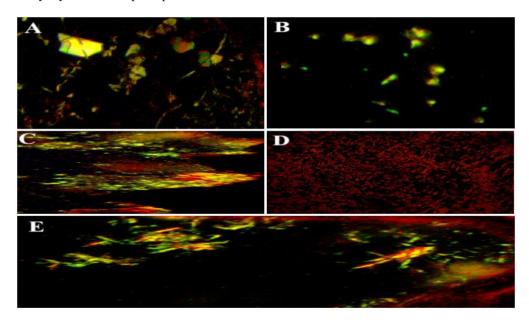


Figure 5: Cross-polarizing photomicrograph of (A) N phase for [V]₄ compound at 244°C (B) N phase for [V]₅ compound at 98°C (C) N phase to isotropic for [V]₆ compound at 199°C (D) SmA phase for [V]₈ compound at 105°C (E) N phase for [V]₈ compound at 166°C (200× magnification).

The DSC thermogram of compound $[V]_6$ is shown in Figure 6, with a phase transition observed at 97.72 °C ($\Delta H = 62.247 \text{ J/g}$). All compounds $[V]_4$, $[V]_5$, and $[V]_6$ exhibited nematic liquid crystal properties, whereas compound $[V]_8$ displayed both smectic A (SmA) and nematic phases. This behavior can be attributed to the terminal-to-lateral chain length ratio (t/l), which significantly affects molecular geometry and, consequently, the type and stability of the mesophase formed. Compound $[V]_3$ did not show any liquid crystalline behavior, likely due to its short alkyl chain length. A high t/l ratio, resulting from longer terminal alkyl chains, generally favors less ordered mesophases, such as nematic, whereas a low t/l ratio promotes more ordered mesophases, such as smectic [20].

Figure 7 illustrates the relationship between the number of carbon atoms in the alkyl chain (n) of the $[V]_n$ series and the corresponding transition temperatures. Compounds $[V]_4$, $[V]_5$, and $[V]_6$ exhibited an odd–even effect on the crystal–nematic and nematic–isotropic transitions.

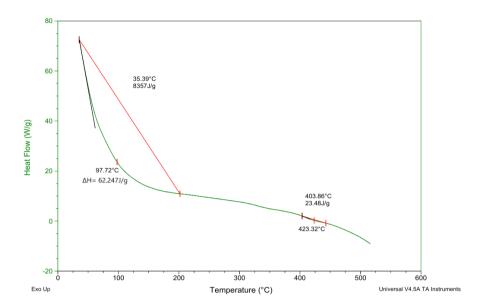


Figure 4: The DSC thermogram for [V]₆ Compound

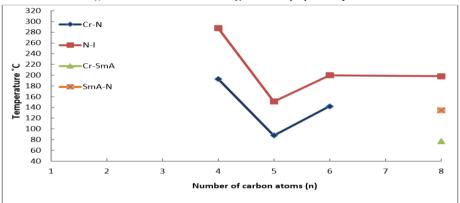


Figure 5: Transition temperature with relation to the quantity of carbon atoms (n) in the alkyl chain of the series [V]n

The liquid crystalline behavior of compounds is generally influenced by the nature of the substituents, the type of linkage groups, and the length of the terminal chains [21]. In particular, cyano-terminated LC molecules play a significant role in stabilizing the LC-water interface. This explains why shells composed of cyanobiphenyl LCs are considerably more stable than those made from LCs with non-cyano-terminated molecules, even when the latter contain aromatic cores [22].

4. CONCLUSIONS

This work reports the synthesis and characterization of a series of liquid crystalline compounds containing 2,4-thiazolidinedione units with varying terminal alkyl chain lengths. Their mesophase behavior was investigated using polarized optical microscopy (POM) and differential scanning calorimetry (DSC). Compounds [V]₄, [V]₅, and [V]₆ exhibited enantiotropic nematic phases, whereas compound [V]₈ displayed a smectic A (SmA) mesophase. Compound [V]₃ showed no liquid crystalline behavior. The observed mesomorphic properties are attributed to the combined effects of molecular geometry, the nature of the linking groups, and the length of the terminal alkyl chains.

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