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Mesogenic Schiff base esters with benzothiazole core: synthesis and phase transition studies

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A series of new rod-shaped mesomorphic compounds, 6-ethoxy-2-(4-alkanoyloxybenzylidenamino)benzothiazoles, consisting of a 2,6-disubstituted benzothiazole core and a Schiff base central linkage, were synthesized and their structures were ascertained via elemental analysis and spectroscopic techniques. Their mesomorphic properties were studied using differential scanning calorimetry (DSC), polarized optical microscopy (POM) and X-ray diffraction (XRD) analysis. All compounds showed enantiotropic mesomorphism. Whilst the lower members of the series, hexanoyloxy and octanoyloxy derivatives exhibited nematic phase, the higher members (decanoyloxy, dodecanoyloxy, tetradecanoyloxy, hexadecanoyloxy and octadecanoyloxy derivatives) exhibited nematic and smectic C phases.

Keywords: Schiff bases; 6-ethoxy-2-(4-alkanoyloxybenzylidenamino)benzothiazoles; enantiotropic; smectic C; nematic

1. Introduction

Over the past few decades, liquid crystals comprising a Schiff base (or azomethine) linkage have received overwhelming attention since the discovery of the room temperature-liquid crystal, 4-methoxybenzylidene-4'-butylaniline (MBBA) [1]. Selection of a mesogenic core, terminal groups and a suitable length of the flexible chain are among the essential criteria in designing new thermotropic liquid crystals [2]. Among the reported rod-like mesogens, a *para*-substituted phenyl ring often serves as an important core unit, which ensures that the molecules have structural linearity and large molecular polarizability, which consequently enables them to exhibit liquid crystalline behavior [3].

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Recently, there has been a continuing interest in the study of heterocyclic-based liquid crystal compounds due to their unique properties [4–6]. An earlier report has shown that the introduction of heterocycles as the core has greatly influenced the mesomorphic properties of the calamatic molecules owing to their unsaturation and/or their more polarizable nature [7]. It has also been revealed that the inclusion of the heteroatom considerably changes the polarity, polarizability and to a certain extent the geometry of the molecule, thereby influencing the type of mesophase, the phase transition temperatures, dielectric constants and other properties of the mesogens [8,9].

In order to further explore the mesomorphic properties of heterocyclic-based liquid crystals comprising a 2,6-disubstituted benzothiazole unit, we report in this article the synthesis and mesomorphic properties of a new homologous series of 6-ethoxy-2-(4-alkanoyloxybenzylidenamino)benzothiazoles with a Schiff base linkage and a terminal ethoxyl group at the sixth position of the benzothiazole core.

2. Experimental

4-Dimethylaminopyridine (DMAP) was obtained from Merck (Germany). 2-Amino-6-ethoxybenzothiazole, 2,4-dihydroxybenzaldehyde, fatty acids ($C_{n-1}H_{2n-1}COOH$ where $n = 6, 8, 10, 12, 14, 16, 18$) and N,N' -dicyclohexylcarbodiimide (DCC) were purchased from Acros Organics (USA). All solvents and reagents were purchased commercially and used without any further purification.

Infrared (IR) spectra were recorded using a Perkin-Elmer System 2000 FT-IR Spectrometer via KBr disc procedure. 1H -NMR (400 MHz) and ^{13}C -NMR (100 MHz) spectra were recorded in $CDCl_3$ using a JEOL LA-400 MHz-NMR spectrometer in the Chemistry Department, Universiti Malaya. EI-MS (70 eV) were measured with a Mass Spectrometer Finnigan MAT95XL-T at a source temperature of 200°C. The sample was introduced using direct inlet probe. Microanalyses were carried out on Perkin-Elmer 2400 LS Series CHNS/O analyzer.

Phase-transition temperatures and enthalpy changes were measured using a Differential Scanning Calorimeter Mettler Toledo DSC823^e at a scanning rate of 10°C min⁻¹. A polarizing optical microscope (Carl Zeiss) equipped with a Linkam heating stage was used for temperature dependent studies of the liquid crystal textures. Phase identification was made by comparing the observed textures with those reported in the literature [10,11].

Synchrotron powder X-ray diffraction (XRD) measurements were performed at beamline BL17A where the X-ray wavelength used was 1.32633 Å. XRD data were collected using imaging plates (IP, of an area = 20 × 40 cm² and a pixel resolution of 100) curved with a radius equivalent to the sample-to-image plate distance of 280 mm, and the diffraction signals were accumulated for 3 min. The powder samples were packed into a capillary tube and heated by a heat gun, where the temperature controller was programmed by a PC with a PID feedback system. The scattering angle theta was calibrated by a mixture of silver behenate and silicon.

2.1. Synthesis of Schiff base liquid crystals

The title compounds were synthesized according to a previously reported method [12–15]. The synthetic route is depicted in Figure 1. A solution of 2-amino-6-ethoxybenzothiazole (0.97 g, 5 mmol) and 4-hydroxybenzaldehyde (0.61 g, 5 mmol) in absolute ethanol (40 mL)

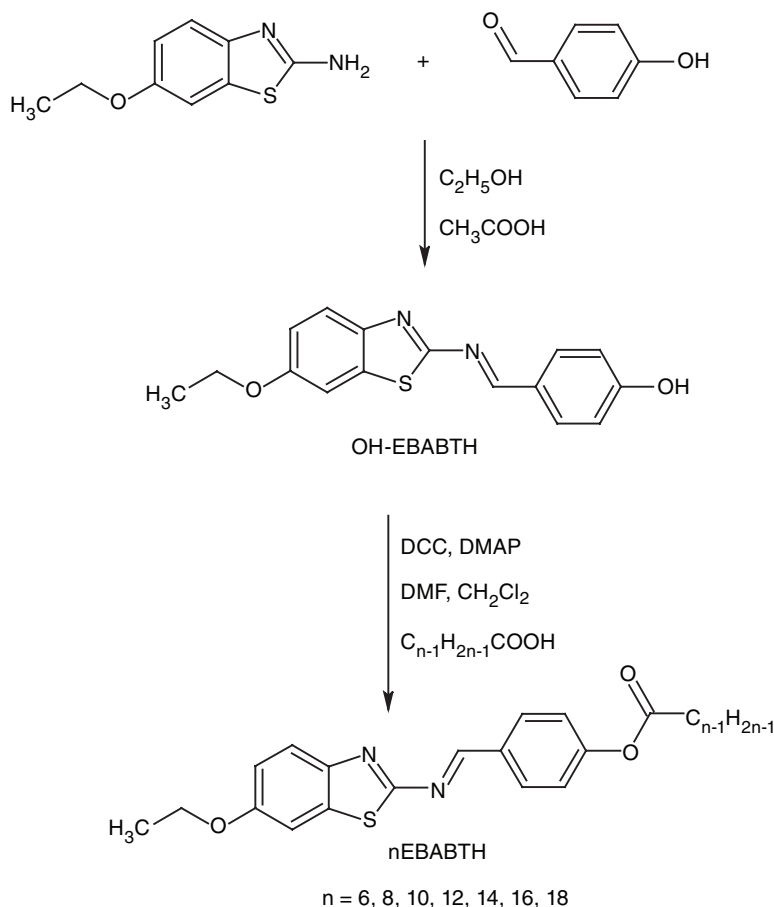


Figure 1. Synthetic scheme of 6-ethoxy-2-(4-*l*-kanoyloxybenzylidenamino)benzothiazoles, nEBABTH.

was heated under reflux for 3 h in the presence of acetic acid (two drops). The Schiff base formed, **OH-EBABTH**, was recrystallized from absolute ethanol.

Schiff base **OH-EBABTH** (0.90 g, 3 mmol), appropriate fatty acid (3 mmol) and DMAP (0.18 g, 1.5 mmol) were dissolved in a mixture of dichloromethane and DMF (40 mL) with the ratio of 7:1 and stirred in an ice bath. To this solution, DCC (0.62 g, 3 mmol) dissolved in dichloromethane (5 mL) was added drop wise upon stirring in the ice bath for an hour. The resulting mixture was stirred at room temperature for another 3 h. The reaction mixture was then filtered and the excess solvent was removed from the filtrate by evaporation. The yellow solid obtained was recrystallized several times using ethanol. The percentage yields and analytical data of the title compounds are tabulated in Table 1. The IR, NMR (^1H and ^{13}C) and mass spectral data for the representative compound, 16EBABTH, are given below.

6-Ethoxy-2-(4-hexadecanoyloxybenzylidenamino)benzothiazole (16EBABTH). Yield 60%. IR (KBr) ν_{max} cm^{-1} 3047 (C–H aromatic), 2922, 2849 (C–H aliphatic), 1755 (C=O ester), 1597 (C=N thiazole), 1450 (C=C aromatic). $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ/ppm 0.9 (t, $J=6.6$ Hz, 3H, CH_3 -), 1.2–1.4 (m, 24H, $\text{CH}_3-(\text{CH}_2)_{12}-\text{CH}_2-\text{CH}_2-\text{COO}$ -),

Table 1. Percentage yield and physical data of *n*EBABTH.

Compound	Yield (%)	Formula	% Found (% Required)		
			C	H	N
6EBABTH	34	C ₂₂ H ₂₄ N ₂ O ₃ S	66.53 (66.64)	6.17 (6.10)	7.00 (7.07)
8EBABTH	43	C ₂₄ H ₂₈ N ₂ O ₃ S	67.79 (67.90)	6.74 (6.65)	6.53 (6.60)
10EBABTH	41	C ₂₆ H ₃₂ N ₂ O ₃ S	69.09 (69.00)	7.15 (7.13)	6.11 (6.19)
12EBABTH	56	C ₂₈ H ₃₆ N ₂ O ₃ S	70.08 (69.97)	7.47 (7.55)	5.77 (5.83)
14EBABTH	55	C ₃₀ H ₄₀ N ₂ O ₃ S	70.75 (70.83)	7.90 (7.93)	5.45 (5.51)
16EBABTH	63	C ₃₂ H ₄₄ N ₂ O ₃ S	71.69 (71.60)	8.19 (8.26)	5.19 (5.22)
18EBABTH	68	C ₃₄ H ₄₈ N ₂ O ₃ S	72.19 (72.30)	8.68 (8.57)	5.04 (4.96)

1.5 (t, $J=6.9$ Hz, 3H, CH₃-CH₂-O-), 1.8 (quint, $J=7.2$ Hz, 2H, -CH₂-CH₂-COO-), 2.6 (t, $J=7.7$ Hz, 2H, -CH₂-COO-), 4.1 (q, $J=7.2$ Hz, 2H, CH₃-CH₂-O-), 7.0 (d, $J=8.8$ Hz, 1H, Ar-H), 7.2 (d, $J=8.5$ Hz, 2H, Ar-H), 7.3 (s, 1H, Ar-H), 7.8 (d, $J=8.8$ Hz, 1H, Ar-H), 8.0 (d, $J=8.5$ Hz, 2H, Ar-H), 9.0 (s, 1H, -N=CH-). ¹³C-NMR (100 MHz, CDCl₃): δ /ppm 171.60 (COO), 168.95 (C=N), 163.53, 157.02, 154.34, 145.90, 135.77, 132.30, 131.19, 123.66, 122.19, 116.04, 104.96 for aromatic carbons, 63.64 (-O-CH₂-CH₃), 34.33, 31.86, 29.63, 29.61, 29.58, 29.53, 29.38, 29.29, 29.18, 29.01, 24.77, 24.62 for methylene carbons [CH₃-(CH₂)₁₄-COO-], 14.75 (-O-CH₂-CH₃), 14.05 [CH₃-(CH₂)₁₄-COO-]. EI-MS m/z (rel. int. %): 536(42) (M)⁺, 298(100).

3. Results and discussion

Structure elucidation of the title compounds was carried out via elemental analysis and spectroscopic techniques such as FT-IR, ¹H and ¹³C-NMR and EI-MS. The percentages of C, H and N from the elemental analysis conform to the calculated values for compounds *n*EBABTH (where $n=6, 8, 10, 12, 14, 16, 18$) and are tabulated in Table 1.

3.1. Phase transition behavior and liquid crystallinity of *n*EBABTH

The transition temperatures and associated enthalpy changes of *n*EBABTH obtained from the DSC measurements are summarized in Table 2. All the compounds exhibited enantiotropic properties whereby their endotherms were characterized by the crystal-mesophase-isotropic transitions occurring above the melting temperatures recorded during the heating and cooling runs. Such transitions were also supported by the enthalpy values of the respective compounds. As a representative illustration, the DSC thermograms of 8EBABTH and 10EBABTH recorded during the heating and cooling cycles are shown in Figure 2. Among all the compounds, only the endotherm of 14EBABTH appeared before the crystal-mesophase transition at 73.9°C in the DSC thermogram. The texture observed under POM further confirmed the presence of crystalline polymorphism before melting [12].

Observation of all the compounds under crossed polarizers revealed the schlieren and marble textures to be typical of a nematic mesophase (Figure 3a), as reported in the literature [10,11]. As the length of the terminal chain increases, the smectic phase emerges. Whilst 6EBABTH and 8EBABTH were purely nematogenic, the higher members,

Table 2. Phase transition and transition enthalpy changes of *n*EBABTH upon heating and cooling.

Compound	Phase transition temperature, °C heating cooling (associated enthalpy change, kJ mol ⁻¹)
6EBABTH	Cr 110.1 (36.0) N 142.0 (0.9) I I 139.8 (1.1) N 67.9 (30.4) Cr
8EBABTH	Cr 92.4 (30.4) N 134.1 (1.1) I I 131.8 (1.0) N 52.9 (26.1) Cr
10EBABTH	Cr 71.9 (33.6) SmC 79.6 (0.7) N 129.7 (1.1) I I 127.9 (1.4) N 76.3 (0.8) SmC 50.6 (27.8) Cr
12EBABTH	Cr 80.1 (36.9) SmC 93.8 (0.7) N 127.9 (1.2) I I 126.0 (1.6) N 91.9 (1.1) SmC 44.8 (19.3) Cr
14EBABTH	Cr ₁ 73.9 (7.6) Cr ₂ 80.5 (45.3) SmC 101.0 (0.9) N 122.8 (1.9) I I 119.0 (2.2) N 95.6 (2.7) SmC 53.0 (38.2) Cr
16EBABTH	Cr 87.0 (42.6) SmC 105.2 (0.8) N 120.4 (1.4) I I 118.1 (2.0) N 102.5 (0.9) SmC 61.4 (32.7) Cr
18EBABTH	Cr 91.4 (46.9) SmC 107.9 (0.6) N 117.2 (2.0) I I 114.1 (2.7) N 103.5 (0.6) SmC 70.6 (37.8) Cr

Note: Cr₁, Crystal 1; Cr₂, Crystal 2; SmC, Smectic C; N, Nematic; I, Isotropic.

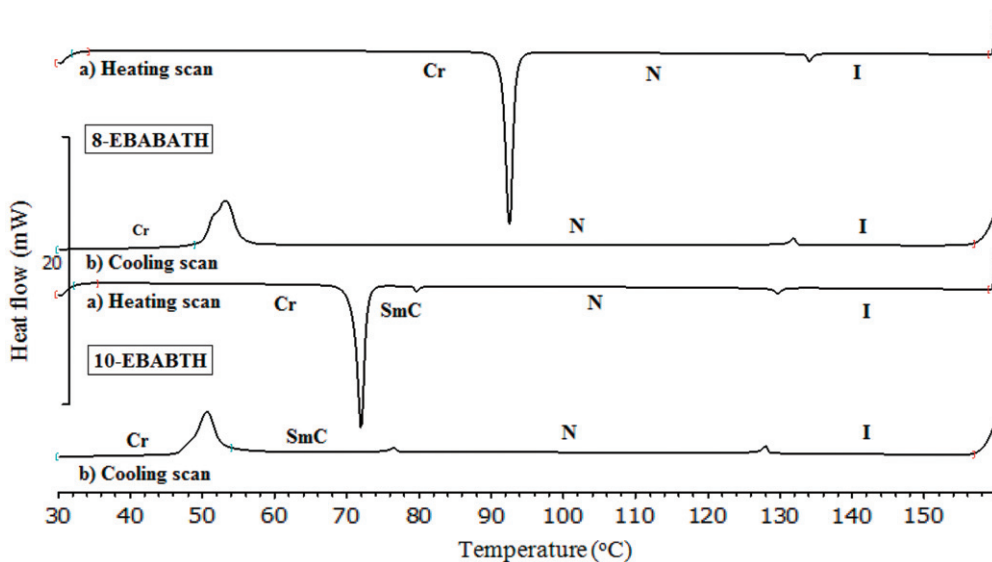


Figure 2. DSC thermograms of 8EBABTH and 10EBABTH.

*n*EBABTH ($n = 10, 12, 14, 16$ and 18) exhibited both nematic and tilted smectic C phases. Figure 3 depicts the representative optical photomicrographs of 10EBABTH during the cooling run. The smectic C phase was identified on the basis of its characteristic grey schlieren texture (Figure 3c) and by the appearance of the familiar fingerprint texture (Figure 3b) during the N-to-SmC transition [16].

A plot of the transition temperatures against the number of carbons in the alkanoyloxy chain during the heating cycle is depicted in Figure 4. According to the plot, it can be

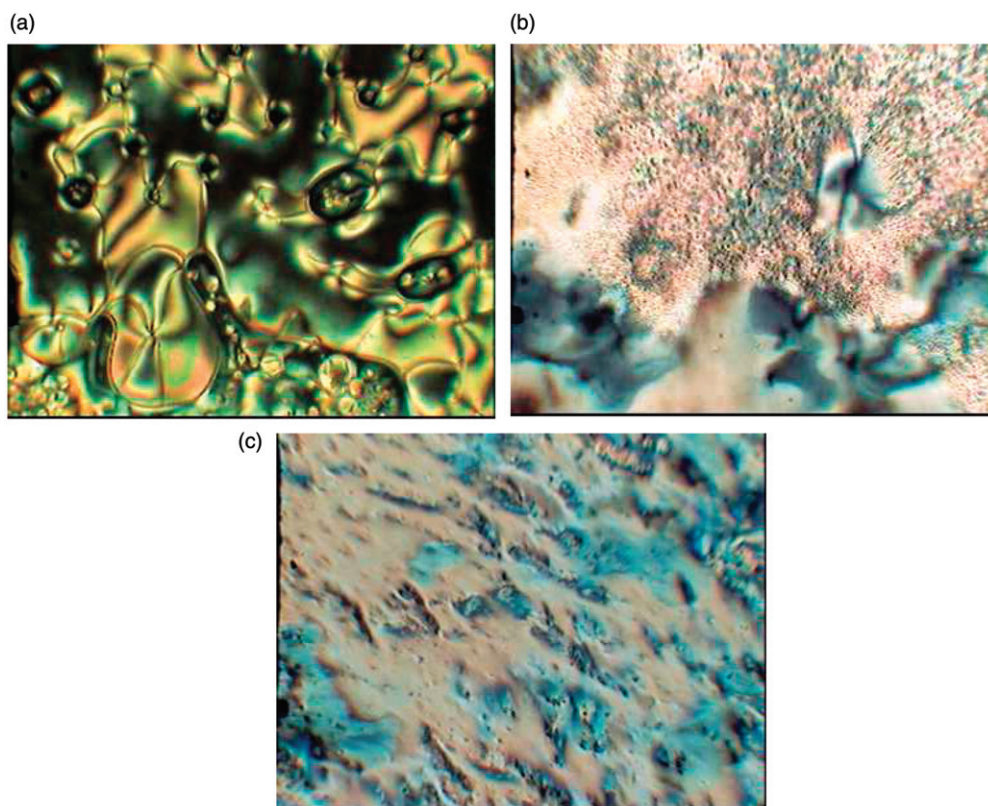


Figure 3. (a) Optical photomicrograph of 10EBABTH displaying nematic phase, (b) optical photomicrograph showing classical ‘fingerprint’ texture of smectic C phase at the N-to-SmC transition, (c) optical photomicrograph exhibiting SmC phase with grey *schlieren* texture in homeotropic cell.

deduced that the mesomorphic behaviors were greatly influenced by the length of the terminal alkanoyloxy chain. High melting point in short chain members is normally attributed to the high rigidity of the molecular core [3]. As can be seen in the graph, the shortest chain member, C₆, possessed the highest melting temperature (110.1°C) among the homologues series. The melting point was found to be decreased as the chain length increased to C₁₀ (71.9°C). This corresponded to the increasing flexibility of the longer alkyl chain. It is also common that the melting temperatures showed an ascending trend from medium chain members onwards after a falling trend from short to medium chain members [15–18]. Increase of melting temperatures was also observed from C₁₀ (71.9°C) to C₁₈ (91.4°C) members. It was probably due to the increasing Van der Waals attractive forces between the molecules. Nevertheless, clearing temperatures give a descending trend as the chain length increased. The smooth falling trend in clearing temperature can be ascribed to the dilution of mesogenic core [19]. Similar falling trend was also reflected on the homologous series of *N,N'*-bis(3-methoxy-4-alkoxybenzylidene)-1,4-phenyldiamine in which the homologue with the longest terminal chain possessed the lowest thermal stability [19].

Looking across the graph, it can be noted that the mesomorphism was also affected by the terminal chain length. The nematic phase range was found to be decreased as the chain

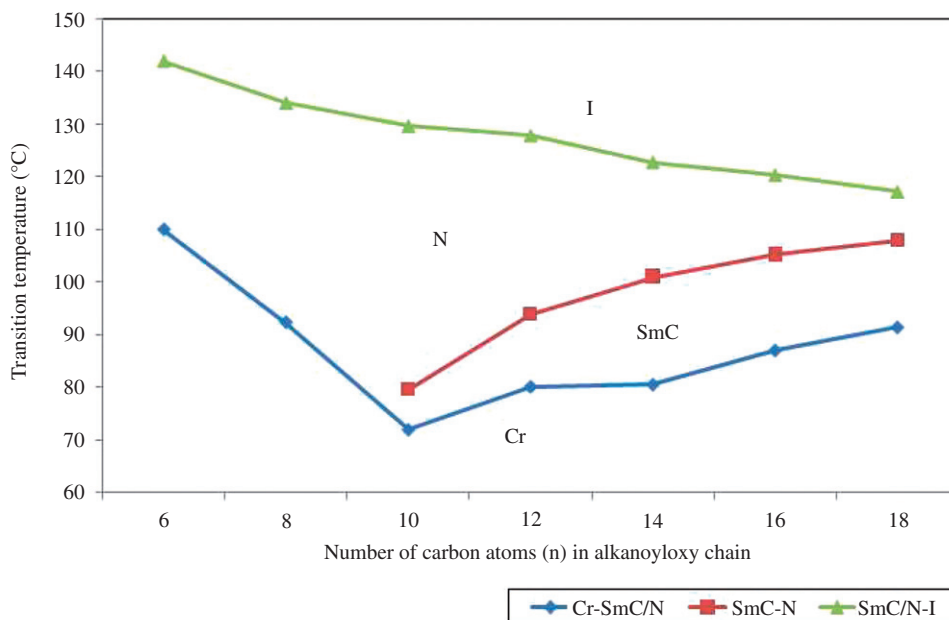


Figure 4. Plot of transition temperatures versus the number of carbons (n) in the alkanoyloxy chain of n EBABTH during heating cycle.

length ascended from C_6 (31.9°C) to C_{10} (50.1°C) member. However, the emergence of SmC phase dramatically depressed the nematic phase range to only 9.3°C in C_{18} members. This may due to the attractions between the long alkanoyloxy chains leading to their intertwining and facilitates the lamellar packing which is essential for the occurrence of the smectic phase [13]. The suppression of nematic phase range was accompanied by the uplifting of SmC phase range. For example, SmC phase range increased from C_{10} (7.7°C) to C_{18} (16.5°C) members. Increasing Van der Waals forces not only raised the melting temperatures but also enhanced the smectic phase stability whereby the SmC phase stability increased from 79.6°C (C_{10}) to 107.9°C (C_{18}).

3.2. X-ray diffraction studies

In order to obtain further information regarding the molecular arrangement in the mesophase, temperature-dependent XRD measurements were performed on 12EBABTH and the diffractogram is depicted in Figure 5, while the X-ray diffraction data are summarized in Table 3.

As seen in Figure 5, the diffraction pattern at 90°C showed a sharp peak at a lower region angle and a weak and broad peak at a wider angle. This kind of diffraction pattern is typical of a layer structure observed for the SmC phase [20]. The sharp diffraction peak at 2.59° implies the formation of a layered structure. On the other hand, the broad diffraction peak in the wide-angle region indicates liquid-like arrangement of the molecules within the layer.

From the XRD analysis data, the d -layer spacing (29.51 Å) was much smaller than the molecular length (33.167 Å) obtained by the MM2 molecular calculation, and

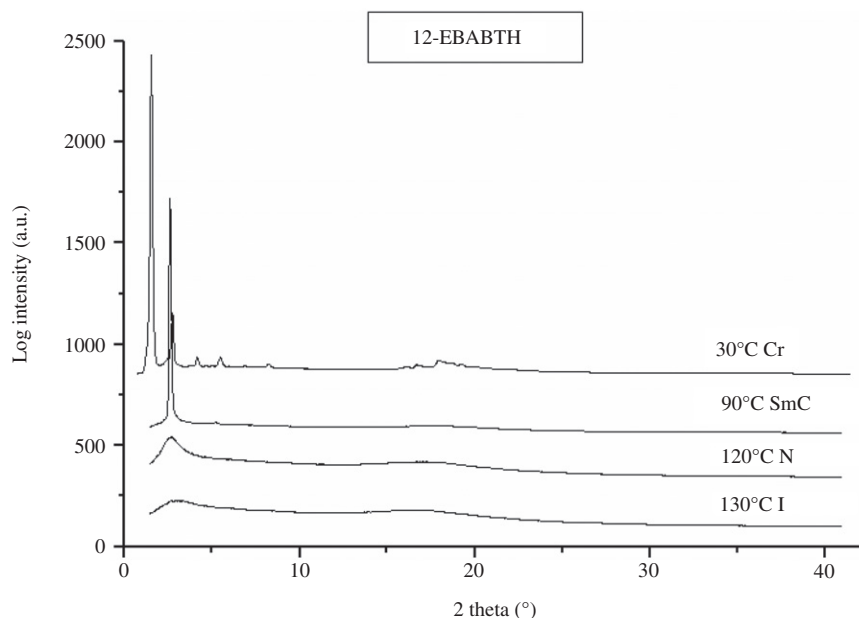


Figure 5. X-ray diffractogram of 12EBABTH at different temperatures upon cooling from the isotropic phase.

Table 3. Powder X-ray diffraction data of 12EBABTH.

Phase observed	SmC
Temperature (°C)	90
2 theta (°)	2.59
<i>d</i> -spacing (Å)	29.51
Length, <i>l</i> (Å)	33.167
Aspect ratio (<i>d/l</i>)	0.89

the *d/l* ratio was 0.89, which confirms a titled SmC phase being observed for 12EBABTH [21].

4. Conclusions

In this article, we have reported the synthesis and mesomorphic properties of a homologues series of 6-ethoxy-2-(4-alkanoyloxybenzylideneamino)benzothiazoles. The enantiotropic nematic phase was observed throughout the entire series. The enantiotropic SmC phase, however, only emerged starting from the decanoyloxy to the octadecanoyloxy derivatives. The study also revealed that the mesophase range was greatly affected by the length of the terminal chain.

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