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Research Article

New Homologues Series of Heterocyclic Schiff Base Ester: Synthesis and Characterization

Yee-Ting Chong,[1] Norazilawati Muhamad Sarih,[1] Sie-Tiong Ha,[2] and Md. Rezaul Karim Sheikh[3]

[1] *Department of Chemistry, Faculty of Science, University of Malaya, 50603 Kuala Lumpur, Malaysia*
[2] *Department of Chemical Science, Universiti Tunku Abdul Rahman, Jalan Universiti, Bandar Barat, 31900 Kampar, Perak, Malaysia*
[3] *Department of Applied Chemistry & Chemical Engineering, University of Rajshahi, Rajshahi 6205, Bangladesh*

Correspondence should be addressed to Yee-Ting Chong; chongyt@siswa.um.edu.my and Norazilawati Muhamad Sarih; nmsarih@um.edu.my

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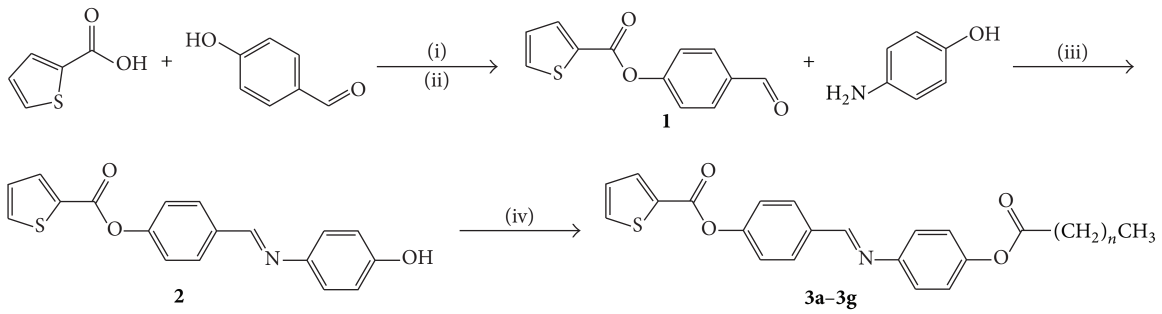
A homologous series of liquid crystal bearing with heterocyclic thiophene Schiff base ester with alkanoyloxy chain $(CH_3(CH_2)_nCOO–$, where $n = 4, 6, 8, 10, 12, 14, 16)$ was successfully synthesized through the modification of some reported methods. The structural information of these compounds was isolated and characterized through some spectroscopic techniques, such as FTIR, ^{1}H, and ^{13}C NMR and elemental analysis. Textural observation was carried out using a polarizing optical microscope (POM) over heating and cooling cycles. It was found that all synthesized compounds (**3a–g**) exhibited an enantiotropic nematic phase upon the heating and cooling cycle with high thermal stability. Moreover, a characteristic bar transition texture was observed for compounds **3f** and **3g** which have shown transition of nematic-to-smectic C phase. This has been further confirmed by obtaining relative phase transition temperature using the differential scanning calorimetry (DSC).

1. Introduction

The design and investigation of new mesogens are fast garnering interest from liquid crystal researchers [1]. Among the synthesized compounds, thermotropic liquid crystal has brought some interests to the researcher. Thermotropic liquid crystalline compounds possess a number of unique properties that are of immense value. Tremendous efforts were made on designing of new compounds. It was found that the molecules with an extended rod-like shape often greatly exhibit thermotropic liquid crystalline phase [2, 3]. A significant amount of work [4–7] has been carried out on rod-shaped liquid crystals, which ultimately led to the application of LC displays. Its molecules could be arranged in smectic layers, making them a viable possibility for technological products [4]. Rod-like liquid crystals are generally categorized into two categories, which are liquid-like smectics (SmA and SmC) and crystal-like smectics (SmB, SmE, SmK, or SmG), where both are based on different distributions on its molecular center of mass [5].

New mesogens with heterocyclic compounds such as benzothiazole [6, 8, 9], benzoxazole [10], 1,2,4-oxadiazole [11], pyrimidine [12], and pyrazole [13] had attracted the interest of scientists, as the insertion of heteroatom will strongly influence the formation of mesomorphic phases. These heterocyclic structures generally incorporate unsaturated atoms, such as O, N, or S, and due to the high electronegativity of these atoms, it often results in reduced symmetry of the molecules and creates a stronger polar induction. The use of unique heterocyclic moieties to produce materials of low symmetry or/and nonplanar structures can be technologically important for liquid crystal applications [14].

There are lots of the compound previously published being synthesized with the insertion of the above heterocyclic ring. However, the reported literatures on the thiophene



Scheme 1: Synthetic route for the target compounds: (i) $SOCl_2$; (ii) benzene, pyridine; (iii) ethanol, acetic acid (cat.); (iv) DCC, DMAP, dichloromethane, and dimethylformamide. Yield of **3a** (19%), **3b** (23%), **3c** (29%), **3d** (35%), **3e** (42%), **3f** (39%), and **3g** (40%), where $n = 4$ (**3a**), 6 (**3b**), 8 (**3c**), 10 (**3d**), 12 (**3e**), 14 (**3f**) and 16 (**3g**).

based liquid crystal are still rare and there are great potential for further investigation of this kind. Despite the presence of heteroatom, S in the thiophene ring, it was also a five-membered heterocyclic compound. The previous works have shown that the five-membered heterocyclic compounds were able to show mesomorphic properties. The presence of a heteroatom can cause profound changes in mesomorphic behavior, due to the differences in their electronegativity in relation to carbon or through changes in molecular geometry [15, 16].

In this paper, we will report a new homologous series of thiophene-2-carboxylate derivative liquid crystals (Scheme 1). The derivatives possess three core units (one thiophene and two phenyl rings) and are connected via a Schiff base and ester linkers, which increases both the molecular broadness and anisotropy.

2. Experimental

2.1. Materials. Decanoic acid, dodecanoic acid, tetradecanoic acid, hexadecanoic acid, octadecanoic acid, 4-aminophenol, and thionyl chloride ($SOCl_2$) were purchased from Merck, while thiophene-2-carboxylic acid, octanoic acid, 4-hydroxybenzaldehyde, and 4-dimethylaminopyridine (DMAP) were procured from Sigma Aldrich. *N,N′*-Dicyclohexylcarbodiimide (DCC) was purchased from Acros Organics (USA). All reagents and solvents were used as received without further purification.

2.2. Synthesis. The synthetic protocol towards the title compounds, 4-(((4-n-alkanoyloxyphenyl)imino)methyl)phenyl thiophene-2-carboxylate (where n = 6, 8, 10, 12, 14, 16, 18), is illustrated in Scheme 1.

*Synthesis of Compounds **1**.* 2-Thiophenecarboxylic (40 mmol) acid was dissolved in 20 mL of thionyl chloride in a round bottom flask. The reaction mixture was allowed to reflux for 3 hours. Upon completion of the reaction, the excess of thionyl chloride was allowed to evaporate. A mixture of 4-hydroxybenzaldehyde (40 mmol) dissolved in 10 mL of pyridine and 40 mL of benzene was then added in slowly. The mixture was refluxed for 2 hours. The precipitate formed was then immediately filtered off and the resulting solution was left for evaporation. The crude precipitate was recrystallized from the mixture of chloroform and hexane in the ratio of 3 : 1.

*Synthesis of Compound **2** [17].* Compound **1** (20 mmol) was dissolved in 40 mL absolute ethanol in a round-bottom flask and a mixture of ethanol and 4-aminophenol (20 mmol) was added dropwise. The mixture was refluxed for 3 hours. The resulting hot solution was then cooled to room temperature and subsequently cooled to below 5°C for 1 hour to allow the solid precipitate out. The crude products were then recrystallized first with a mixture of chloroform and hexane in the ratio of 3 : 1 and further on with ethanol.

*Synthesis of Compound **3** [17].* Compound **2** (2 mmol) was initially dissolved in a minimum amount of dimethylformamide (**DMF**) and added into a mixture of corresponding fatty acid (2 mmol) and dimethylaminopyridine (**DMAP**) (0.2 mmol), which has been already dissolved in dichloromethane (20 mL). *N,N′*-Dicyclohexylcarbodiimide (**DCC**) (2 mmol) in dichloromethane (5 mL) was added dropwise into the mixture upon stirring at 0°C for an hour and was then stirred at room temperature for another three hours. Finally, the mixture was filtered, and excess solvent was removed from the filtrate via evaporation. The white product was recrystallized first with a mixture of chloroform and hexane in the ratio of 3 : 1 and further with ethanol until its transition temperature remained constant. The purity was detected by the use of TLC with the chloroform as solvent.

2.3. Characterization. The percentage yield and analytical data of the representative compound **3c** are as follows.

Yield: 29%, IR (KBr, cm^{-1}): 3077 (aromatic C–H stretch), 2957, 2922, 2873, 2849 (aliphatic C–H stretch), 1728 (ester C=O stretch), 1626 (imine C=N stretch), 1602, 1468 (C=C aromatic ring stretch), 1205, 1142 (ester C–O stretch), 864 (C–S–C stretch) ^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 0.87–0.91 (t, 3H, J = 6.7 Hz, $-CH_2C\underline{H}_3$), 1.29–1.43 (m, 12H, $-CH_2(C\underline{H}_2)_6CH_3$), 1.73–1.80 (qt, 2H, J = 7.5 Hz, $-C\underline{H}_2CH_2COO-$), 2.55–2.58 (t, 2H, J = 7.5 Hz, $-C\underline{H}_2COO-$), 7.10–7.12 (d, 2H, J = 8.8 Hz, Ar–H), 7.19–7.21 (t, 1H, J = 4.3 Hz, Th), 7.22–7.24 (d, 2H, J = 8.7 Hz, Ar–H), 7.34–7.36 (d, 2H, J = 8.6 Hz, Ar–H), 7.69–7.70 (d, 1H, J = 4.9 Hz, Th), 7.95–7.98

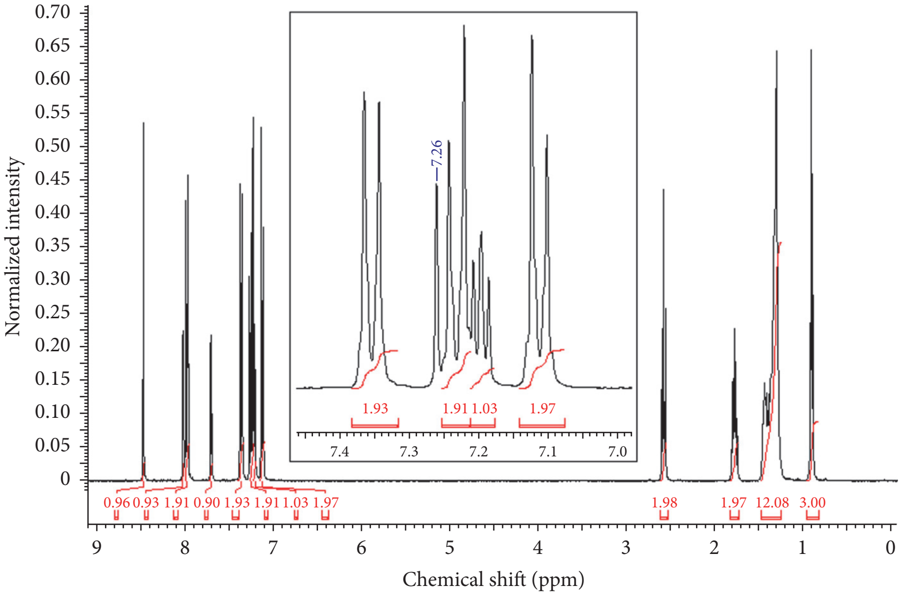


FIGURE 1: ^{1}H NMR spectra of **3c**.

(d, 2H, J = 8.6 Hz, Ar–H), 8.00–8.01 (d, 1H, J = 3.7 Hz, Th), 8.46 (s, 1H, $C\underline{H}=N$), ^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 14.12 ($-CH_3$), 22.68 ($-CH_2CH_3$), 24.97 ($-\underline{C}H_2CH_2COO-$), 29.13, 29.27, 29.43, 31.87 for methylene carbon ($-(\underline{C}H_2)-$), 34.43 ($-\underline{C}H_2COO-$), 121.77. 122.14, 122.25, 128.14, 130.06, 132.54, 133.85, 133.95, 134.98, 148.98, 149.38, 152.97, for aromatic carbon, 159.16 ($-\underline{C}H=N$), 160.21, 172.45 ($-\underline{C}OO$), Anal. calcd. For $C_{28}H_{31}NO_4S$: C, 70.41%, H, 6.54%, N, 2.93%: found: C, 70.42%, H, 6.77%, N, 2.98%.

3. Results and Discussion

All of the synthesized compounds showed the presence of a single spot in thin layer chromatography (TLC) plates. Structural elucidation of **3c** is discussed as a representative compound from the homologous series, supported by the results of spectroscopic analysis (IR, ^{1}H and ^{13}C NMR).

Based on the FT-IR spectrum of **3c**, absorption peaks at 2957, 2922, 2873, and 2849 cm^{-1} can be assigned to aliphatic groups, such as methyl (CH_3) and methylene (CH_2) groups in the long alkyl chain of the molecule. The presence of peaks at 1728, 1205, and 1142 cm^{-1} indicates the existence of the ester group (C=O). The absorption of the Schiff base group (C=N) was found at 1626 cm^{-1}.

In the ^{1}H NMR spectrum of **3c** (Figure 1), the triplet at δ = 0.87–0.91 ppm and multiplet at δ = 1.29–1.43 ppm were, respectively, ascribed to the methyl and methylene group ($-CH_2CH_2\underline{CH_3}$) of longer alkyl chain. The triple at δ = 2.55–2.58 ppm was assigned to the methylene ($-\underline{CH_2}COO-$) next to the ester group, while β-proton from the ester group ($-\underline{CH_2}CH_2COO-$) was found at δ = 1.73–1.80 ppm in the form of a quintet. The four doublets observed at δ = 6.5–8.0 ppm were attributed to aromatic protons of the two phenyl rings. Two doublets and one triplet observed at δ = 7.69–8.01 ppm were assigned to the 3 protons in the thiophene ring. The singlet at δ = 8.46 ppm supported the presence of imine linking group in the structure of the compound **3c** [9].

By referring to the ^{13}C spectrum of **3c** (Figure 2), the peak at range δ = 22.68–34.43 ppm was assigned to the methylene carbon of the longer alkyl chain, whereas the peak at δ = 14.12 ppm represents the methyl carbon of the longer alkyl chain. Sixteen aromatic carbons were found that resonated within δ = 121.77–152.97 ppm and four of the peaks were more intense compared to the other four peaks. This suggested that the high intensity peaks can be assigned to two equivalent aromatic carbons of the phenyl ring which had a 1,4-disubstitute structure. Peaks at δ = 159.16 ppm further confirmed the presence of azomethine carbon and it is compatible with the value as stated in the literature [17]. Meanwhile, peaks at δ = 160.21 and 172.45 ppm indicated the presence of two carbonyl ester groups in the molecule [10, 17].

Mesomorphic properties of the title compounds were observed through an optical polarizing microscope attached to a video camera. The optical micrograph of the mesophases of **3b** and **3g** was given in Figure 4. The identification for the observed textures was then compared to previously reported samples [18, 19].

All the synthesized compounds exhibited enantiotropic nematic phase properties, with a higher-ordered monotropic SmC phase emerging from hexadecanoyloxy derivatives, while the enantiotropic SmC phase was observed in the octadecanoyloxy derivative. Representative optical photomicrographs of compounds **3b** and **3g** are shown in Figure 3. The presence of nematic in **3b** (Figure 3(a)) was evidenced by a typical thread-like texture upon cooling of the compounds from the isotropic liquid phase. Upon further cooling of the homeotropic nematic phase (Figure 3(b)) of **3g** to a lower temperature, a higher-order SmC was formed.

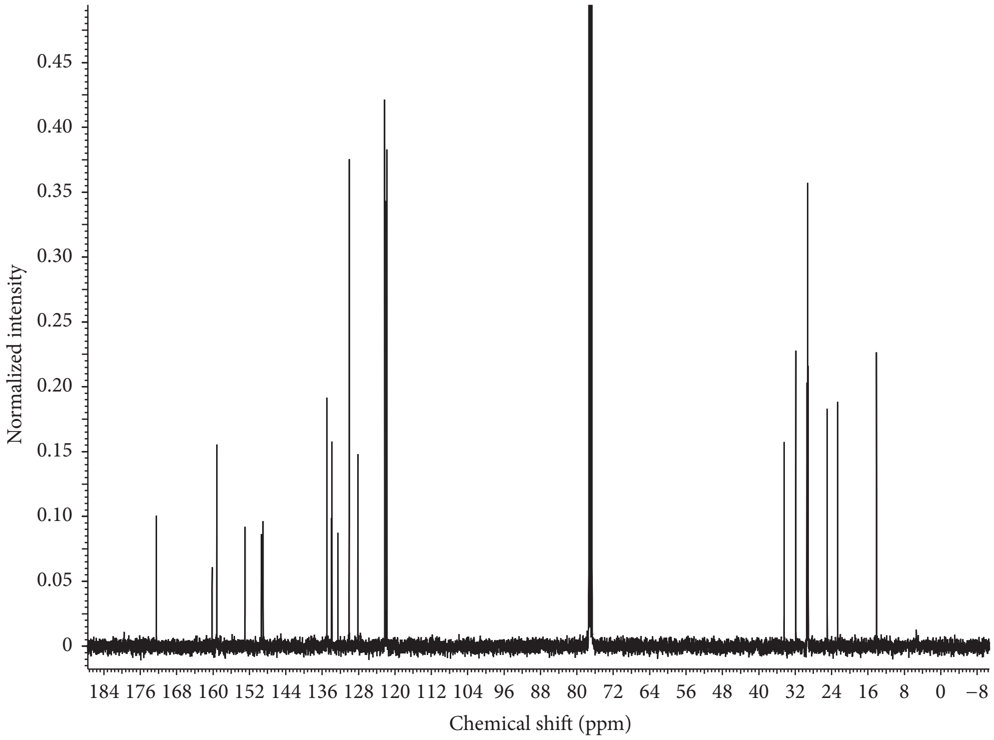
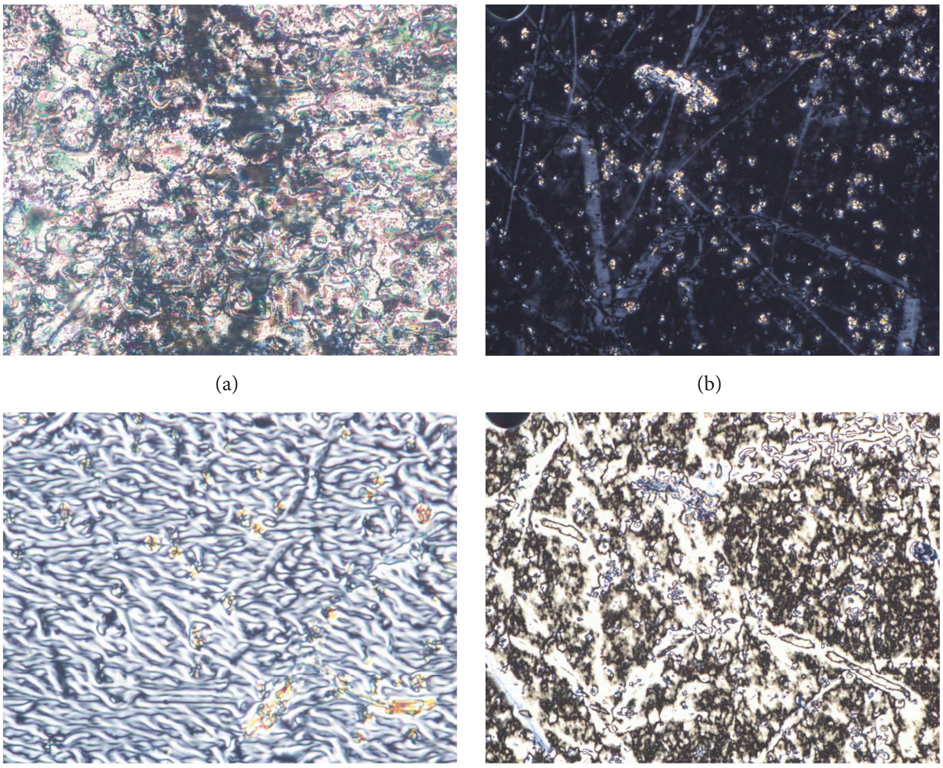


Figure 2: ^{13}C NMR spectra of **3c**.

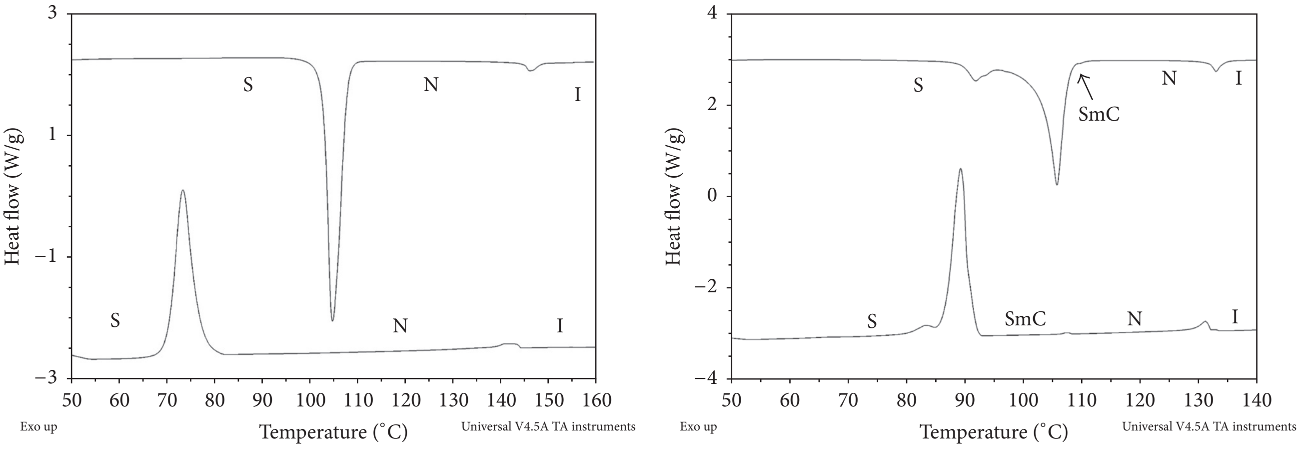

(a)

(b)

(c)

(d)

Figure 3: (a) Thread-like nematic at 103°C of **3b** (20x magnificent). (b) Homeotropic nematic at 110°C of **3g** (20x magnificent). (c) N-SmC transition bar at 105°C of **3g** (20x magnificent). (d) Schlieren smectic C of at 98°C of **3g** (20x magnificent).



(a)

(b)

Figure 4: (a) DSC thermogram of **3d**. (b) DSC thermogram of **3g**.

Table 1: Phase transition temperature and transition enthalpy changes for compounds **3a–g**.

Compound	Phase transition, °C (corresponding enthalpy changes, kJmol^{-1})
3a	Cr$_1$ 85.08 (5.88) Cr$_2$ 95.43 (24.65) N 156.81 (0.55) I
	I 156.69 (0.47) N 75.86 (20.72) Cr
3b	Cr 104.75 (38.67) N 146.10 (1.06) I
	I 143.30 (1.02) N 73.49 (33.61) Cr
3c	Cr$_1$ 84.45 (0.95) Cr$_2$ 105.51 (28.03) N 141.04 (1.09) I
	I 136.30 (0.71) N 81.14 (29.07) Cr
3d	Cr$_1$ 89.51 (0.81) Cr$_2$ 108.08 (31.92) N 142.25 (0.91) I
	I 141.15 (1.01) N 84.53 (34.72) Cr
3e	Cr$_1$ 89.53 (0.66) Cr$_2$ 108.96 (38.21) N 141.93 (1.27) I
	I 139.27 (1.37) N 82.90 (40.61) Cr
3f	Cr$_1$ 100.34 (3.88) Cr$_2$ 111.01 (33.26) N 135.67 (2.10) I
	I 134.15 (2.37) N 105.48 (0.32) SmC 89.75 (47.45) Cr
3g	Cr$_1$ 91.78 (2.67) Cr$_2$ 105.76 (32.30) SmC 109.76 (0.02) N 132.92 (2.01) I
	I 131.05 (1.60) N 107.43 (0.14) SmC 90.82 (42.39) Cr

The characteristic transition bar texture (Figure 3(c)) [20], which usually appeared in the transition of a nematic phase into SmC phase, was observed there. This further confirmed the presence of the Schlieren smectic C-phase (Figure 3(d)).

The DSC results of compounds **3a–3g** are consistent with the results obtained via polarizing microscopy in terms of transition temperatures for all heating and cooling cycles. The DSC traces of representative compounds **3d** and **3g** are given in Figures 4(a) and 4(b), respectively. The transition temperatures of the mesomorphic change data were tabulated in Table 1.

A plot of transition temperatures against the number of carbons in the alkanoyloxy chain during the heating cycle is shown in Figure 5. Firstly, it is evident that both melting (Cr-SmC/N) and clearing (N-I) points were greatly influenced by the length of alkanoyloxy chain in the compounds. The melting point of the title compounds showed an ascending trend as a function of the number of carbon atoms in the alkanoyloxy chain. This may be due to the increase in the van der Waals force as the size of the molecules is elongated. However, the clearing temperature-isotropic temperature is inversely proportional to the number of carbons. The additional number of carbon atoms in the terminal chain provides extra flexibility to the molecular structure. Secondly, a higher-ordered SmC phase was observed only in the homologous with relatively longer chain members, C18 in this case [21]. It can be understood that a longer terminal alkanoyloxy chain may favor the lamellar arrangement in the smectic layer structure due to the increased of van der Waals interactions and the possibilities of intertwining between alkanoyloxy chains [22]. Finally, the nematic phase range was reduced while the SmC phase range increased as the chain length increased. The increasing van der Waals attractive forces provide stronger lateral attractive interactions, which in turn resulted in the formation of layer packing.

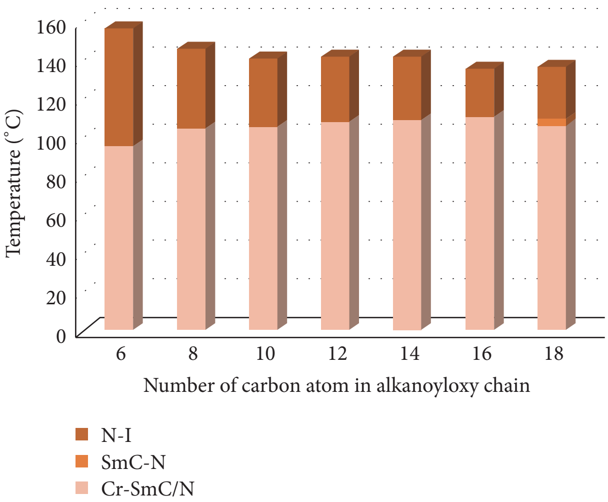


Figure 5: Transition temperatures against the number of carbons in the alkanoyloxy chain of the title compounds during the heating cycle.

4. Conclusion

All the synthesized compounds exhibited liquid crystal behavior, where nematic and SmC phases were observed. The length of the terminal alkanoyloxy chain has greatly influenced the properties of the liquid crystal, such as melting and clearing isotropic temperatures and liquid crystal phase ranges. This can be shown where the title compounds exhibit enantiotropic N phase with relatively high thermal stability (>30°C) and the existence of smectic C for the longer carbon chain compounds ($n > 14$). Even numbers of flexible chain were chosen as the intermolecular attractive forces of the even number compounds are stronger as compared to the odd numbers compounds due to its molecular structure conformation which are more linear. Heterocyclic such as thiophene is an essential core unit as it will enhance the mesomorphic properties and greater anisotropy properties of the liquid crystal materials. For future work, we will rearrange and modify the molecular structure of the homologous series for higher thermal stable liquid crystal compounds by introducing the thiophene rings in the middle of core unit which is expected to provide a bent shape of core unit.

Competing Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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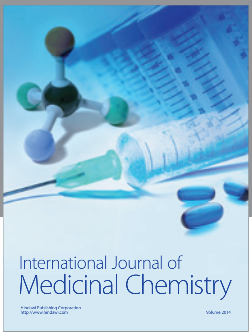
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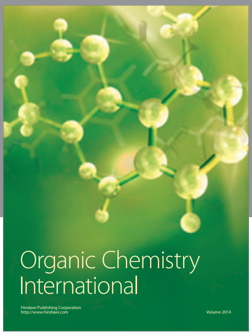
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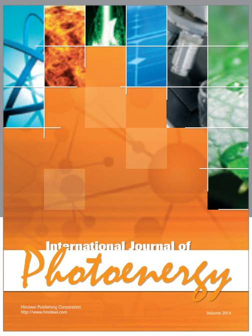
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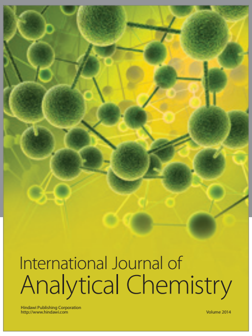
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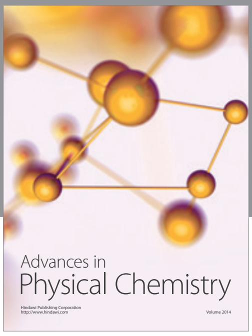
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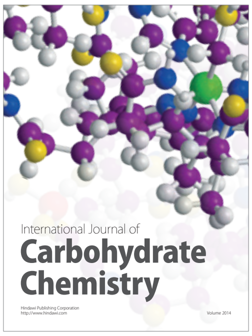
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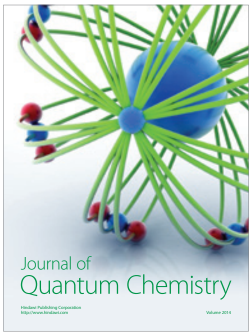
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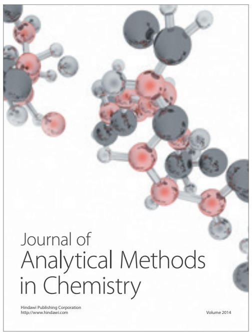
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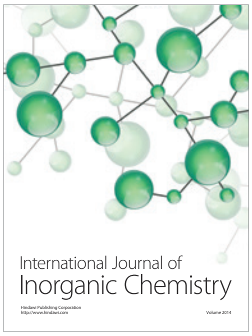
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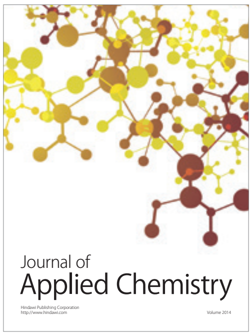
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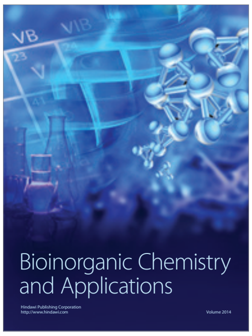
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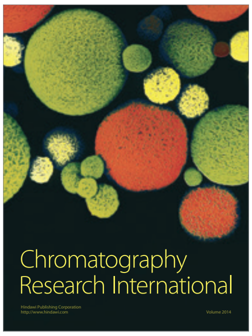
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