

Synthesis, Characterization, and Potential Biomssssedical Applications of Novel Symmetrical Liquid Crystal Mesogens

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ABSTRACT

Background: The synthesis and characterization of novel liquid crystalline compounds have garnered significant attention due to their potential applications in biomedical sciences, including drug delivery systems, biosensing, and diagnostic tools. This study focuses on synthesizing and characterizing new thiazolothiadiazole-based liquid crystals and evaluating their mesophase properties. **Methods:** A series of novel compounds containing 5H-thiazolo[4,3-b] [1,3,4] thiadiazole units were synthesized via multi-step chemical reactions. The synthesis involved the reaction of chloroethyl acetate with 4-hydroxybenzaldehyde to yield an aldehyde intermediate, followed by subsequent transformations using hydrazine hydrate, ethyl acetoacetate, and 1,2-dichloromethane or 1,2-dibromoethane. Hydrolysis of an ester intermediate resulted in a carboxylic acid derivative, which was further reacted with 2-phenylenediamine to obtain the final product. **Characterization:** The molecular structures of the synthesized compounds were confirmed using Fourier Transform Infrared Spectroscopy (FTIR) and ¹H Nuclear Magnetic Resonance (¹H-NMR) spectroscopy. Liquid crystal properties were assessed through Differential Scanning Calorimetry (DSC) and Polarized Optical Microscopy (POM) to evaluate phase transitions and mesophase characteristics. **Results:** The study revealed that compound [V]2 exhibited dimorphic behavior, forming smectic C (SmC) and nematic phases, while compounds [V]1, [VI], and [VII] displayed nematic mesophases. The presence of intermolecular hydrogen bonding in compound [VI] extended the rigid-rod moiety, enhancing terminal molecular interactions and stabilizing the nematic liquid crystal phase. **Conclusion:** The synthesized thiazolothiadiazole-based liquid crystalline compounds demonstrate promising mesophase behaviors, which could be further explored for biomedical applications such as biosensing, diagnostic imaging, and targeted drug delivery systems. Their structural properties and phase behavior suggest potential use in pathology-related molecular diagnostics and biomaterial research.

Keywords: Heterocyclics, Liquid Crystal, Mesogen, Mesophase, Thiazolo[4,3-B] [1,3,4] Thiadiazol.

INTRODUCTION

Liquid crystals LC are substances in a state between the characteristics of traditional liquids and those of solid crystals [1]. The elastic force between LC molecules, which has been confirmed to be the main driving force of the self-assembly, is relatively weak and makes it feasible to change the orientation of the molecule [2-4]. Therefore, LCs have inborn capabilities of response to external stimuli, including surface alignment, magnetic field, and electric field [5-7] (above the Fréedericksz threshold). In addition, the optical properties, such as birefringence, scattering and reflection, that are derived from the inherent arrangement of the molecules, are also basic characteristics of LCs, and can be easily observed by the naked eye using some optical techniques [8, 9]. Heterocyclic compounds are employed in many applications, numerous researchers created liquid crystals by adding heterocyclic to the structures of these molecules [10]. It is common knowledge that the molecular structure of organic materials plays a significant role in determining their physical properties. When creating the material to create the gadget, it is quite helpful to look at the structure-activity interactions applications [11-13]. They also have amazing optical qualities that are essential for displays and other state readouts. Recently, liquid crystals have been utilized as a model system that responds to the environment's molecular makeup [14-18].

In the last two decades, the use of five-member heterocyclic rings as molecular building blocks has received a lot of interest. Recent advances [19] were evaluated and summarized to prepare the thiazolothiadiazoles. Subclasses of fused 5H-thiazolo[4,3-b]-1,3,4-thiadiazoles are formed by the relative configurations of sulfur and nitrogen atoms, as well as the numerous sites for fusion between the

thiazole and thiadiazole rings. This work includes the synthesis, structural characterization, and investigation of the liquid crystal behavior for compounds containing the 5H-thiazolo[4,3-b][1,3,4]thiadiazol, dihydro-3H-pyrazol-3-one and benzimidazol rings as part of our investigation of liquid crystal derivatives containing heterocyclic units [20-27]. Also investigate the influence of heterocyclic on the liquid crystal behavior.

MATERIALS AND METHODS

4-hydroxybenzaldehyde, and Potassium hydroxide from Fulka, Anhydrous potassium carbonate, Thioglycolic acid, Hydrazine hydrate, Sulfuric acid, Ammonia solution, Thiosemicarbazide, Ethyl acetoacetate from GPR, o-phenylene diamine, Sodium hydroxide from aldrich, and Ethanol absolute from Riedel –deHeam. ¹H-NMR spectra were measured by company: Bruker, at 400 MHz, and were reported in Part per million (ppm) Chemical shift (δ); the compounds were dissolved in dimethyl sulfoxide deuterated (DMSO d_6) solution with the tetramethylsilane (TMS) as the internal standard. FTIR spectra were recorded by SHIMADZU (IR Affinity-1) FTIR spectrometer in the wave number range 4000-600 cm^{-1} . The Gallen Kamp apparatus's measurement of melting point. The POM model Leica DM2500 M was used to examine the mesophase textures. The samples were examined using cross polarizers at normal temperature with a magnification of 10 and elevated temperature with a magnification of 20. The temperature was originally raised quickly (at a rate of 5 $^{\circ}C/min$). DSC measurements were taken using the Q600, a German-made device with an average heat rate of 5 degrees Celsius per minute.

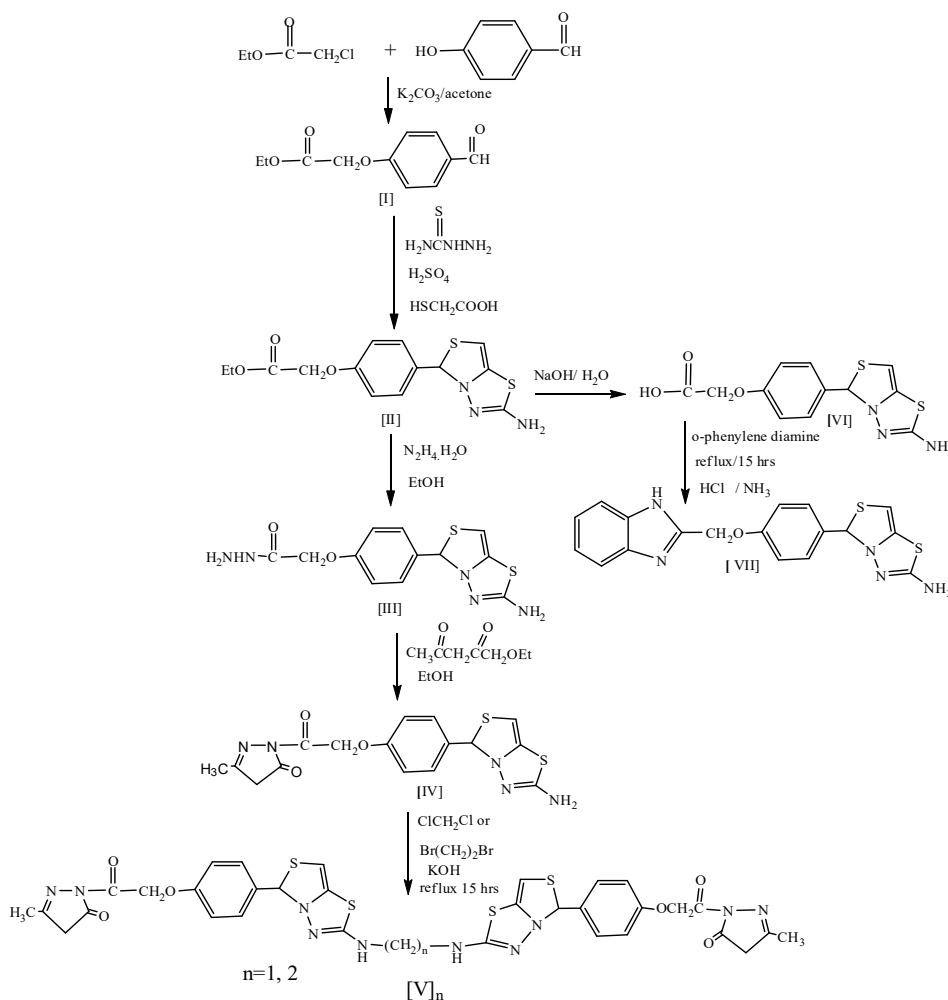


Figure 1: Scheme of Synthesis Compounds

Synthesis of 1-((2-Ethoxyallyl) Oxy)-4-Vinylbenzene [I]

Mixture of anhydrous potassium carbonate 1.31 gm, 0.001 mol with 4-hydroxy benzaldehyde 0.85 gm, 0.001 mol and, chloro ethyl acetate 0.12 gm, 0.001 mol in 5mL acetone [28], refluxing the mixture for 6 hours. After being added, the ethyl acetate to remove the organic layer, dried, and then recrystallized from ethanol to product compound [I] yield ;97%, color dark yellow, molecular formula $C_{11}H_{12}O_4$, m.p. oily. FTIR (KBr): 1755 ($C=O$ ester), 1689 ($C=O$ aldehyde), and 1600 ($C=C$) cm^{-1} . ^1H NMR (400 MHz, $DMSO-d_6$): δ 1.06-1.24 (t, 3H, CH_3), 4.11-4.88 (q, 2H, OCH_2CH_3), 4.94 (s, 2H, OCH_2), 6.71-7.93 (m, 4H, Ar-H), 9.89 (s, 1H, CHO).

Synthesis of Ethyl 2-(4-(2-Amino-5H-Thiazolo[4,3-B] [1,3,4] Thiadiazol-5-Yl) Phenoxy) Acetate [II]

Mercapto acetic acid 0.92gm, 0.01mol and compound [I] 2.08gm, 0.01mol were combined for 20–25 minutes. Thiosemicarbazide 0.91 g, 0.01 mol was added, and concentrated sulfuric acid 7.5 mL was added in portions after cooling. The mixture was placed in a freezer set at 20 °C for 24 hours. The pH was then adjusted to 7-8, using 20 g of crushed ice and 40% aqueous sodium hydroxide. The precipitate was dried and filtered [29], recrystallized from ethanol, color: dark yellow; yield: 77%; m.p. $>300^\circ C$. FTIR (KBr): 3302, 3167 (NH_2), 1735 ($C=O$ ester), 1620 ($C=N$), 1593 ($C=C$) cm^{-1} . ^1H NMR (400MHz, $DMSO-d_6$): δ 1.09-1.22 (t, 3H, CH_3), 3.43-3.75 (q, 2H, OCH_2CH_3), 3.77 (s, 2H,

OCH_2 4.83 (s, H, S-CH-N cycl.), 4.20 (s, H, S-CH=C cycl.), 6.85 (s, 2H, NH_2), 7.14-8.12 (m, 4H, Ar-H).

Synthesis of 2-(4-(2-Amino-5H-Thiazolo[4,3-B] [1,3,4] Thiadiazol-5-Yl) Phenoxy)-1-(1 λ 5-Diazanyl) Ethan-1-One [III].

Mixture of ester compound [II] 0.3 gm, 0.006 mol and hydrazine hydrate 1.5 mL, and the combination was refluxed for 3 hours in 5mL of absolute ethanol. After the mixture reached room temperature, the solvent was expelled, to give the product [30], recrystallized from ethanol, color: yellowish-green; yield: 70%; m.p. $>300^\circ C$. FTIR (KBr): 3429-3183 (NH, NH_2), 1678 ($NHC=O$), 1627 ($C=N$), and 1616 ($C=C$) cm^{-1} . ^1H NMR (400MHz, $DMSO-d_6$): δ 3.60 (s, 2 H, OCH_2), 3.77 (s, 2 H, $NHNH_2$), 3.84 (s, 2 H, NH_2), 4.57 (s, H, S-CH-N cycl.), 4.53 (s, H, S-CH=C cycl.), 6.88-8.10 (m, 4 H, Ar-H), and 8.32 (s, 1 H, $NHC=O$).

Synthesis of 2-(2-(4-(2-Amino-5H-Thiazolo [4,3-B] [1,3,4] Thiadiazol-5-Yl) Phenoxy) Acetyl)-5-Methyl-2,4-Dihydro-3H-Pyrazol-3-One [IV]

A mixture of acid hydrazide compound (III) 0.90 g, 0.0028mole, ethyl aceto acetate 0.364g, 0.0028 mole in absolute ethanol 20 mL refluxed for 4 hours. The cured product was allowed cool and be formed precipitate after that filtered then recrystallized from ethanol, color; light brown; yield 68%; m.p. 105-107°C, FTIR (KBr): 3294, 3190 (NH_2), 2982, 2890 (CH aliphatic), 1732 for ($C=O$ cycl.), 1685 ($C=O$), 1655 ($C=N$), 1608 ($C=C$) cm^{-1}

Synthesis of 2,2'-(2,2'-(((methylene or ethane-1,2-bis(azanediyl)) bis(5H-thiazolo[4,3-b] [1,3,4] thiadiazole-2,5-diyl)) bis(4,1-phenylene)) bis(oxy)) bis(acetyl)) bis(5-methyl-2,4-dihydro-3H-pyrazol-3-one) [V]₁ and [V]₂

Amine compound [IV] 0.16 gm, 0.1 mole was dissolved in 5mL of ethanol at 25°C with stirring. After the amine compound [IV] was dissolved 0.11 gm, 0.1 mole of KOH in 2 mL of distilled water was added dropwise. The reaction mixture was heated at reflux and 0.05 mole of 1,2 dichloromethane or 1,2 dibromoethane were then added. The reaction mixture was refluxed 20 hours. As the reaction proceeded, potassium bromide precipitated. From this mixture 50 mL of ethanol was removed by evaporation. The remained of reaction ethyl acetate was added to extract organic impurities, then recrystallized from ethanol. The compound [V]₁, Color: dark red; m.p. 115-118°C; yield 70%. FTIR (KBr): 3336 (NH), 2982,2854 (CH) aliphatic, 1705 (C=O in cyclic), 1685 (C=O), 1620 (C=N), 1608 (C=C).cm⁻¹. The compound [V]₂, Color : dark red; m.p. 155-157°C; yield 72%, FTIR (KBr): 3194 (NH), 2962,2895 (CH aliphatic), 1720(C=O cycl.), 1701 (C=O), 1635 (C=N), 1608 (C=C) cm⁻¹. ¹H NMR (400MHz, DMSO-d₆): δ 11.4 (s, 2H, 2NH), 6.8-8.4 (m, 8H, Ar-H), 4.1- 4.3 (2 S-CH-N) and (2 S-CH=C cycl.) and (s, 4H, 2CH₂ cycl.) , 3.38 (s, 4H, 2OCH₂) , 1.98-2.20 (s, 4H, 2CH₂) , and 1.70 (s, 6H, 2CH₃).

Synthesis of 2-(4-(2-Amino-5H-Thiazolo [4,3-B] [1,3,4] Thiadiazol-5-Yl) Phenoxy) Acetic Acid [VI]

Ester compound [II] 0.9 gm, 0.06 mole was dissolved in 25 mL 10% NaOH. The mixture was refluxed for 4 hours then cooled. The solution was acidified with 10 % H₂SO₄. 10 mL of ethyl acetate was used

to extract organic impurities, then recrystallized from ethanol, color: yellowish-green; yield: 70%; m.p. >300°C. FTIR (KBr): 3429,3180 (NH₂), 2935,2800 (CH aliphatic), 1700(C=O carboxylic acid), 1615 (C=N) and 1608 (C=C) cm⁻¹

Synthesis of 5-(4-((1H-Benzo [D] Imidazol-2-Yl) Methoxy) Phenyl)-5H-Thiazolo[4,3-B] [1,3,4] Thiadiazol-2-Amine [VII]

A mixture of carboxylic acid compound [VI] 3.09g, 10 mmole and o-phenylene diamine 1.08g, 10 mmole in 4N HCl 20mL was refluxed for 10 hours. Then the solution was neutralized with ammonia to precipitate benzimidazole [31]. The product was filtered and recrystallized from ethanol. Color: yellow; yield 72% m.p >300°C. FTIR (KBr): 3383, 3363 (NH₂), 3197 (NH), 3035(CH aromatic), 2962,2850 (CH aliphatic), 1631 (C=N), 1593 (C=C) cm⁻¹

Results and Discussion

New compounds with the 5H-thiazolo[4,3-b] [1,3,4] thiadiazol unite were subsequently synthesized as in Fig.1 and described. All of the observed FTIR and ¹HNMR spectroscopy values of the produced compounds matched the corresponding compounds' structures. Starting create 1-(2-ethoxyallyl) oxy)-4-vinylbenzene [I]. The FTIR spectrum showed stretching bands for carbonyl C=O for the ester group at (1755 cm⁻¹) and carbonyl C=O for the aldehyde group at (1689 cm⁻¹). ¹HNMR spectrum as Fig.2 showed triplet signal at δ 1.06-1.24ppm for CH₃ group and showed quartet signal at δ 4.11- 4.88 ppm for OCH₂ group, respectively also showed signal at δ 9.89 ppm due to the proton of aldehyde group.

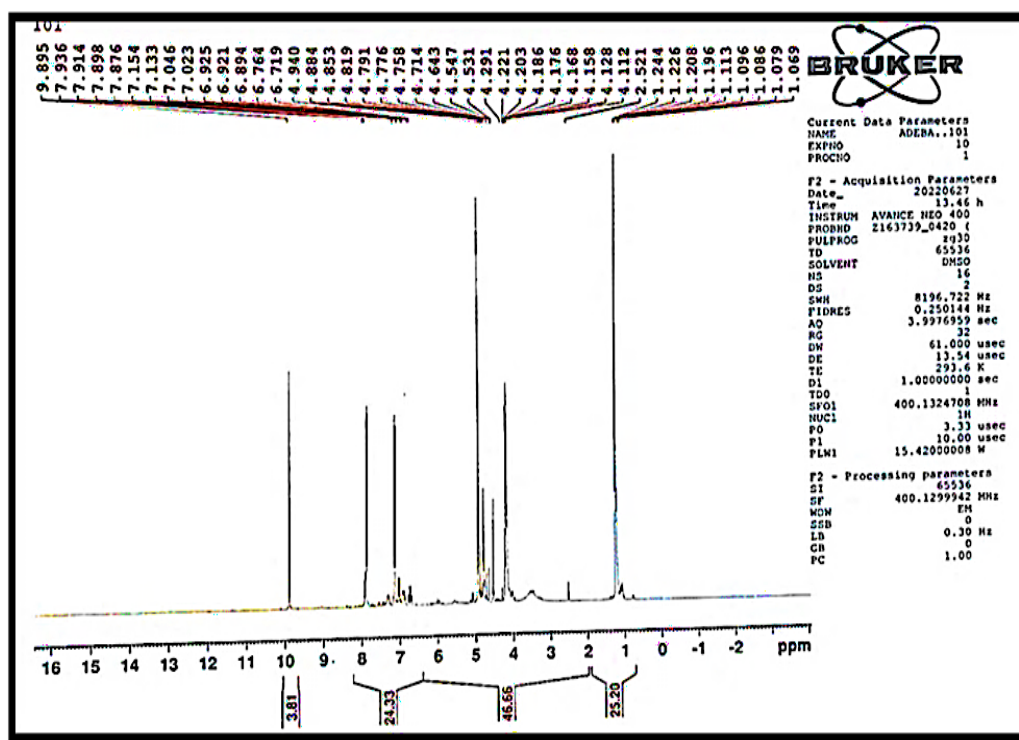


Figure 2: The ¹H NMR Spectrum of Compound [I]

From the compound [I] produce compound [II]. The FTIR spectrum showed appearance of new characteristic bands at 3302, 3167 cm⁻¹, and 1620 cm⁻¹, which belong to the NH₂ and C=N groups [32, 33], respectively. The ¹HNMR spectrum showed a peak at δ (1.19-

1.22), (3.43-3.75) ppm as triplet and quartet signals for CH₃ and OCH₂ of ester proton, in addition showed signals at δ 3.77, 4.83, 4.20 ppm for OCH₂, (S-CH-N) in cyclic, (S-CH=C) in cyclic, and signals in δ 6.85, 7.14-8.12 ppm for NH₂ and four aromatic protons respectively.

Then, compound [III] was created, the FTIR spectrum demonstrated the elimination of absorption bands of ester group and revealed the emergence of new distinctive bands for the amide group at 1678 cm^{-1} C=O and bands for the NH and NH_2 groups at $3429\text{--}3182\text{ cm}^{-1}$ [34, 35], the ^1H NMR spectrum showed signals at δ 3.60, 3.77, 3.84ppm of OCH_2 , NHNH_2 , NH_2 , respectively and signal at δ 4.57, 4.53ppm of (S-CH-N) and (S-CH=C) in cyclic in addition signals at δ 6.88-8.10, 8.32ppm of four aromatic protons and NHC=O groups, respectively. After that, the compound [IV] was product, FTIR spectrum showed bands at $3294, 3190$ and $2982, 2890\text{ cm}^{-1}$ of (NH_2) and (C-H) aliphatic also showed bands at $1732, 1685\text{ cm}^{-1}$ for (C=O) in cyclic and (C=O) group, respectively. Also, reaction compound [IV] and 1,2 dichloromethane or 1,2 dibromoethane to form compounds [V]1 and [V]2, respectively.

The FTIR spectra showed new stretching bands at $1705, 1720\text{ cm}^{-1}$ of (C=O) group in cyclic, respectively, this is good evidence to formation these compounds. ^1H NMR spectrum showed signals at 11.4 ppm for CH_3 and 6.8-8.4 ppm of Ar-H, 4.1-4.3 ppm for (2 S-CH-N) and (2 S-CH=C) in cyclic and (s,4H, 2CH_2) in cyclic, signal at 3.38 ppm for OCH_2 groups, 1.98-2.20 ppm for (CH_2) and 1.70 due to the proton

of 2CH_3 groups. In addition, hydrolysis ester compound [II] to product carboxylic acid compound [VI], The FTIR of compound [VI] showed disappeared bands of starting material and showed new stretching bands at 1700 and 3433 cm^{-1} of (C=O and OH) of carboxylic acid groups respectively. Finally, synthesized benzimidazol compound [VII], the FTIR of this compound showed disappearance bands of carboxylic group of compounds [VI] and appearance bands at $3383, 3363, 3197, 1631\text{ cm}^{-1}$ of NH_2 , NH and C=N groups, respectively.

The Liquid Crystalline Properties

The L.C. properties of compounds were investigated by polarized optical microscopy (POM) and differential scanning calorimetry (DSC). The transition temperatures and types of mesophase given in Table 1. The texture of the mesophase was identified by microscopy studies using the classification systems [36, 37]. The compound [V]₁ exhibits nematic phase only as in Fig. 3a while the compound [V]₂ is found to exhibit dimorphism smectic C (SmC) and nematic phases as in Fig. 3b and Fig. 3c, respectively. In addition, the compounds [VI] and [VII] showed nematic phase as Fig. 3d of nematic phase of compound [VII]. The DSC thermogram in heating for compound [VI] as in Fig. 4.

Table 1: Phase Transition Temperatures (oC) of Compounds[V]_{1,2} -[VII]

Comp.No.	Transition Phase
[V] ₁	Cr $\xrightarrow{190}$ N $\xrightarrow{300}$ I
[V] ₂	Cr $\xrightarrow{240}$ SmC $\xrightarrow{310}$ N $\xrightarrow{350}$ I
[VI]	Cr $\xrightarrow{264}$ N $\xrightarrow{309}$ I
[VII]	Cr $\xrightarrow{170}$ N $\xrightarrow{285}$ I

Cr, Crystalline Phase; Smc, Smectic C Phase; N, Nematic Phase And I, Isotropic

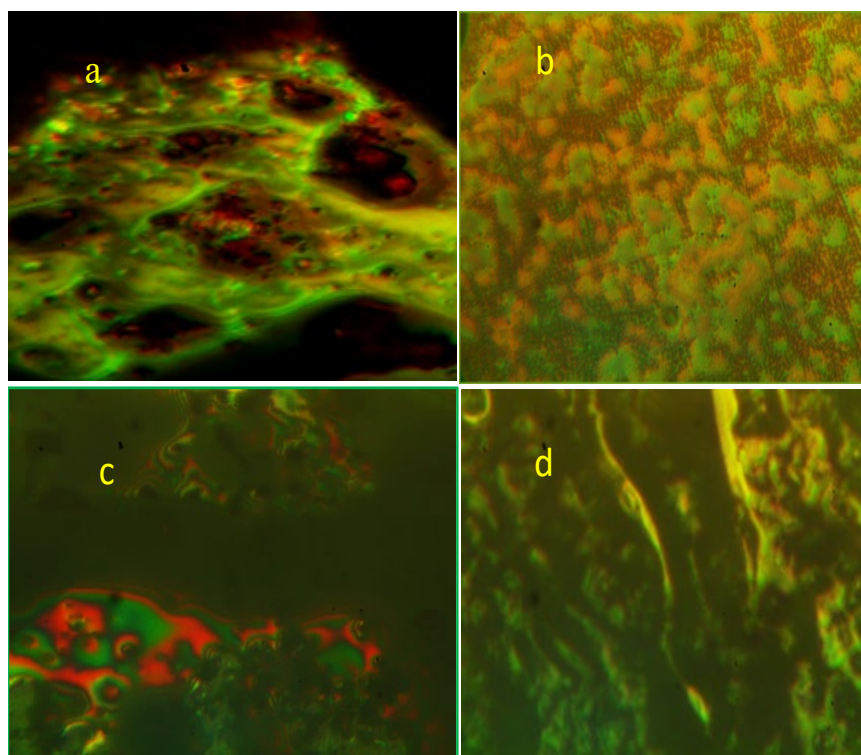


Figure 3: (a) nematic for Compound [V] 1 At Heating $275\text{ }^{\circ}\text{C}$ (B) Smectic C For Compound [V]2 At Heating $250\text{ }^{\circ}\text{C}$ (C) Nematic Phase for Compound [V]2 At Heating $320\text{ }^{\circ}\text{C}$, (D) Nematic Phase for Compound [VII] At Heating $200\text{ }^{\circ}\text{C}$.

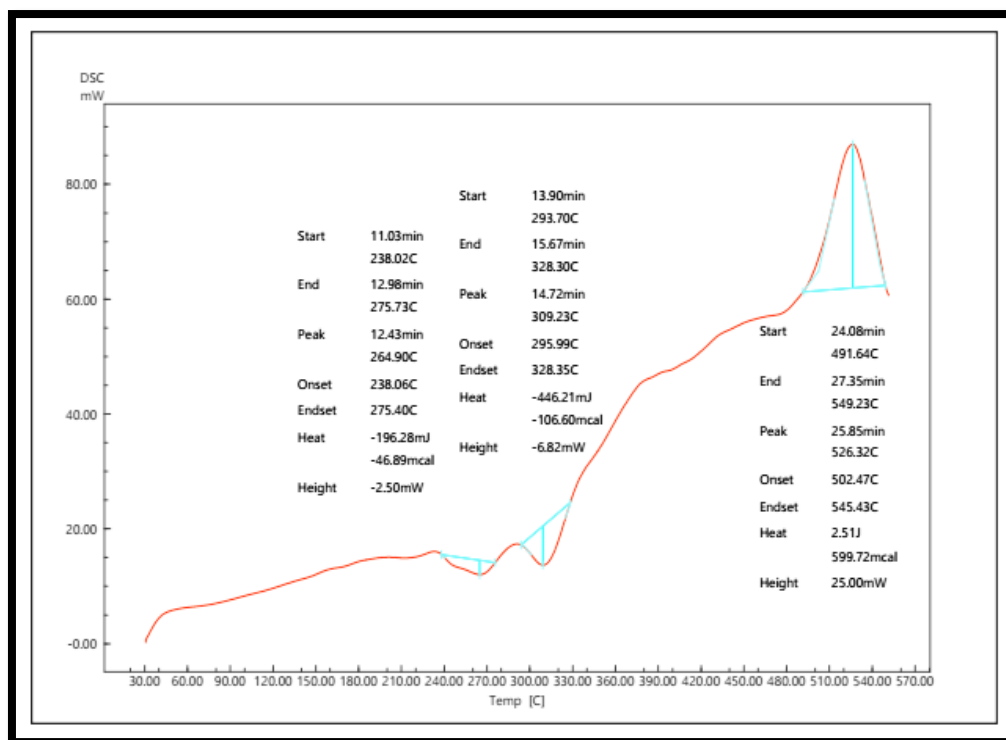


Figure 4: DSC Thermogram for Compound [VI]

Compounds [V]₁ and [V]₂ have the same molecular skeleton with thiazolo[4,3-b][1,3,4]thiadiazol unit except the number of carbonyl atoms in central core, the appearance of smectic C (SmC) beside on nematic phase of compound [V]₂ can be explained by the presence of (-CH₂CH₂-) group in this compound and this increasing the solidity and polarizability of the core and rising extended conjugated system common. Further, liquid crystal dimers, in which mesogenic groups are linked to a flexible spacer, displays a fascinating behavior that based on the descriptions of the valence, length of the spacer [38] and chemical structure. The geometrical shape of the compounds containing (CH₂)_n linking group showed ability to form mesophase,

the dimethylene bridge gives significantly higher stabilities to both the smectic and nematic phases this is agreement with another study [39] Besides, the effect of the heterocyclic on the stability of the mesophase can be considered as one of the factors contributing to the temperature difference for the transition from the mesophase to the isotropic phase, Compound [VI] shows purely nematic phase, this may be displays presence the terminal of carboxylic group that leading to the form of intermolecular hydrogen bonding. The intermolecular hydrogen bonding [40-42] leads to an extending of the rigid-rod moiety and rised the terminal molecular interaction of this group, that led to show nematic liquid crystal behavior as Fig.5.

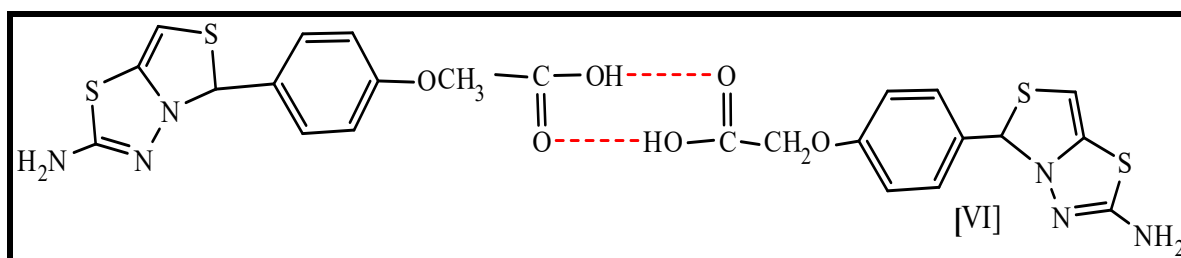


Figure 5: Intermolecular Hydrogen Bonding Formation in Compound [VI]

Conclusion

Successful multi-step preparing of novel symmetrical compounds with two arms containing 5H-thiazolo[4,3-b][1,3,4]thiadiazol and dihydro-3H-pyrazol-3-one unites and connected to the core by the number of methylene groups in the alkyl spacer, also synthesis compound including benzimidazol unite. The chemical structures of these compounds were tested by FTIR, and ¹HNMR spectroscopy. Results are accepted with the consideration molecular structure. The mesomorphic characterization and optical textures of the resulted compounds were tested by POM and DSC. Consequently, it can be

concluded that so as to manage the phase-transition properties of LC compounds, it is significant to consider the molecular geometry and the length of the flexible alkyl spacer. The compound [V]₂ shows smectic C besides to N phases, while the compounds [V]₁, [VI] and [VII] shows just N phase.

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