



Synthesis of A New Series of Benzothiazole Compounds and Study of their Liquid Crystal Properties

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ABSTRACT

A series of benzothiazole Schiff bases with different benzyloxy chain lengths (4-alkoxy-benzylidene)-(6-pentyloxy-benzothiazol-2-yl)-amines are synthesized possessing different carbon chains at the alkoxy substituent ($O-C_nH_{2n+1}$, $n = 1$ to 6). Synthesized compounds are thoroughly characterized using proton nuclear magnetic resonance (1H -NMR) and Fourier-transform infrared spectroscopy (FT-IR). Differential polarizing optical microscopy (POM) and scanning calorimetry (DSC) are used to investigate the mesomorphic phase behavior of the synthesized Schiff bases. Liquid crystal phases have been observed for each compound within the series ($n = 1$ to 5). All of the substances show both the N and SmC phases, particularly when $n=6$, the N phase is not visible, and only the SmC phase is present.

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Keywords: Benzothiazole, Liquid crystals, Mesophase properties, Schiff bases.

1. Introduction

Liquid crystals (LCs) are a novel type of organic semiconductor that can be utilized to construct organic light-emitting diodes^[1]. Studies have been conducted on mesomorphic behavior and how it relates to the structure and properties of many different liquid crystalline symmetrical and unsymmetrical dimers and trimers with aromatic cores. Investigations have been done on aromatic cores that are connected by aliphatic spacer groups and also on those that have aliphatic chains situated at the ends^[2, 3]. Most of segmented dimers have a nematic phase and have been studied as models for the corresponding oligomers and polymers to study odd-even effects as a function of molecular shape and conformation^[4, 5]. Research has been done on the production of heterocyclic compounds with a range of functional groups and lengths of chains^[6, 7]. Heterocyclic compounds have intriguing properties that could be used in various applications, such as drugs (biologically active compounds), polymers, semiconductors, film photonics, and liquid crystalline properties^[8-11]. Recently, synthetic chemists have been drawn to the liquid-crystalline behavior of these compounds to prepare different functional groups in heterocyclic compounds and study their liquid-crystalline properties^[12]. Compounds having liquid crystalline characteristics can be utilized to produce semiconductors and other electronic materials. Many aspects can affect liquid crystalline properties, such as the shape and

geometry of molecules, their firmness, and the general arrangement of molecules^[13]. The existence of heterocycle rings (benzothiazole, pyridine, oxadiazole) has a substantial influence on crystalline features as well^[14]. Furthermore, the presence of additional functional groups and the presence of a long methylene chain may influence such properties. Benzothiazole compounds are among the most frequently chosen heterocycle compounds for their liquid crystalline properties. A series of benzothiazole-derived compounds was synthesized in 1976 and demonstrated nematic mesomorphic properties^[15]. Funa Hashi and J. Hana et al. highlighted in a separate investigation that the Benzothiazole ring is beneficial in eliciting a sematic phase due to the sulfur atom's electron-rich trait^[16]. Ha et al. revealed the formation of Schiff bases with benzothiazole rings (3-hydroxy-4-[(6-methoxy-1,3-benzothiazol-2-yl)iminol]methylphenyl palmitate) having similar characteristics^[17].

In this study, we created a new series of Schiff base compounds with varying benzyloxy chain lengths containing the benzothiazole ring. Our Schiff bases were synthesized by combining a 6-Pentyloxy-benzothiazol-2-ylamine unit with p-alkoxy aldehydes to produce the desired compounds.

2. Materials and Procedures

2.1 Experimental

All starting materials were purchased from a Merck company supplier in the highest purity. Electrothermal was used to determine the melting points of the synthesized compounds. Shimadzu KBr discs were used to record FT-IR spectra

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(Chemistry Department, College of Education Science, Salahaddin University/Erbil). Bruker 400 MHz (CDCl₃ solvent) was utilized to record ¹H-NMR spectra. Several techniques, such as Polarizing Optical Microscopy (POM), Differential Scanning Calorimetry (DSC), Differential Thermal Analysis (DTA), X-Ray Diffraction (XRD), and Raman spectroscopy, were employed for the study of liquid crystal properties.

2.2 Synthesis of 4-pentyloxy-acetaldehyde (2)

Potassium hydroxide (1.75 g, 31.25 mmol in 2 mL) was added slowly (during 2 min) to an ethanolic solution of *p*-hydroxy acetanilide (3.75 g, 24.80 mmol in 20 mL) at 0-5 °C, followed by the addition of an ethanolic solution of pentyl bromide (4.12 g, 27.31 mmol in 6 mL). The mixture was gradually allowed to reach r.t and then heated under reflux for 3h with continuous TLC monitoring to confirm the reaction completion. The product was added to a small beaker containing 6 mL of cold water to give the precipitate of the product. The filtrate was precipitated, neutralized by washing with ice-cold water to produce the product, and purified by recrystallization with a 2:1 ethanol-water solution, giving a white solid of 4-benzyloxy-acetanilide (4.52 g, 75%) with a melting point of 86.3-87 °C.

2.3 Synthesis of 4-pentyloxy-aniline (3)

Potassium hydroxide (4mL, 20 mmol) was added slowly (during 2 min) to an ethanolic soln. of 4-pentyloxy-acetaldehyde (2.50 g, 11.29 mmol in 15 mL) at r.t. The reaction was refluxed for 5 hrs. Once TLC had verified that the reaction was finished, the ethanol was removed and 5 mL of water was added. The product was extracted with benzene (3 × 15 mL), the combined organic layers were dried over MgSO₄, and the solvent was rota-evaporated to give the product 4-pentyloxy-aniline (1.62 g, 80%) as a pale reddish liquid, which showed identical spectral data to the literature^[18].

2.4 Pentyloxy-benzothiazol-2-ylamine (4)

In a 2-neck 100 mL round bottom flask, 4-pentyloxy-aniline (1.00 g, 5.57 mmol) and potassium thiocyanate (1.62 g, 16.74 mmol) were dissolved in (20 mL) glacial CH₃COOH and cooled to below 5 °C. Bromine solution (1.78 g, 11.16 mmol) in glacial CH₃COOH (4 mL) was added to the above solution dropwise over 20 minutes maintaining temperatures below 5 °C. The reaction mixture was allowed to reach r.t and then stirred at room temperature for 24 hrs. An ammonia solution of 6 mL was used to neutralize the reaction mixture, which was then added to a beaker that contained 100 mL of hot water. The product was extracted with EtOAc (3 × 15 mL), and the collected organic layers were dried over magnesium sulfate, and the solvent was rota-evaporated to give the crude, purified by re-crystallization with benzene to give a yellow-brownish solid of 6-pentyloxy-2-amino benzothiazole (0.85 g, 65%); m.p 95-95.2 °C.

2.5 General procedure for synthesizing 4-alkoxy-benzaldehyde (6a-f)

4-alkoxy benzaldehyde (n=5 to 10) was obtained by refluxing 4-hydroxy benzaldehyde (1 equiv.) with alkyl bromide "(n=5 to 10, 1.2 mol. eq.)" in the appropriate amount of cyclohexanone in the presence of potassium carbonate "(4 mol. eq.) at 78 °C for 3 hrs"

with continuous TLC monitoring. The residue was filtered and washed with the appropriate amount of diethyl ether several times. The filtrate was distilled to eliminate the solvent, resulting in a white semi-solid of 4-alkoxybenzaldehyde as the desired product. Yields of (94, 91, 93, 87, 90, 93%) were obtained for n=5 to 10 respectively.

2.6 Synthesis of (4-alkoxy-benzylidene)-(6-pentyloxy benzothiazol-2-yl)-amine (7a-f)

(4-alkoxy-benzylidene) -(6-pentyloxy-benzothiazol-2-yl)-amine (7a-f, n=5 to 10) were synthesized by refluxing 4-alkoxy benzaldehyde (6a-f, n=5 to 10, 1 equiv.) with benzothiazole (4, 1.05 equiv.) in appropriate amount of ethanol as a solvent with few drops of glacial CH₃COOH at 70 °C for 3 hrs. When TLC showed no starting material was left, the solvent was rota-evaporated to give the crude product, purified by recrystallization in Ethanol/water (3:1) to get a bright yellow solid of the Schiff bases (7a-f) The yield gave (71, 74, 74, 73, 70, 75%) for n=5 to 10 respectively.

3. Results and Discussion

3.1 characterization of Synthesized compounds

The benzothiazole (4) (Figure 1) was synthesized starting from *p*-hydroxy acetanilide (1) which is known as acetaminophen. It was reacted with pentyl bromide via the standard Williamson method to produce the pentyloxy acetanilide (2). The hydrolysis of the acetanilide group was performed using the ethanolic solution of potassium hydroxide and reflux for 5 hrs to produce *p*-pentyloxy aniline (3) in 80% yield. Finally, cyclization of (3) was conducted using potassium thiocyanate, and bromine solution in acetic acid and stirred vigorously at r.t for 24 hrs to give the desired benzothiazole unit (4) in 65% yield. The products are characterized by FT-IR and ¹H-NMR techniques.

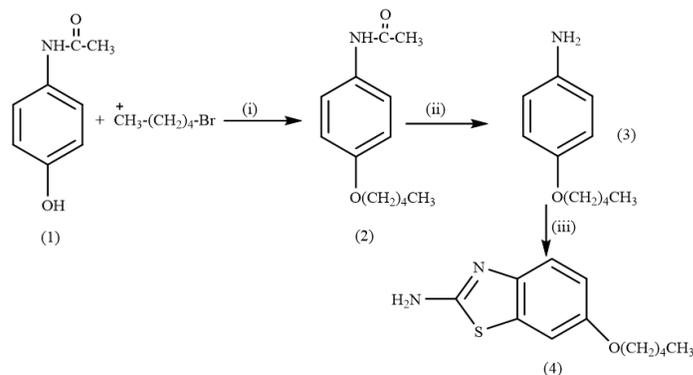
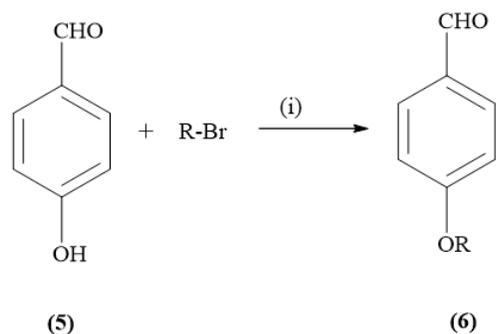


Figure 1: Synthesis of 6-pentyloxy-2-amino benzothiazole: (i) R-Br, KOH, ethanol, reflux (3 hrs) 82%; (ii) KOH, ethanol, reflux (5 hrs) 80%; (iii) KSCN, Br₂, CH₃COOH, (24 hrs), 65%.

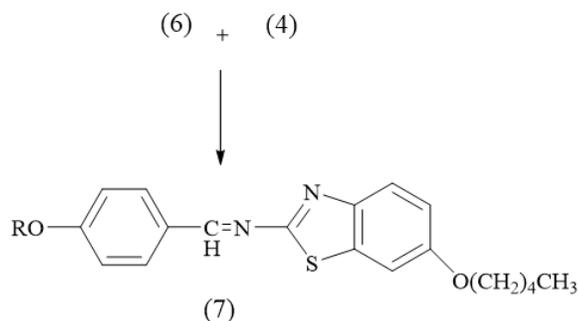
p-alkoxy aldehydes (6a-6f) unit with other chain lengths (R= 5, 6, 7, 8, 9, and 10) was obtained by reacting of *p*-hydroxy benzaldehyde with different alkyl bromides again by using the standard Williamson method to produce the *p*-alkoxy aldehydes (6a-6f) (Figure 2).



R= a=(CH₂)₅, b=(CH₂)₆, c=(CH₂)₇, d=(CH₂)₈, e=(CH₂)₉, f=(CH₂)₁₀

Figure 2: Synthesis of p-alkoxy benzaldehyde: (i) R-Br, K₂CO₃, cyclohexanone, reflux (3 hrs).

Finally, the desired Schiff bases were produced by refluxing of 6-Pentyloxy-benzothiazol-2-ylamine unit (4) with p-alkoxy aldehydes (6a-6f) and a few drops of acetic acid for 3 hrs. (7a-7f). Figure (3). The success of the reactions was confirmed using both ¹H-NMR and FT-IR spectroscopy as shown in Table 1. The ¹H-NMR spectrum (Figure 4) belongs to compound 7d, which shows clearly the expected peaks belong to the protons of the final target compound. The ¹H-NMR spectrums of compounds 7a-7f were similar to compound 7d, apart from the difference in the chain length of the alkoxy group of two protons for each



R= a=(CH₂)₅, b=(CH₂)₆, c=(CH₂)₇, d=(CH₂)₈, e=(CH₂)₉, f=(CH₂)₁₀

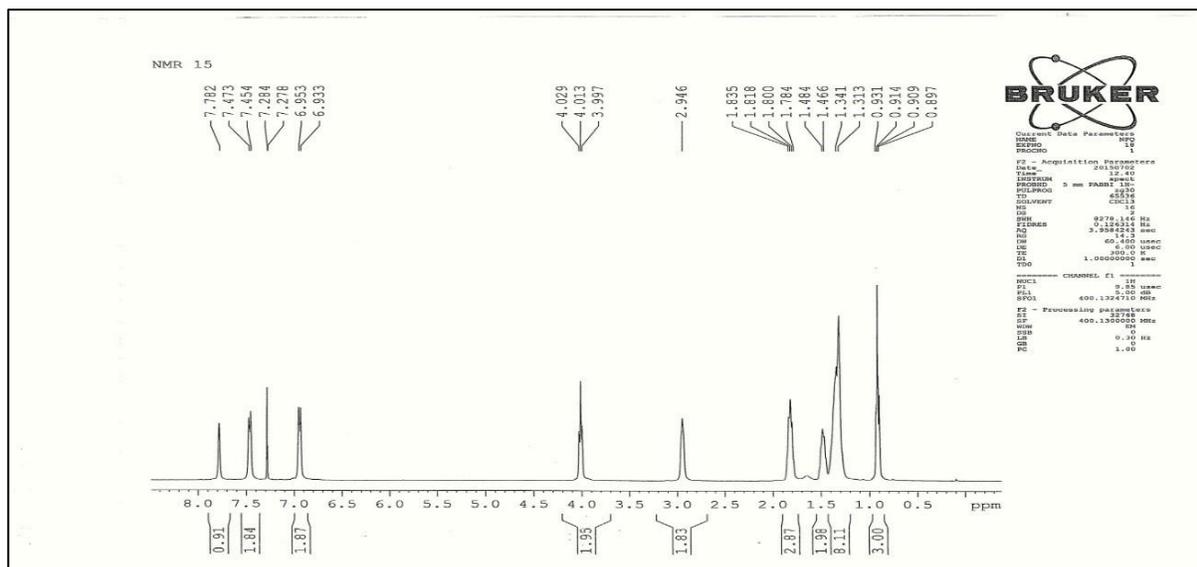
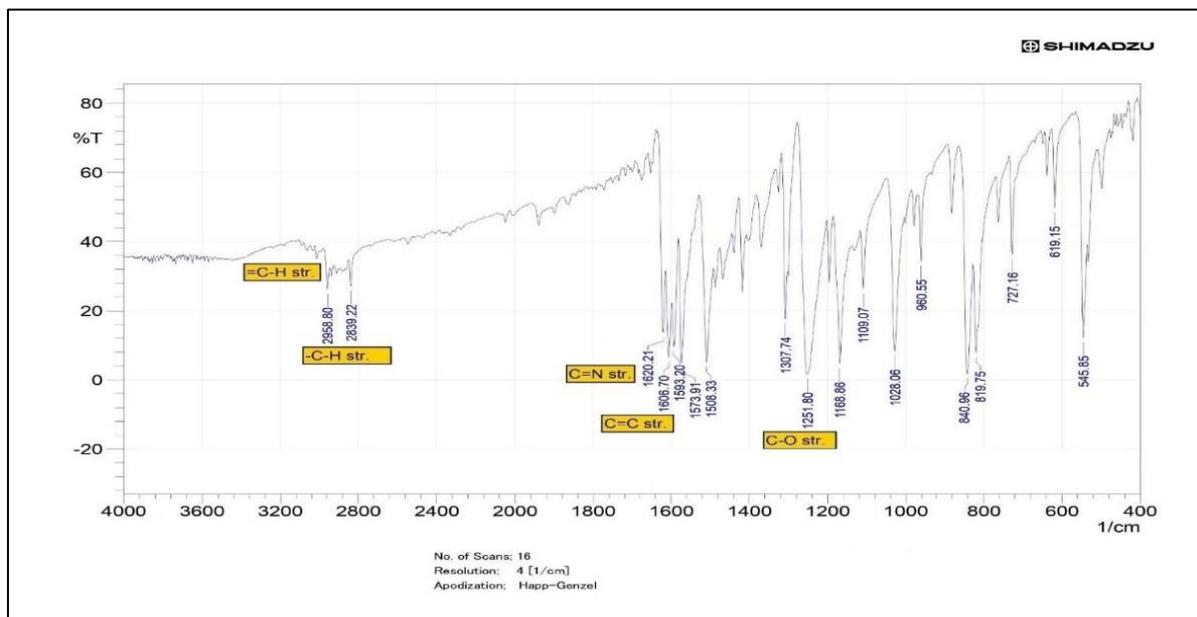
Figure 3: Synthesizing lead molecule: (4-alkoxy-benzylidene)-(6-pentyloxy-benzothiazol-2-yl)-amine (7a-f): Reagents and conditions: (i) ethanol, CH₃COOH, reflux (3 hrs).

Table 1: ¹H-NMR chemical shift of the compounds (6,7a, 7b, 7c, 7d, 7e, and 7f).

Comp.	R	Chemical shifts in ppm
6		m.p 95-95.2 °C; δ_{H} : 7.437(1H, d J 8.8 Hz, Ar-H), 7.134(1H, d J 2.4 Hz, Ar-H), 6.925 (1H, dd J 8.8, 2.4 Hz, Ar-H), 5.387(2H, s, NH ₂), 3.985 (2H, t J 6.8 Hz, -OCH ₂), 1.841 (2H, quint. J 6.8 Hz, -OCH ₂ -CH ₂), 1.46 (4H, m, OCH ₂ -CH ₂ -CH ₂ -CH ₂ -), 0.97(3H, t J 6.8Hz, OCH ₂ -CH ₂ -CH ₂ -CH ₂ -CH ₃); ν_{max} : 3429, 3290 NH(m), 3080 C-H AROMATIC (w), 2953 C-H ALIPHATIC(s), 2872 (m) C-H ALIPHATIC, 1635 C=N S, 1604 C=C AROMATIC S, 1537 N-H BENDING AMIDE II, 1460, 1388 CH ₃ UMBRELLA, 1276, 1205 C-N, 1078 C-O-R, 1058, 846, 808, 646 cm ⁻¹ .
7a	-(CH ₂) ₅	7.779 (1H, s, CH=N), 7.473 (3H, d J 8.4 Hz, Ar-H), 6.953 (4H, d J 8.4 Hz, Ar-H), 4.014 (4H, t J 6.8 Hz, -OCH ₂), 2.943 (4H, br. t J 5.6 Hz, -OCH ₂ -CH ₂), 1.827 (4H, quint. J 6.4 Hz, -OCH ₂ -CH ₂ -CH ₂), 1.46 (4H, m, OCH ₂ -CH ₂ -CH ₂ -CH ₂ -), 0.96(6H, t J 7.2 Hz, OCH ₂ -CH ₂ -CH ₂ -CH ₂ -CH ₃).
7b	-(CH ₂) ₆	7.936 (1H, s, CH=N), 7.755 (3H, d J 8.8 Hz, Ar-H), 6.999 (4H, d J 8.4 Hz, Ar-H), 4.014 (4H, m, -OCH ₂), 1.823 (4H, quint. J 6.4 Hz, -OCH ₂ -CH ₂ -CH ₂), 1.32 (10H, m, OCH ₂ -CH ₂ -CH ₂ -CH ₂ -), 0.96(6H, m, OCH ₂ -CH ₂ -CH ₂ -CH ₂ -CH ₃).
7c	-(CH ₂) ₇	7.790 (1H, s, CH=N), 7.443 (3H, br.s, Ar-H), 6.950 (4H, br.s, Ar-H), 4.029 (4H, br.s, -OCH ₂), 3.023(4H, br.s, -OCH ₂ -CH ₂), 1.823 (4H, br.s, -OCH ₂ -CH ₂ -CH ₂), 1.481-1.33 (8H, m, OCH ₂ -CH ₂ -CH ₂ -CH ₂ -), 0.923(6H, br.s, OCH ₂ -CH ₂ -CH ₂ -CH ₂ -CH ₃).
7d	-(CH ₂) ₈	7.782 (1H, s, CH=N), 7.473 (3H, d J 7.6 Hz, Ar-H), 6.953 (4H, d J 8.0 Hz, Ar-H), 4.029 (4H, t J 6.4 Hz, -OCH ₂), 2.946 (4H, br.s, -OCH ₂ -CH ₂), 1.800 (4H, br. quint. J 6.8 Hz, -OCH ₂ -CH ₂ -CH ₂), 1.46 (10H, m, OCH ₂ -CH ₂ -CH ₂ -CH ₂ -), 0.914(6H, t J 6.8 Hz, OCH ₂ -CH ₂ -CH ₂ -CH ₂ -CH ₃).
7e	-(CH ₂) ₉	7.786 (1H, s, CH=N), 7.467 (3H, br.s, Ar-H), 6.950 (4H, br.s, Ar-H), 4.016 (4H, br.s, -OCH ₂), 2.947(4H, br.s, -OCH ₂ -CH ₂), 1.825 (4H, br.s, -OCH ₂ -CH ₂ -CH ₂), 1.481-1.320 (12H, m, OCH ₂ -CH ₂ -CH ₂ -CH ₂ -), 0.916 (6H, br.s, OCH ₂ -CH ₂ -CH ₂ -CH ₂ -CH ₃).
7f	-(CH ₂) ₁₀	778 (1H, s, CH=N), 7.470 (3H, d J 8.4 Hz, Ar-H), 6.951 (4H, d J 8.4 Hz, Ar-H), 4.005 (4H, t J 6.8 Hz, -OCH ₂), 2.935 (4H, br. t J 5.6 Hz, -OCH ₂ -CH ₂), 1.816 (4H, quint. J 6.4 Hz, -OCH ₂ -CH ₂ -CH ₂), 1.49-1.30 (14H, m, OCH ₂ -CH ₂ -CH ₂ -CH ₂ -), 0.91(6H, t J 7.2 Hz, OCH ₂ -CH ₂ -CH ₂ -CH ₂ -CH ₃).

Table 2: FT-IR data in cm^{-1} of the compounds (7a-7f).

No.	Compound	C-H _{str.} Aliphatic ν/cm^{-1}	C=N _{str.} Imine ν/cm^{-1}	=C-H _{str.} Aromatic ν/cm^{-1}	C=C _{str.} Aromatic ν/cm^{-1}	C-O _{str.} , ν/cm^{-1}
1	7a	2958, 2839m	1620m	3055w	1606s	1250s

**Figure 4:** ^1H -NMR Spectra of compound 7d.**Figure 5:** FT-IR Spectra of compound 7a.

3.2 Liquid crystal study 7d

The temperature of phase transitions was recorded using a differential scanning calorimeter (DSC-60 Shimadzu) conducted at (Amman University- Jordan). The mesophase texture of the compounds was identified using POM (Leica DM750P),

connected to a Linkam Hotstage. The texture of the Schiff bases was monitored using polarised light in which the sample was sandwiched between two glass slides to form of a thin film. The transitions have been shown for all compounds that exhibited thermotropic properties which were carefully monitored by POM, and are summarized in Table 3. The phase transition

temperatures and the (DSC) analysis were conducted at the rate of (10 °C/min) with heating from room temperature (25 °C) to (250 °C) The phase transitions are characterized by the changes of enthalpy (ΔH) and entropy (ΔS). All of the synthesized

compounds showed expected liquid crystalline properties parallel with increasing the length of the alkoxy chain.

Table 3: Phase transition temperatures Changes/°C Recorded on Dsc & Pom.

Compound	Cr – Cr	Cr-S _m C		SmC-N		Mesophase-I	
	DSC	DSC	POM	DSC	POM	DSC	POM
7a	53.73	68.57	68.80	95.34	96.00	112.00	115.00
7b	86.47	99.33	100.10	117.22	117.80	131.67	132.00
7c	-----	87.32	88.00	115.19	116.00	133.90	134.00
7d	-----	97.21	98.30	116.33	118.30	131.43	133.00
7e	-----	102.60	102.70	114.00	114.00	133.80	133.00
7f	91.00	118.06	121.00	-----	-----	137.50	138.00

3.3 Mesophase Thermal Stability Ranges (ΔT_m):

As shown in Table 3, an interesting wide range of nematic thermal stability range ($\Delta T_N=19$ °C) was exhibited by the first number (n=1) in the series. Another widest smectic thermal stability range ($\Delta T_{Sm}=28$ °C) is also observed in the third member (n=3).

3.4 Differential scanning calorimetry (DSC) measurements:

Enthalpy changes (ΔH) of transitions are measured and recorded by (DSC) instrument as in all DSC curves see Figure 6.

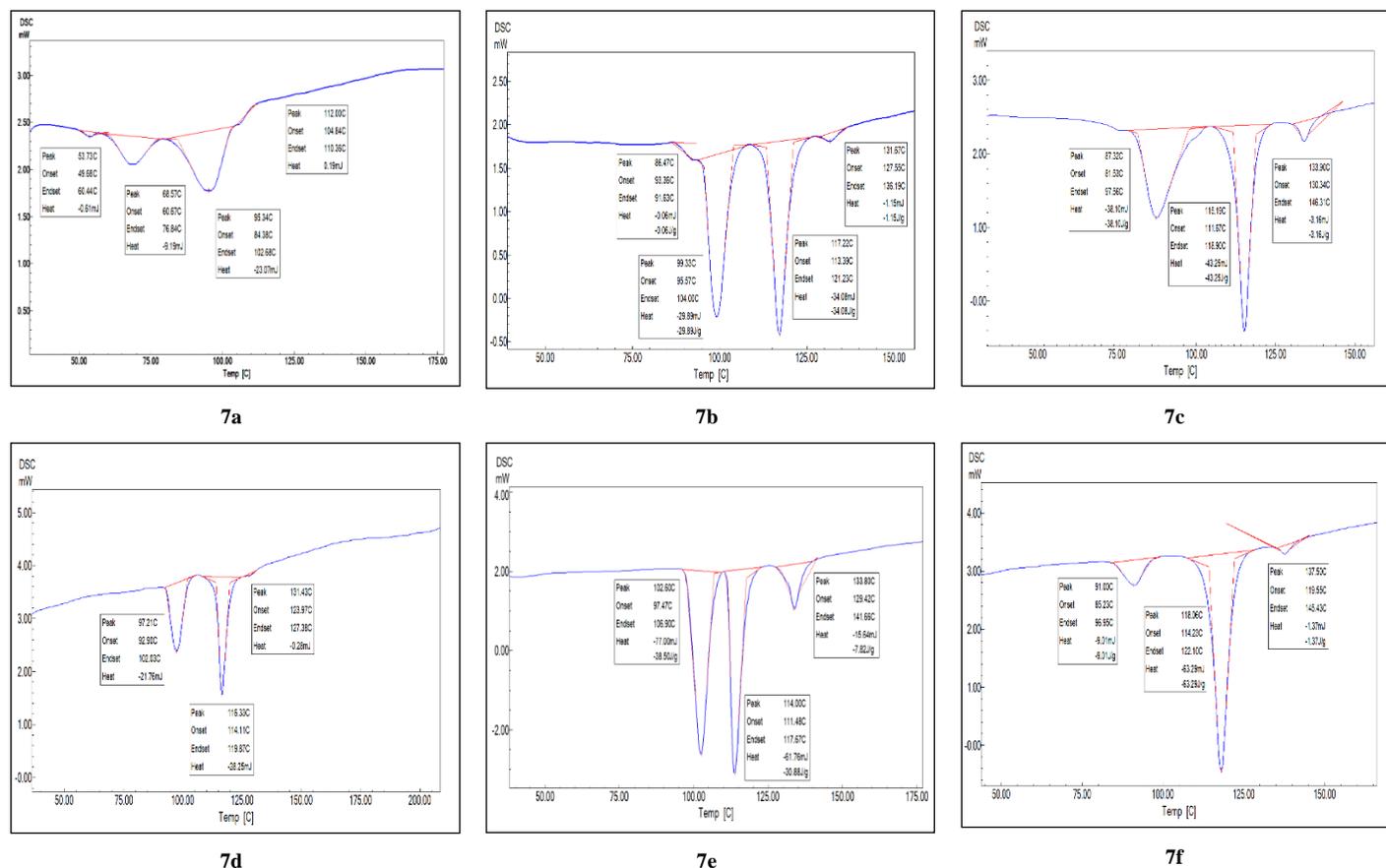


Figure 6: DSC for compound 7a-7f, show the ΔH change.

3.5 Liquid Crystal Characterization of Textures

3.5.1 Nematic Phase Textures

The compounds (7a-e, n=5 to 9) revealed a clear nematic (N) phase with a thread-like texture which is the schlieren texture of the nematic liquid crystals (NLC) and the droplet texture. The schlieren texture showed dark brushes, which belong to the extinction orientation of the (NLC)^[19]. Generally, in this study, the schlieren texture shows two brushes meet instead of four brushes (Figure 7) and there are droplet textures (Figure 8). The only compound which has no nematic phase is the last one (7f), and this is attributed to the terminal alkyl chain with six carbons, going toward the favoring smectic (S_m) phases^[20].

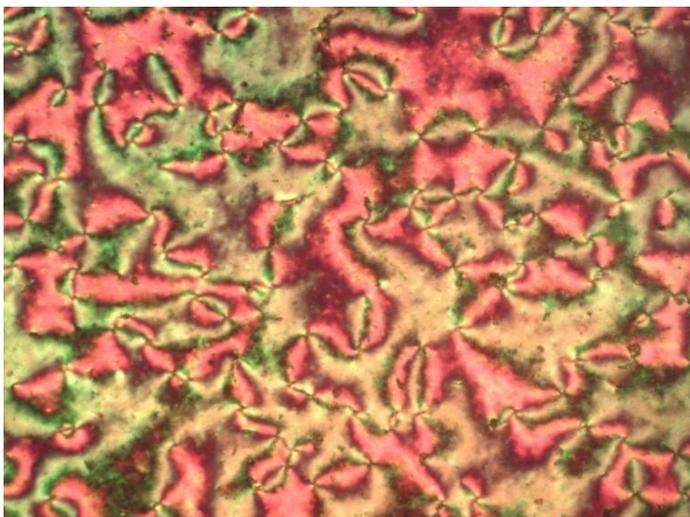


Figure 7: Nematic mesophase with thread-like (Schlieren) textures of (7a) at 99.2 °C.

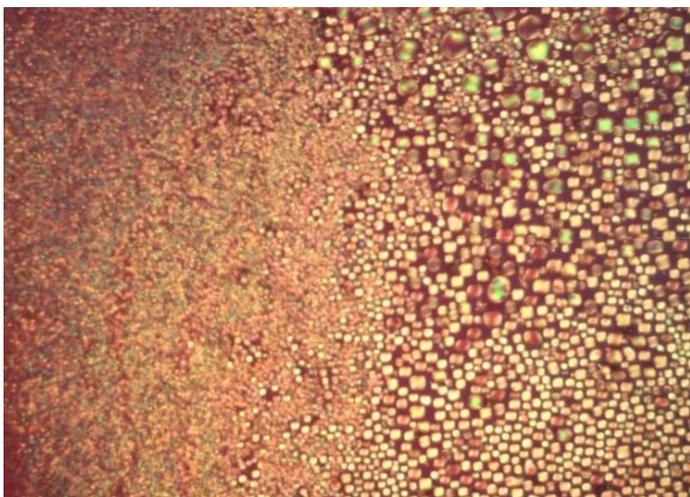


Figure 8: Nematic mesophase with droplet textures of (7e) at 129.5 °C.

3.5.2 Smectic (S_m) Phase Textures

All Schiff base compounds have the Smectic C (S_mC) mesophase which exhibits the schlieren textures^[21] (Figure 9 and 10) are the S_mC mesophases of both A_2 and A_6 compounds respectively.

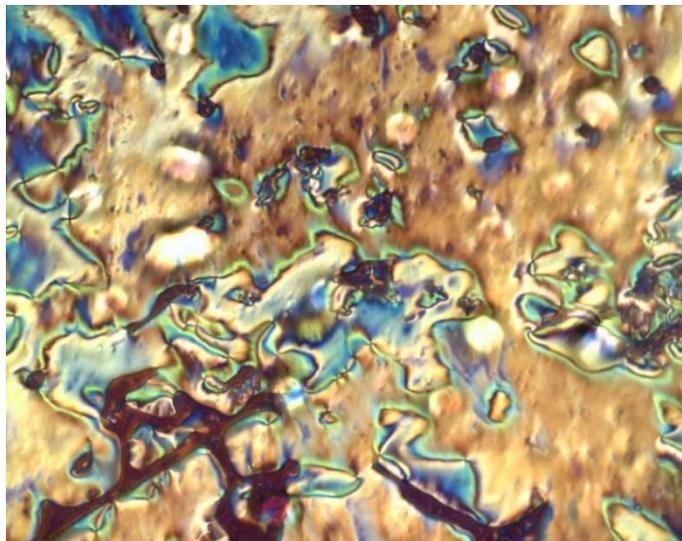


Figure 9: Smectic C mesophase with Schlieren texture of (7b) at 110.0 °C.

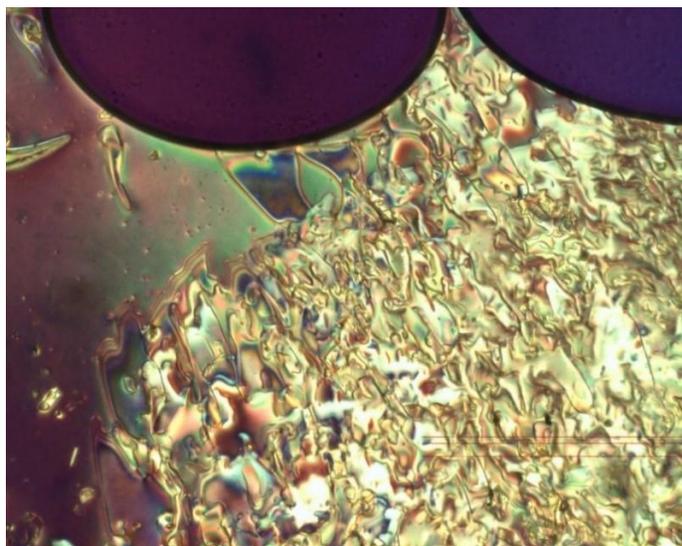


Figure 10: The transition from Schlieren texture of (S_mC) to the isotropic state of (7f) at 135.5 °C.

Conclusion

A new series of benzothiazole Schiff bases with interesting mesophase properties were synthesized and characterized based on FT-IR, and ¹H-NMR spectroscopies. Differential scanning calorimetry (DSC) showed the phase transitions of every single compound with clearly the highest purity, the mesophase textures have been observed using polarized optical microscopy (POM). All Schiff base compounds have the Smectic C mesophase which exhibits the schlieren textures. Particularly the compounds (7a-e, n=5 to 9) exhibited a clear nematic (N) phase with thread-like texture; which are the schlieren texture and the droplet texture. The highest thermal stability range trend for nematic mesophase ($\Delta T_N=19$ °C) for the first member (n=1) and ($\Delta T_{S_m}=28$ °C) in the third member (n=3) in the series have been noticed.

Authors contribution

All authors contributed to Experimental working, writing, and controlling the manuscript.

Conflict of interests

The authors of this manuscript have neither financial nor personal relations with individuals or organizations which could inappropriately influence or bias the content of the manuscript.

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