Liquid Crystalline Behavior and Optoelectronic Potential of Novel Schiff Base Derivatives

Jehan A. Sulaiman[1], Hanaa K. Salih[1], Mohammed M. Aftan[2] and Nagham Q. Kadhim[1]
[1]*Department of Chemistry, College of Science, Tikrit University, 34001 Tikrit, Iraq*
[2]*Department of Chemical Engineering, College of Engineering, Tikrit University, 34001 Tikrit, Iraq*
Jihan.A.Sleman.chem419@st.tu.edu.iq, h_kaain@tu.edu.iq,mohamedmizher@tu.edu.iq, Naghamkassim@tu.edu.iq

Keywords: Schiff Bases, Metformin Derivatives, Liquid Crystal, Smectic Phase, Nematic Phase.

Abstract: In this study, a new series of Schiff bases was synthesized through the condensation of azo dyes with the antidiabetic drug metformin. The structures of the synthesized compounds were confirmed using FT-IR and ^^{1}H-NMR spectroscopy, while their liquid crystalline behavior was investigated using POM and DSC techniques. The results revealed that most of the synthesized compounds exhibited Smectic-A and Nematic liquid crystalline phases upon heating, indicating monotropic behavior. This behavior is attributed to the rod-like molecular structure formed by the aromatic rings, the polarizable terminal substituents (Cl, Br), and the azomethine (C=N) and azo (N=N) groups, which enhance intermolecular interactions and molecular alignment .It was also observed that the presence of a chloro substituent provides higher thermal stability to the nematic phase compared to the bromo substituent due to its higher electronegativity and polarization effect .FT-IR results confirmed the disappearance of the characteristic bands of the primary amine and aldehyde carbonyl groups, along with the appearance of a new band corresponding to the azomethine group at 1630 cm^{-1}. Additionally, ^^{1}H-NMR spectra showed characteristic signals consistent with the proposed structures of compounds 1, 2, and 3, whereas compounds 5, 6, and 7 lacked the signal attributed to the methoxy group due to its absence in their structures. Rod-like Schiff bases with aromatic, azomethine, and azo groups formed stable smectic phases, while polarized terminal groups promoted nematic phase stability. Chlorine substitution enhanced the thermal stability of the nematic phase compared to bromine.

1 INTRODUCTION

As a definition of Liquid crystals, is a physical state that found between liquids and solids, sometime known as a fourth state of matter [1] - [3]. The Liquid crystals state have three-dimensional lattice which have a radical limits of motion. In other words, liquid crystals shown certain level of molecular order but still have the ability to flow like a liquid [4]. In liquids stste, have molecules that easy to move around freely and randomly. On the other hand, a solid-state materials, its molecules are fixed and have a perfect arranged, the systematic configuration leading to structural fidelity [5, 6]. On the other hand, Liquid crystals state have different characteristic of both solid and liquid states, its molecules shown some degree of coordination and arrangement. A Liquid crystal is adorable combination state of structure of liquidity gave it highly reactive to external influences, such as electric fields conditions or temperature changes [7]-[10]. The transition capacity between structured and unstructured states is the essential characteristic are taking place through temperature changes [11]. This makes them beneficial situations especially where heating has been controlled [12].

According to the shapes of their molecules, Liquid crystals can be divided into four general types. The first type is composed of rod-like molecules is called linear liquid crystals [13]. While the second type which have molecules shaped looks like flat discs is called discotic liquid crystals. Furthermore, Liquid crystals can be divided into four general types, the first type is linear liquid crystals, which are composed of rod-like molecules. The second type is discotic liquid crystals, with molecules shaped like flat discs. The third type of liquid crystals called a bent-core which have a particular bent shape that gives them unique polar properties [14]. The fourth type is called cubic liquid crystals which have an excessively complicated three-dimensional structure. This extent range of molecular structures authorize scientists to exactly modify the physical properties of liquid crystals for certain applications.

According to these characteristics, liquid crystals have become an excessively multilateral useful material class, being applied for the latest advanced scientific research and variety industrial uses [15]-[18] The derivatives of Phenanthrene, and their rigid polyaromatic frameworks, enhance the order of mesophases and thermal stability through improved π–π stacking interactions [19], [20] The structural synergy of these derivatives enables fine-tuning of their optical and electro-optical properties as well as supports the development of robust thermotropic liquid crystalline phases [21], [22]. Azo compounds are widely used dyes characterized by the presence of the –N=N– functional group, offering vivid colors and versatile chemical reactivity [23]. Schiff bases are formed by the condensation of primary amines with carbonyl compounds, containing the azomethine (–C=N–) group, and are known for their biological, catalytic, and liquid crystalline properties. Combining azo moieties with Schiff bases allows the design of multifunctional molecules with tunable optical and structural features [24]. The study assumes the synthesis of Schiff base compounds through the condensation reaction between azo dyes and metformin, followed by their characterization and investigation of their liquid crystalline phases.

2 MATERIALS AND METHODS

The Griffin apparatus, manufactured in London-Britain, serial number 90-01-154, was used to measure the melting point of the produced materials. The FT-IR spectra of the resultant materials were recorded using Fourier transform infrared spectrophotometer- Shimadzu 8400-S scale 4000-400 cm-^{1} with (KBr). The ultra-Shield 400 MHz Switzerl, University of Isfahan / Iran was used to register 1H-NMR. The study of liquid crystalline behavior was used the differential scanning calorimeter (DSC) - Shimadzu and the polarizing optical microscope (POM)- Beam Engineers /India.

3 GENERAL PROCEDURE

3.1 Synthesis of Diazonium Salt and Azo Compounds [25]

3.1.1 Synthesis of Diazonium Salts

In a conical flask, a 1:1 ratio of 36% HCl was mixed with water and carried out to ice bath then 0.01 mole of aniline substitutes were added to it. Stir the combinationfor 10 min. until anilinium salt is formed, and gradually add 0.690 gm 0.01 mole of sodium nitrite dissolved in 10 ml of distilled water to the mixture. Leave the combined reactantsfor 30 min. on the ice bath, while maintaining the temperature of the combinationbetween 0-5° C.

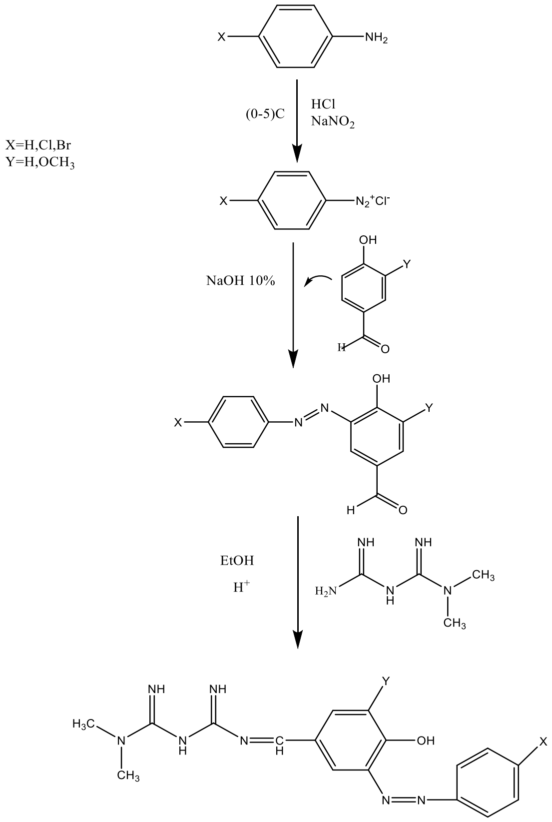


Figure 1: Synthesis of schiff bases derived from azo dye and metformin.

3.1.2 Synthesis of Conjugation Solution (Phenolic Azo Dye)

Dissolve 0.01 mole of phenol compounds in 20 ml of 10% NaOH solution in a conical flask 250 ml and place in an ice bath and stir for a few min. Then, gradually add the diazonium salt that prepared in the first step 1. Leave the prepared mixture in an ice bath for a duration of 60 minutes. With continuous stirring until the reaction is completed. Filtered and wash it with cold water until the acidity is neutralized pH=7. Then the formed precipitate was rinsed with 20 ml of petroleum ether 60-80 °C. Dry the precipitate and then recrystallize it from absolute ethanol.

3.2 Synthesis of Schiff Bases [20]

Dissolved 0.002 mole of the compound resulting from the step one 1 with 15 ml of absolute ethanol in a round-bottomed flask 100 ml, and added a few drops of glacial acetic acid with stirring for 15 min., then, 0.002 mole of metformin compound was add. The prepared mixture was refluxed for 6-8 hours. The prepared mixture was filtered, washed using some of petroleum ether 60-80 °C and dried at 40 °C. Then recrystallize it from absolute ethanol.

4 RESULTS AND DISCUSSION

As shown in Figure 1, a new series of Schiff bases were synthesized by reacting Azo dye with Metformin, and the prepared compounds were characterized by FT-IR and ^{1}H-NMR spectrum were recorded.

4.1 Identification of N1-((E)-4-hydroxy-3-methoxy-5-((E)-phenyldiazenyl) benzylidene)-N3, N3-dimethylmalonimidamide (Comp. Y1)

Properties: orange-reddish powder, Yield 77%. m.p. = 85-88 °C.

IR cm^{-1} (KBr): 3393 cm^{-1} (N-H), (3165-3177) cm^{-1} (C-H Aromatic), 3102 cm^{-1} (C-H Aliphatic), 1632 cm^{-1} (C=N), 1598 cm^{-1} (C=C Aromatic).

^{1}H NMR (500 MHz, DMSO-d6) δ 9.46 (s, 1H)→OH , 8.86 (s, 1H) →C≡N, 8.38 (s, 1H) →=N-H(c), 7.97 (d, J = 2.1 Hz, 1H), 7.68 – 7.63 (m, 2H), 7.58 – 7.52 (m, 2H), 7.47 – 7.41 (m, 2H) →Aromatic ring, 6.24 (s, 1H) →=N-H(b), 6.19 (s, 1H) →=N-H(a), 3.94 (s, 3H) →OCH3, 2.97 (s, 6H) →2CH3. As shown in Figure 2.

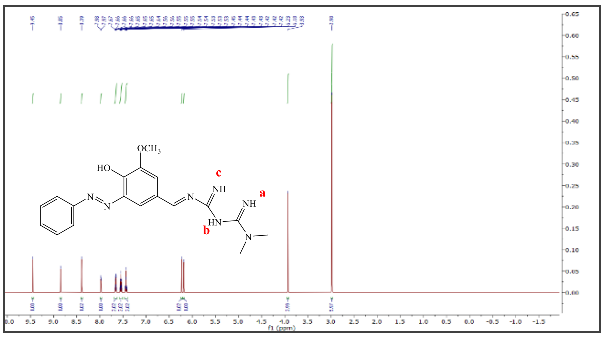


Figure 2: ^{1}HNMR of comp. Y1.

4.2 Identification of N1-((E)-3-((4-bromophenyl) diazenyl)-4-hydroxy-5-methoxybenzylidene)-N3, N3-dimethylmalonimidamide (Comp. Y2)

Properties: brown powder, Yield 84%. m.p. = 136-139 °C,

IR (KBr): 3372 cm^{-1} (N-H), 3163 cm^{-1} (C-H Aromatic), (2971-2932) cm^{-1} (C-H Aliphatic), 1628 cm^{-1} (C=N), (1588-1507) cm^{-1} (C=C Aromatic).

^{1}H NMR (500 MHz, DMSO-d6) δ 9.38 (s, 1H), 8.78 (s, 1H), 8.32 (s, 1H), 7.91 (d, J = 2.1 Hz, 1H), 7.68 –7.56 (m, 4H), 7.34 (s, 1H), 6.16 (s, 1H), 6.10 (s, 1H), 3.84 (s, 3H), 2.91 (s, 6H). As shown in the Figure 3.

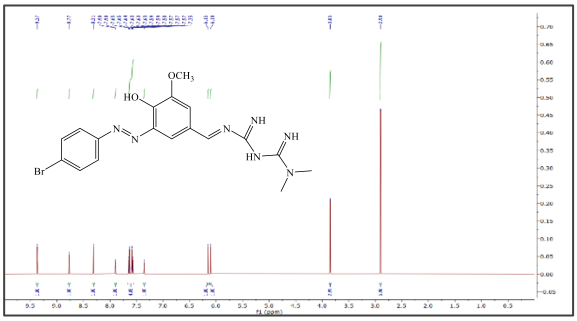


Figure 3: 1HNMR of Comp. Y2.

4.3 Identification of N1-((E)-3-((4-chlorophenyl) diazenyl)-4-hydroxy-5-methoxybenzylidene)-N3, N3-dimethylmalonimidamide (Comp. Y3)

Properties: deep yellow powder, Yield = 89%. m.p. = 131-133 °C

IR (KBr): 3394 cm^{-1} (N-H), 3162 cm^{-1} (C-H Aromatic Sp_2), (2971-2936) cm^{-1} (C-H Aliphatic), 1628 cm^{-1} (C=N), (1578-1571) cm^{-1} (C=C Aromatic).

^1H NMR (500 MHz, DMSO-d6) δ 9.45 (s, 1H), 8.85 (s, 1H), 8.37 (s, 1H), 7.98 (d, J = 2.1 Hz, 1H), 7.85 – 7.76(m, 2H), 7.51– 7.46(m, 2H), 7.44(d, J = 2.2 Hz, 1H), 6.23 (s, 1H), 6.18 (s, 1H), 3.93(s, 3H), 2.98 (s, 6H). As shown in the Figure 4.

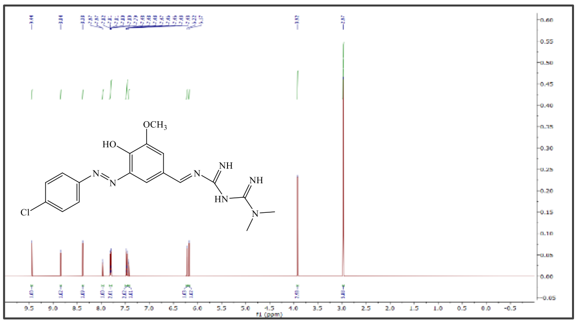


Figure 4: 1HNMR of Comp. Y3.

4.4 Identification of N1-((E)-3-((E)-(4-chlorophenyl) diazenyl)-4-hydroxybenzylidene)-N3, N3-dimethylmalonimidamide (Comp. Y4)

Properties: deep green powder, Yield = 94%. m.p. = 127-130 °C
IR (KBr):3394cm^{-1}(N-H), 3096 cm^{-1} (C-H Aromatic), 1632 cm^{-1} (C=N), (1596-1513) cm^{-1} (C=C Aromatic).
^1H NMR (500 MHz, DMSO-d6) δ 9.01 (s,1H)→OH, 8.41 (s,1H) →=N-H(c), 8.16 (d,J = 2.0 Hz,1H) →=N-H(b), 7.98 – 7.63 (m, 3H), 7.63–7.07 (m, 3H) →Aromatic ring, 6.24(d, J = 56.4 Hz, 2H) →=N-H(a), 2.98 (s, 6H) →2CH3. As shown in Figure 5.

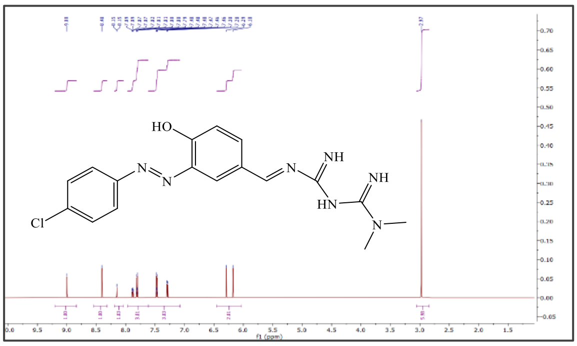


Figure 5: 1HNMR of Comp. Y4.

4.5 Identification of N1-((E)-3-((E)-(4-bromophenyl) diazenyl)-4-hydroxybenzylidene)-N3, N3-dimethylmalonimidamide (Comp. Y5)

Properties: deep brown powder, Yield = 73.3%. m.p. = 149-152 °C
IR (KBr):3372cm^{-1}(N-H), 3164 cm^{-1} (C-H Aromatic), 1632 cm^{-1} (C=N), (1588-1511) cm^{-1} (C=C Aromatic).
^1H NMR (500 MHz, DMSO-d6) δ 8.96 (s, 1H), 8.36 (s, 1H), 8.11 (d, J = 2.0 Hz, 1H), 7.84 (dd, J = 8.1, 2.0 Hz, 1H), 7.72 – 7.52 (m, 4H), 7.23 (d, J = 8.1 Hz, 1H), 6.19 (d, J = 56.4 Hz, 2H), 2.93 (s, 6H). As shown in Figure 6.

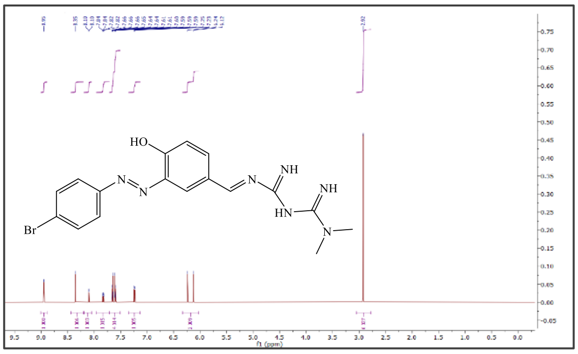


Figure 6: 1HNMR of Comp. Y5.

4.6 Identification of N1-((E)-4-hydroxy-3-((E)-phenyldiazenyl) benzylidene)-N3, N3-dimethylmalonimidamide (Comp. Y6)

Properties: deep brown powder, Yield = 95%. m.p. = 142-145 °C.

IR (KBr):3316cm^{-1}(N-H), 3175 cm^{-1} (C-H Aromatic), 1632 cm^{-1} (C=N), (1582-1474) cm^{-1} (C=C Aromatic).

^1H NMR (500 MHz, DMSO-d6) δ 9.09 (s, 1H), 8.47 (s, 1H), 8.24 (d, J = 2.0 Hz, 1H), 7.97 (dd, J = 8.1, 2.0 Hz, 1H), 7.82 – 7.68 (m, 2H), 7.69 –7.57 (m, 2H), 7.57 – 7.48 (m, 1H), 7.38 (d, J = 8.1 Hz, 1H), 6. 38 (s, 1H), 6.26 (s, 1H), 3.04 (s, 6H). As shown in Figure 7.

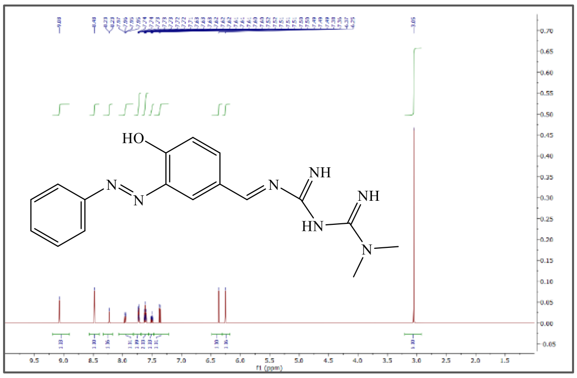


Figure 7: 1HNMR of Comp. Y6.

4.7 Identification the Liquid Crystalline Behavior

The liquid crystal phases and thermal transitions were studied using a polarizing optical microscope (POM) and a differential scanning calorimeter (DSC), by taking a sample quantity of (0.05–0.1 g) [26]. The thermal transitions were monitored by heating the sample in the POM at a rate of 5–8 °C/min. The results of POM and DSC showed that most of the compounds in this series exhibited liquid crystalline phases with high thermal transitions, as presented in Table 1 and Figures 8 - 19. It was observed that the compounds containing polarized terminal groups such as (Cl, Br) exhibited two types of liquid crystalline phases: smectic (SA type) and nematic. The results also revealed that the thermal stability of the nematic phase increased in the order (Cl > Br) [27]. It was concluded that most of the prepared compounds exhibited liquid crystalline properties only upon heating, i.e., they were of the monotropic type, with relatively stable smectic phases over a wide temperature range in addition to a broad nematic phase [28]. The study also demonstrated consistency between the results obtained from DSC and those obtained from POM analyses [29].

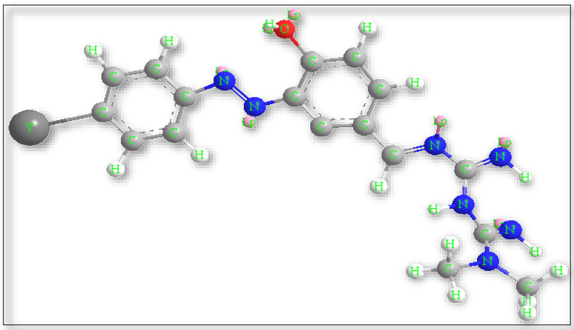
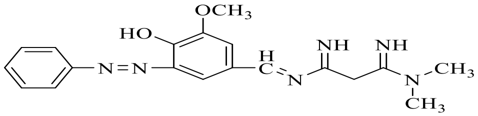
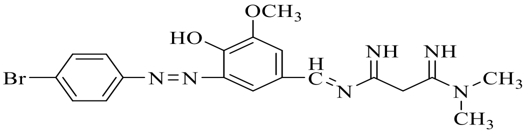
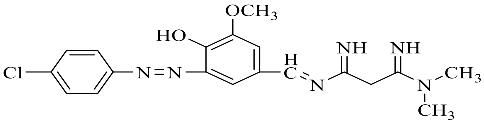
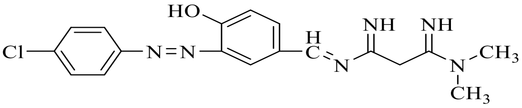
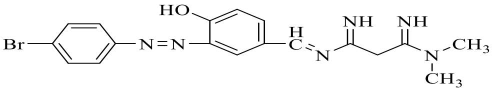
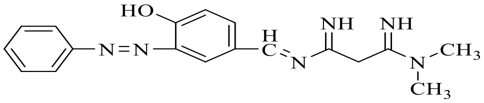


Figure 8: Three-dimensional spatial structure of the prepared Schiff bases.

Table 1: Thermal transitions and liquid crystalline phases of the prepared compounds.

Comp.	Heat	Cr	SA	N	ΔSA	ΔN	
Y1	DSC						
	POM	86					
Comp.	Heat	Cr	SA	N	ΔSA	ΔN	
Y2	DSC	138	188	209	50	21	
	POM	137	186	208	49	22	
Comp.	Heat	Cr	SA	N	ΔSA	ΔN	
Y3	DSC	135	154	182	19	28	
	POM	136	157	183	21	26	
Comp.	Heat	Cr	SA	N	ΔSA	ΔN	
Y4	DSC	134		171		37	
	POM	133		173		40	
Comp.	Heat	Cr	SA	N	ΔSA	ΔN	
Y5	DSC	153	174	205	21	31	
	POM	153	172	206	19	34	
Comp.	Heat	Cr	SA	N	ΔSA	ΔN	
Y6	DSC						
	POM	143					

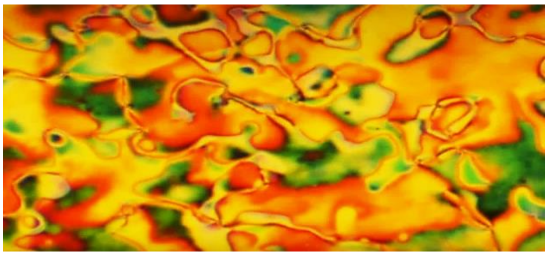


Figure 9: The SA phase of Comp. (Y2).

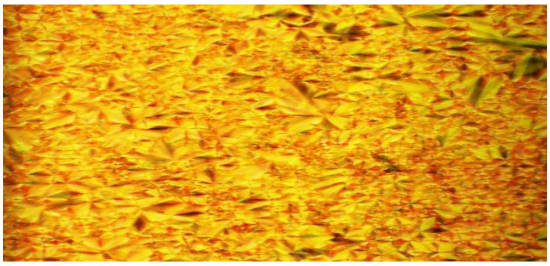


Figure 10: The SA phase of the Comp. (Y3).

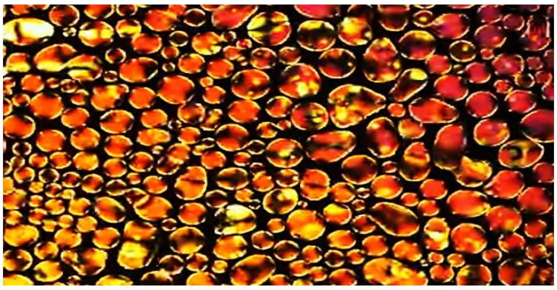


Figure 11: The nematic phase N of Comp. (Y2).

Figure 12: The nematic phase N of Comp. (Y3).

Figure 13: The nematic phase N of Comp. (Y5).

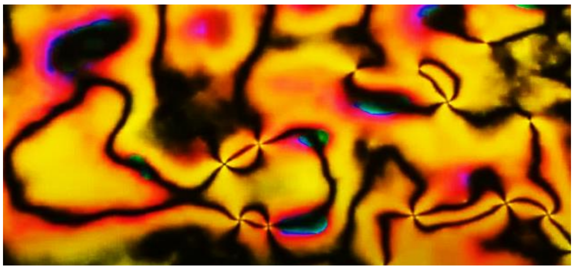


Figure 14: The SA phase of the Comp. (Y6).

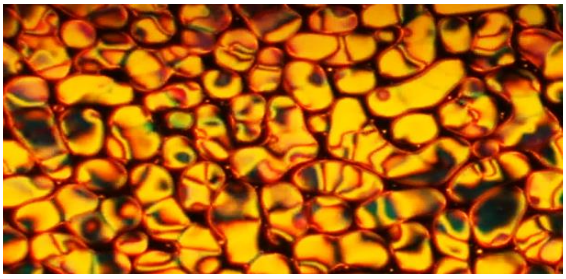
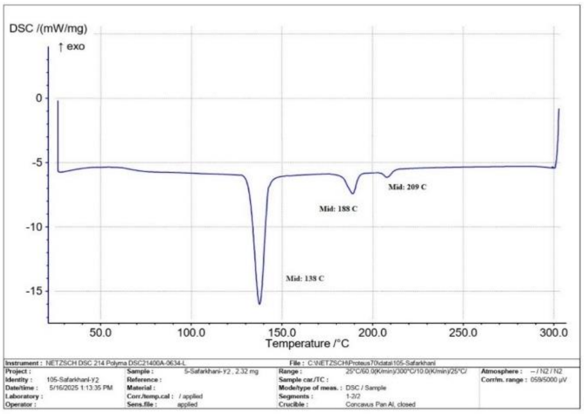


Figure 15: The nematic phase N of Comp. (Y6).



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Laboratory :
Operator :
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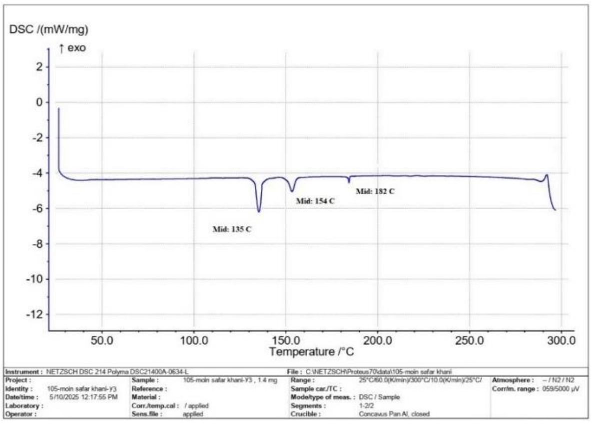
Reference :
Material :
Corr./temp.cal : / applied
Sens.file : applied
Range : 25°C/60.0(K/min)/300°C/10.0(K/min)/25°C/
Sample car./TC :
Mode/type of meas. : DSC / Sample
Segments : 1-2/2
Crucible : Concavus Pan Al, closed
Atmosphere : -- / N2 / N2
Corr/m. range : 059/5000 µV

Figure 16: DSC measurement of Comp. (Y2).



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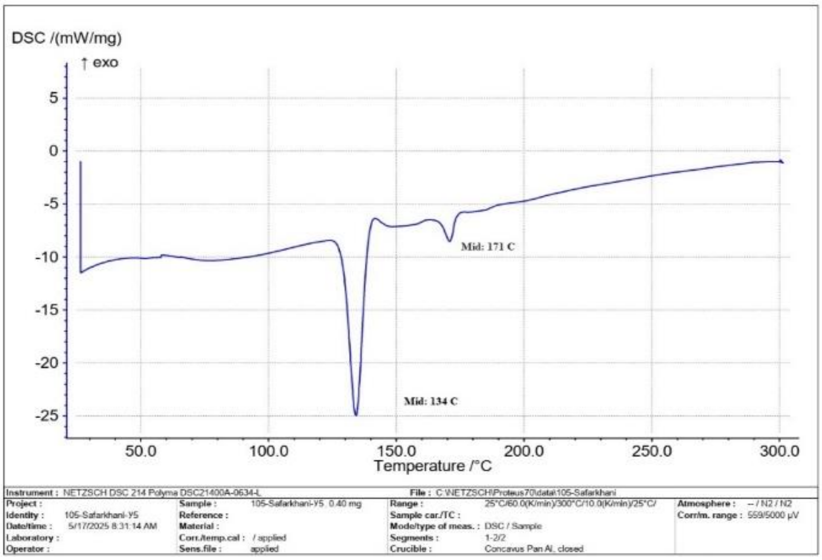
Reference :
Material :
Corr./temp.cal : / applied
Sens.file : applied
Range : 25°C/60.0(K/min)/300°C/10.0(K/min)/25°C/
Sample car./TC :
Mode/type of meas. : DSC / Sample
Segments : 1-2/2
Crucible : Concavus Pan Al, closed
Atmosphere : -- / N2 / N2
Corr/m. range : 059/5000 µV

Figure 17: DSC measurement of Comp. (Y3).



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Operator :
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Reference :
Material :
Corr./temp.cal : / applied
Sens.file : applied
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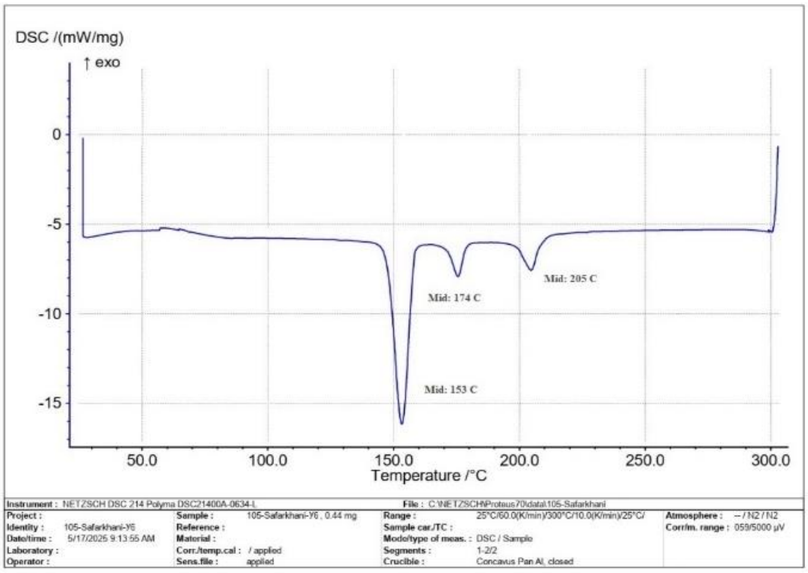
Sample car./TC :
Mode/type of meas. : DSC / Sample
Segments : 1-2/2
Crucible : Concavus Pan Al, closed
Atmosphere : -- / N2 / N2
Corr/m. range : 559/5000 µV

Figure 18: DSC measurement of Comp. (Y5).



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Project :
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Laboratory :
Operator :
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Reference :
Material :
Corr./temp.cal : / applied
Sens.file : applied
Range : 25°C/60.0(K/min)/300°C/10.0(K/min)/25°C/
Sample car./TC :
Mode/type of meas. : DSC / Sample
Segments : 1-2/2
Crucible : Concavus Pan Al, closed
Atmosphere : -- / N2 / N2
Corr/m. range : 059/5000 µV

Figure 19: DSC measurement of Comp. (Y6).

5 DISCUSSION THE LIQUID CRYSTALLINE BEHAVIOR

These results can be attributed to the fact that the compounds were designed in a way that gives them a rod-like molecular shape with high regularity, as the molecules contain a core composed of two aromatic rings which increases the rigidity of the molecule [30]. The evidence of bonding groups including the azomethine group (C=N) and the azo group (N=N) enhances the electronic conjugation along the molecular axis, giving the molecules a planar structure that promotes stacking and regular arrangement. This leads to the formation of well-ordered smectic phases [31]. Such molecular organization increases the lateral intermolecular forces, thereby supporting the appearance and stability of the smectic liquid crystalline phases. Moreover, the presence of polarized terminal groups enhances intermolecular attractive forces, which favors the emergence and stabilization of the nematic phase. The increased thermal stability of the nematic phase in compounds containing chlorine compared to bromine can be explained by the higher electronegativity of chlorine [32], which increases polarization and terminal bonding forces, thereby positively affecting the thermal stability of the nematic phase.

6 CONCLUSIONS

A new series of Schiff bases was successfully synthesized via the condensation of metformin with azo dyes, and their structures were confirmed using spectroscopic techniques. Most of the synthesized compounds exhibited monotropic liquid crystalline behavior, forming stable Nematic and Smectic-A phases upon heating. The presence of linkage groups (C=N and N=N), aromatic cores, and polar substituents contributed to enhanced molecular planarity and efficient stacking, which promoted the formation of liquid crystalline phases. Furthermore, terminal substituents (Cl and Br) significantly influenced phase stability, with chloro-substituted compounds displaying higher thermal stability of the Nematic phase compared to bromo-substituted analogs. The observed mesophase transitions were confirmed, and excellent agreement was found between POM and DSC analyses, validating the consistency of the experimental results.

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