

Synthesis and Characterization of New Derivatives of Thiazole with Liquid Crystalline properties

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Abstract

A new two series of liquid crystalline Schiff bases containing thiazole moiety with different length of alkoxy spacer were synthesized, and the relation between the spacer length and the liquid crystalline behavior was investigated. The molecular structures of these compounds were performed by elemental analysis and FTIR, ¹HNMR spectroscopy. The liquid crystalline properties were examined by hot stage optical polarizing microscopy (OPM) and differential scanning calorimetry (DSC). All compounds of the two series display liquid crystalline nematic mesophase. The liquid crystalline behaviour has been analyzed in terms of structural property relationship.

Keywords: Thiazole, Liquid crystal, Heterocyclic, Schiff base

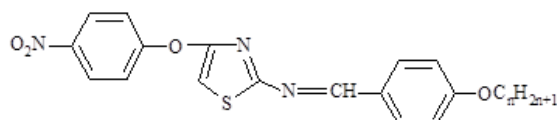
1. Introduction

Thiazole is a heterocyclic compound containing both nitrogen atom and sulfur atom as a part of aromatic five-membered ring. Heterocyclic compounds containing thiazole rings represent a very important group of organic compounds, which are also found in certain natural products i.e. vitamin B1 (thiamine) and the penicillin (Masquelina and Obrecht, 2001).

Thiazole can be prepared using both classical and nonclassical approaches example. 2-Aminothiazole and its derivatives generally are prepared by the reaction of thiourea with α -halogencarbonyl compounds (Land et al., 1947)

Many previous studies were investigated the effect of the structure of azomethines with thiazole rings on the type of liquid crystalline properties and its temperature range, with introduction of alkoxy groups with varying length of the aliphatic chain into the benzylidene component (Murza et al., 1996, 1995). Kuvatov et. al., (Kuvatov et al., 2004) have been synthesized azomethines containing a thiazole ring and their liquid crystal properties have been investigated. The influence of the central group and substituent in the azomethine component on the type and temperature range of the mesomorphous properties of the compounds synthesized has been elucidated. 2-(4-Alkoxybenzylideneamino)-4-[(4'-nitrophenyl)amino] thiazoles have been synthesized (Golovanov et al., 1997). They showed enantiotropic nematic type mesomorphism in range 41-184°C.

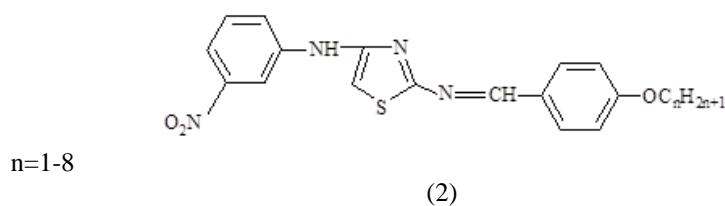
Murza et. al., (Murza et al., 1999) have been synthesized 2-(4-alkoxybenzyliden-amino-4-(4'-nitro- phenoxy) thiazoles (1), which possess monotropic mesomorphism of the nematic type over the range 68-160 °C.



n=1-8

(1)

Also, Murza et. al., (Murza et al., 2001) have been synthesized new liquid crystalline azomethines containing thiazole ring (2) displaying nematic mesomorphism at 42-137°C and studied a correlation between the geometric parameters and liquid crystalline properties.



The rapid development of liquid crystal research is related to the unique optical properties of these substances which have led to their wide application in the data processing industry. Therefore, in this study Schiff bases of 2-aminothiazole containing symmetrical and unsymmetrical terminal alkoxy substituents in one side and both sides was synthesized and their liquid crystalline properties were investigated.

2. Experimental

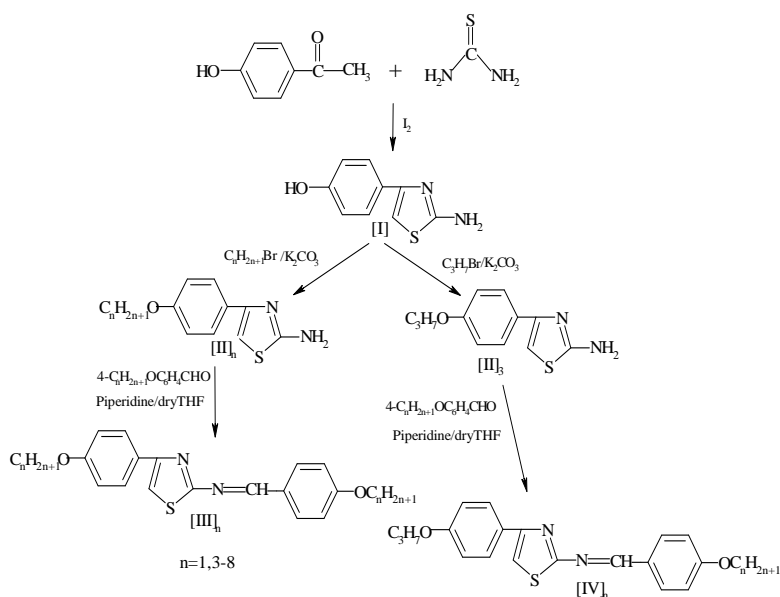
2.1. Chemicals

All the chemicals used in this study were supplied by Aldrich Chemicals and were used as received.

2.2. Techniques

Elemental analysis was carried out using a Perkin-Elmer model 2400 instrument. Infrared spectra were recorded with a Shimadzu 8000 FT-IR spectrophotometer in the wave number range $4000-400\text{ cm}^{-1}$ with samples embedded in KBr disc. $^1\text{H-NMR}$ spectra were obtained with Bruker, model: ultra-shield 300 MHz using suitable deuterated solvent and TMS as internal standard. $^{13}\text{C-NMR}$ spectra of the compound were recorded on Em-360 MHz spectrometer. The optical behavior observations were made using Olympus BX40 microscope equipped with a Leitz Laborlux 12 Pols hot stage and PR600 controller. The textures shown by the compounds were observed using polarized light with crossed polarizers with the sample in a thin film sandwiched between a glass slide and cover slip. Differential scanning calorimetry (DSC) measurements were conducted with TA instruments Q1000 DSC, Ramp rate: 10 degree centigrade per minute under nitrogen atmosphere. Temperature and heat flow calibrated with standard indium of purity >99.99%.

The chemical structure and synthetic route for the two series are illustrated in Scheme 1. The experimental details are described as follows:



Scheme 1. The preparation route of series [III]_n and [IV].

2.3. Synthesis of 4-n-Alkoxybenzaldehyde

4-n-alkoxybenzaldehyde was obtained following the procedure described in (Tomma et al., 2009).

2.4. Synthesis of 2-amino-4-(4'-hydroxyphenyl)-1,3-thiazole [I]

In a flask of 250 mL added (7.61 g, 0.03 mol) of iodide, (4.56 g, 0.06 mol) of thiourea and (4.56 g, 0.03 mol) of 4-hydroxyacetophenone. The mixture was heated for 8 h on the water bath. Cooled, washed by diethyl ether and solution of sodium thiosulfate. The precipitate was filtered off and crystallized from ethanol. Yield 89%, mp = 148-150 °C, Lit.(King and Hlavacek, 1950), 151 °C.

2.5. Synthesis of 2-Amino-4-(4'-alkoxyphenyl)-1,3-thiazole [II]_n

A mixture of compound [I] (0.28 g, 1.5 mmol) and anhydrous potassium carbonate (0.80 g, 2 mmol) dissolved in 20 mL acetone, then n-alkylbromide (4 mmol) was added, the mixture was refluxed overnight. Afterward, the mixture poured in to ice water the precipitate filtered and washed with water, dried and recrystallized from ethanol.

n	1	2	3	4	5	6	7	8
Yields (%)	60	85	83	75	75	65	85	75

2.6. Synthesis of 2-(4'-Alkoxybenzylideneamino)-4-(4'alkoxyphenyl)-1,3-thiazole[III]_n

A mixture of compound [II]_n (0.042 mol) and 4-n-alkoxybenzaldehyde (0.042 mol) in THF(40 mL) was refluxed for 4 h in the presence of a catalytic quantity of piperidine. The solvent was evaporated and the residue was extraction by diethyl ether and purified by column chromatography.

n	1	2	3	4	5	6	7	8
Yields (%)	80	70	85	80	75	85	85	90

2.7. Synthesis of 2-(4'-alkoxybenzylideneamino)-4-(4'propanoxyphenyl)-1,3-thiazole [IV]

These compound were prepared analogously using compound [III]₃ instead of compounds [III]_n.

n	1	2	3	4	5	6	7	8
Yields (%)	70	70	80	70	75	65	70	75

3. Results and Discussion

3.1. Characterization

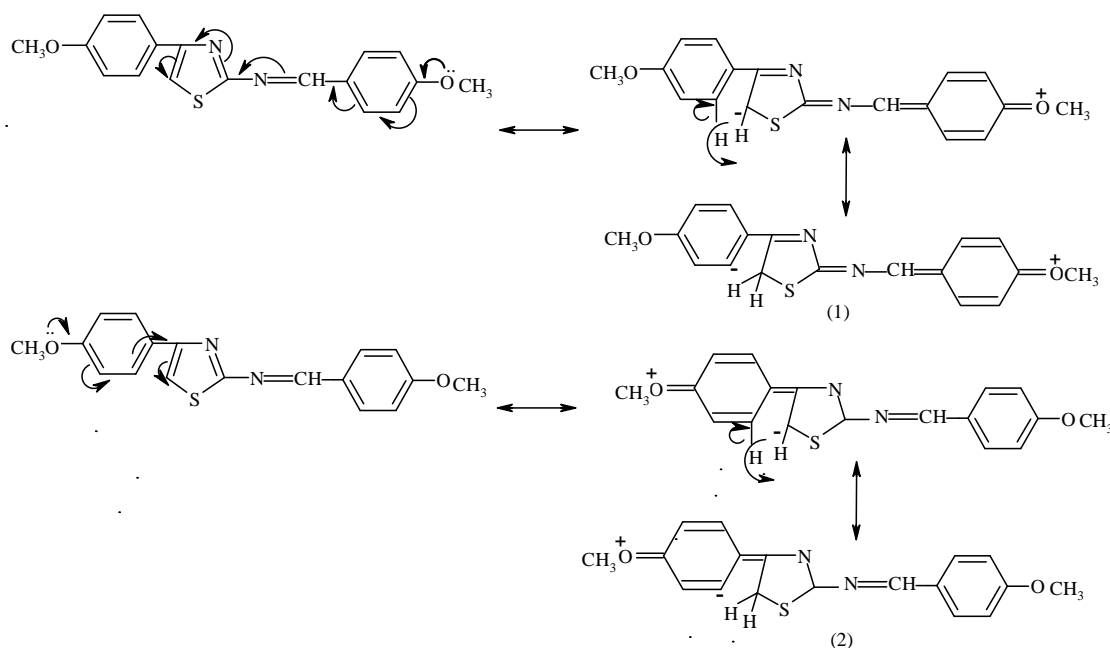
The compounds [II]_n were prepared by the reaction of 2-amino-4-(4'-hydroxyphenyl)-1,3-thiazole [I] with different 4-n-alkylbromide in alkaline media. All synthesized compounds of series [II]_n were characterized by elemental analysis and FTIR. The FTIR spectra of compounds [II]_n show a characteristic stretching bands of the aliphatic ν_{C-H} group in the regions (2997-2854 cm^{-1}) for alkyl group. Elemental analysis result of compound [II]₅ as an example is in good agreement with the theoretical data. Calc. for $\text{C}_{14}\text{H}_{19}\text{ON}_2\text{S}$ [II]₅; C, 65.11; H, 6.97; N, 10.85; S, 12.40; Found: C, 64.84; H, 6.64; N, 10.55; S, 12.61.

The Schiff bases of series [III]_n and [IV]_n were prepared by refluxing of compounds [II]_n or [II]₃ respectively with 4-n-alkoxybenzaldehyde in THF solution and piperidine as a catalyst, the synthesized compounds were checked by TLC and purified by column chromatography to yield pure products. All synthesized compounds of series [III]_n and [IV]_n were characterized by elemental analysis and FTIR, ^1H NMR and ^{13}C NMR spectroscopy. The elemental analysis of compounds [III]₄ and [IV]₄ as an example are consistent with their proposed structure. Calc. for $\text{C}_{24}\text{H}_{29}\text{O}_2\text{N}_2\text{S}$ [III]₄; C, 70.58; H, 6.86; N, 6.86; S, 7.84; Found: C, 70.12; H, 6.99; N, 6.25; S, 8.36. Calc. for $\text{C}_{23}\text{H}_{27}\text{O}_2\text{N}_2\text{S}$ [IV]₄; C, 70.05; H, 6.59; N, 7.10; S, 8.12; Found: C, 70.35; H, 6.32; N, 7.12; S, 8.26.

The FTIR spectra of compounds [III]_n and [IV]_n showed the disappearance of bands of NH₂ groups for compounds [II]_n or [III]₃ respectively, with the appearance of stretching bands in the regions (1681-1689 cm⁻¹) and (1694-1693 cm⁻¹) respectively, due to azomethine ν_{CH=N} groups. The ¹HNMR spectrum (in DMSO as a solvent) for compound [III]₁ as an example showed: a weak signals at δ9.05 ppm for one proton of N=CH group, a singlet at δ7.82 ppm for one proton of thiazole ring, two doublet at δ (7.91-8.03) ppm that could be attributed to the aromatic protons also two doublet at δ (7.03-7.019) ppm for another aromatic protons, two singlet at δ 3.82 and 3.88 ppm due to six protons of two OCH₃ groups.

The ¹³CNMR spectrum of compound [III]₁ displays the signals corresponding to the different non-equivalent carbon atoms at different values of δ as follows: at δ55.66 and 56.07 ppm for two group of (OCH₃) corresponding to carbon atoms of methoxy groups; four signals at δ 111.25 – 115.15 ppm due to (C=C) carbon atom of benzene ring; also four signals at δ 127.76 –132.29 ppm due to aromatic carbon atoms for another benzene ring ; at δ152.75 ppm due to C=C (C5) carbon atom of thiazole ring; at δ159.71 ppm due to C=C (C4) carbon atom of thiazole ring; at δ 163.80 ppm due to (C=N) carbon atom of azomethine and at δ 172.51 ppm due to C=N (C2) carbon atom of thiazole ring.

The dept 135 spectrum of compound [III]₁ displays the signals corresponding to carbon atoms with odd hydrogens (positive peaks) will carbon atoms with even hydrogen and quaternary disappearance (negative peaks) (Silverstein et al., 2005), therefore appearance at δ55.66 and 56.07 ppm for two group of (OCH₃) corresponding to carbon atoms of methoxy groups; four signals at δ 111.25 – 115.14 ppm due to (C=C) carbon atom of benzene ring; three signals at δ 127.75 – 132.29 ppm due to aromatic carbon atoms for benzene ring near the thiazole ring ;at δ 163.80 ppm due to (C=N) carbon atom of azomethine. This spectrum show the disappearance of signals at δ 152.75,159.71 and 172.51 ppm corresponding to carbon atoms C5, C4and C2 respectively, this is evidence by the following tautomerizm in Scheme 2. In Scheme 2, (1) and (2) show electron density (availability of π electrons) at C5 and activated this position, which is thus attacked easily by electrophilic reagents (Ganapathi et al., 1952), Scheme 2 show electron density at C5 and attacked hydrogen from near benzene ring therefore C5 containing tow hydrogens and disappearance signals .



Scheme 2. Tautomerizm of compound [III]₁

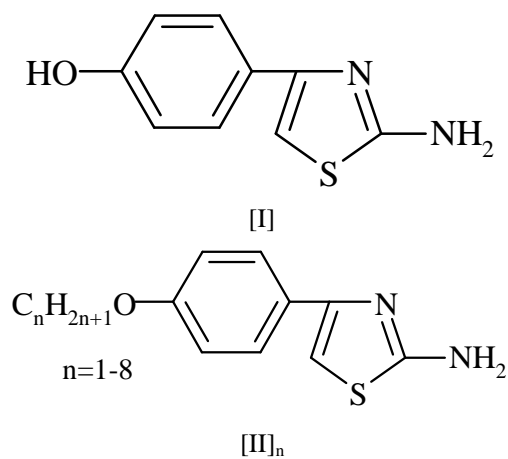
The ¹HNMR spectrum (in CDCl₃ as a solvent) for compound [III]₆ showed: a sharp signals at δ9.81 ppm for one proton of N=CH group, two doublet at δ (6.91-7.96) ppm that could be attributed to the eight aromatic protons, signal at δ6.86 ppm proton of thiazole ring, triplet at δ(3.73-3.69) ppm for four proton of two OCH₂ groups, multiplet at δ(1.20-1.83) ppm for sixteen proton of eight CH₂ groups, signal at δ(0.65-1.00) ppm for six proton of two CH₃ groups.

The ^1H NMR spectrum (in DMSO as a solvent) for compound [IV]₆ showed: a sharp signals at δ 9.61 ppm for one proton of N=CH group, two doublet at δ (6.59-7.82) ppm that could be attributed to the eight aromatic protons of phenyl rings and proton of thiazole ring, triplet at δ (3.62-4.39) ppm for four proton of two OCH₂ groups, multiplet at δ (1.15-1.84) ppm for ten proton of five CH₂ groups, signal at δ (0.70-1.00) ppm for six proton of two CH₃ groups.

3.2. Liquid Crystalline Properties

The transition temperatures and mesophase type (texture identity) of all compounds were investigated using hot-stage optical polarizing microscopy (OPM) and differential scanning calorimetry (DSC).

3.2.1. Mesomorphic Properties of Compound [I] and Series [II]_n



The phase transition temperatures for compound [I] and series [II]_n compounds were summarized in Table 1. All compounds [I] and [II]_n display enantiotropic mesomorphism. The compound [I] shows enantiotropic nematic texture under OPM as shown in Figure 1, while the compounds of series [II]_n exhibit an anantiotropic dimorphism smectic C (SmC) and nematic (N) phases. The nematic phase of compound [I] could be attributed to the formation of intermolecular hydrogen bonding (Figure 2), i.e., terminal molecular interaction which is a favor of nematic mesophase formation (Byron et al., 1980).

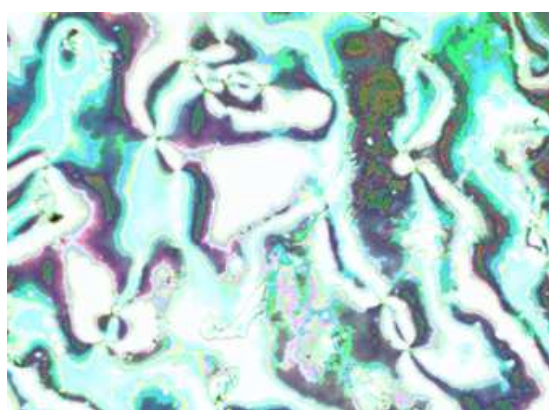


Figure 1. Cross polarizing Optical textures of the nematic mesophase obtained on cooling (Magnification 200X) for compound [I] at 145°C.

Table 1. Phase transition temperatures (°C) of compounds [I] and [II]_n.

Compound	Phase transition
[I]	Cr $\xrightleftharpoons{122}$ N $\xrightleftharpoons{150}$ I
[II] ₁	Cr $\xrightleftharpoons{155}$ N $\xrightleftharpoons{171}$ I
[II] ₂	Cr $\xrightleftharpoons{235}$ N $\xrightleftharpoons{250}$ I
[II] ₃	Cr $\xrightleftharpoons{132}$ N $\xrightleftharpoons{151}$ I
[II] ₄	Cr $\xrightleftharpoons{73}$ N $\xrightleftharpoons{119}$ I
[II] ₅	Cr $\xrightleftharpoons{58}$ N $\xrightleftharpoons{113}$ I
[II] ₆	Cr $\xrightleftharpoons{75}$ Sc $\xrightleftharpoons{94}$ N $\xrightleftharpoons{106}$ I
[II] ₇	Cr $\xrightleftharpoons{54}$ Sc $\xrightleftharpoons{70}$ N $\xrightleftharpoons{102}$ I
[II] ₈	Cr $\xrightleftharpoons{49}$ Sc $\xrightleftharpoons{85}$ N $\xrightleftharpoons{110}$ I

Cr, crystalline phases; Sc, smectic C phase; N, nematic phase; I, isotropic liquid

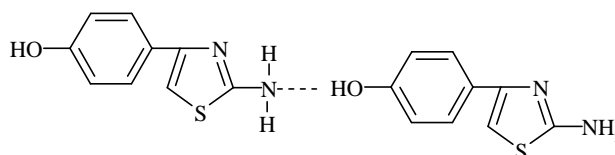


Figure 2. Hydrogen bonding formation for compound [I].

For this reason the compounds [II]₁-[II]₅ present a nematogenic enantiotropic behaviors, because the alkoxy chain is shorter led to a higher ratio of t/l intermolecular attraction forces. While compounds [II]₆-[II]₈ display smectic and nematic phases, in this case increase in the alkoyl chain length which increases the isolation of the polarity and polarizability of the terminal group (Collings, 1995), leading to a lower ratio of t/l intermolecular attractions, which is a favor of smectic mesophase formation. In addition, these compounds containing amino group that conjugate with thiazole ring increasing lateral forces, forming both smectic and nematic mesophases. Figure 3 display the smectic C (SmC) for compound [II]₇.

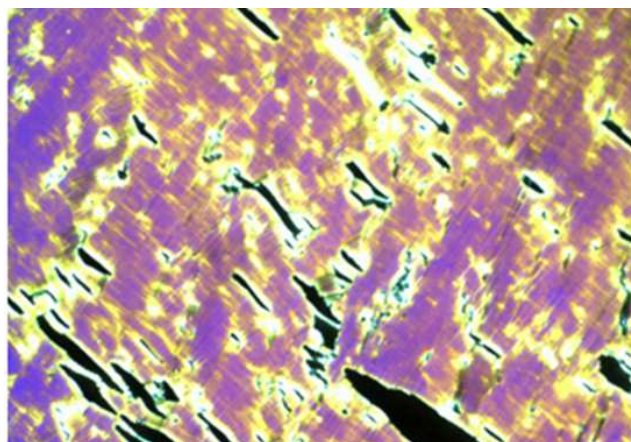
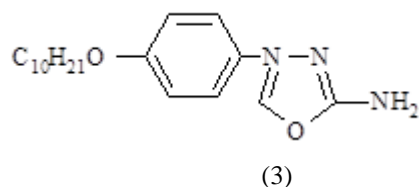
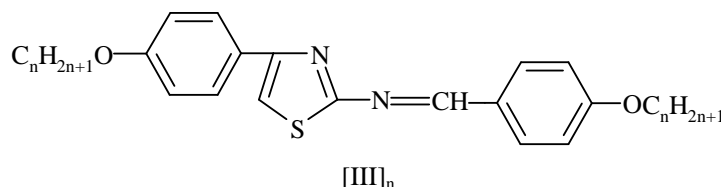


Figure 3. Cross polarizing Optical textures of the smectic C, SmC mesophase obtained on cooling (Magnification 200X) for compound [II]₇ at 60°C.

In order to show the different possibilities that thiazole ring can offer in the liquid crystal field, it is interesting to compare the present homologous series [II]_n and amine [103] prepared by Chudgar et. al., (Chudgar 1989) the former have thiazole ring at a core of molecules, but the latter have oxadiazole ring instead of thiazole ring. By comparison it was found that amino oxadiazole compound (3) is not mesogenic, whereas all amino thiazole in series [II]_n showed liquid crystal properties.



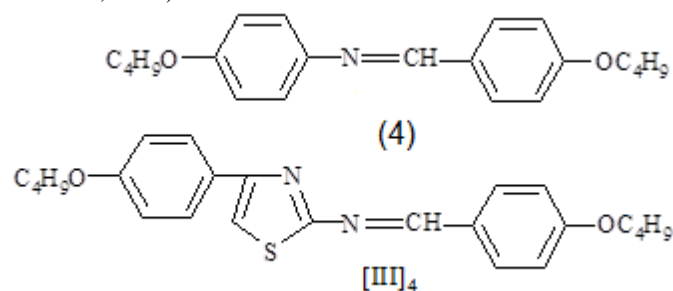
3.2.2. Mesomorphic Properties of Series [III]_n



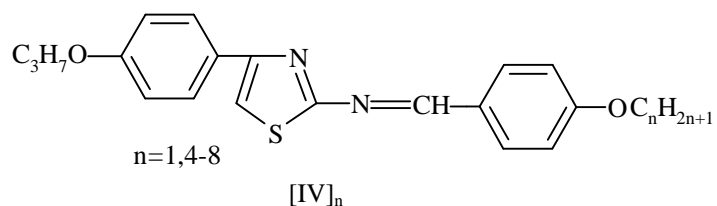
The phase transition temperatures for compounds [III]_n are summarized in Table 2. All compounds of series [III]_n were found to be enantiotropic nematic phase. The replacement of amino group for compounds [II]_n by imino linkage in compounds [III]_n led to decrease of lateral attraction forces and absence of smectic phase. In addition, Schiff base make molecules more linear, and for this reason these compounds showed nematic phase only.

It is interesting to compare compound [III]₄ with compound (4) synthesized by Yousif et. al., (Yousif et al., 1990). Compounds [III]₄ and (4) have the same central rigid core and the same alkoxy chain. The difference between those compounds is the thiazole ring. The results show that the presence of thiazole ring has a profound influence on the mesogenic properties. While compound [III]₄ exhibit a nematic phase, compound

(4) display a SmA phase. Clearly, the presence of thiazole ring in central core favours the nematic phase, indicating that the thiazole ring induce flexibility in molecular structure to dominate phase structure in nematic liquid crystal (Parra et al., 1997).



3.2.3. Mesomorphic Properties of Series [IV]_n



The phase transition temperature of this series is collected in Table 3. All compounds of series [IV]_n display liquid crystalline properties except the compound [IV]₈. Compounds [IV]₁-[IV]₇ display enantiotropic mesomorphism. The texture observed by POM on cooling from the isotropic liquid are consistent with the presence of nematic mesomorphism, with a typical thread like texture as shown in Figure 4. The homologue [IV]₈ do not reveal any liquid crystalline behavior, but simply changes from the solid crystalline state to the isotropic liquid at 78-80°C. The extended of terminal alkoxy substituent could be responsible for diminishing of mesomorphism in compound [IV]₈ (Champa, 1974).

Table 2. Phase transition of series [III]_n

Compound	Phase transition
[III] ₁	Cr $\xrightleftharpoons{40}$ N $\xrightleftharpoons{127}$ I
[III] ₃	Cr $\xrightleftharpoons{91}$ N $\xrightleftharpoons{148}$ I
[III] ₄	Cr $\xrightleftharpoons{60}$ N $\xrightleftharpoons{82}$ I
[III] ₅	Cr $\xrightleftharpoons{68}$ N $\xrightleftharpoons{79}$ I
[III] ₆	Cr $\xrightleftharpoons{51}$ N $\xrightleftharpoons{75}$ I
[III] ₇	Cr $\xrightleftharpoons{39}$ N $\xrightleftharpoons{50}$ I
[III] ₈	Cr $\xrightleftharpoons{48}$ N $\xrightleftharpoons{85}$ I

Table 3. Phase transition of series [IV]_n

Compound	Phase transition
[IV] ₁	Cr $\xrightleftharpoons{35}$ N $\xrightleftharpoons{65}$ I
[IV] ₄	Cr $\xrightleftharpoons{110}$ N $\xrightleftharpoons{123}$ I
[IV] ₅	Cr $\xrightleftharpoons{46}$ N $\xrightleftharpoons{152}$ I
[IV] ₆	Cr $\xrightleftharpoons{62}$ N $\xrightleftharpoons{81}$ I
[IV] ₇	Cr $\xrightleftharpoons{102}$ N $\xrightleftharpoons{201}$ I
[IV] ₈	Cr $\xrightleftharpoons{78-80}$ I

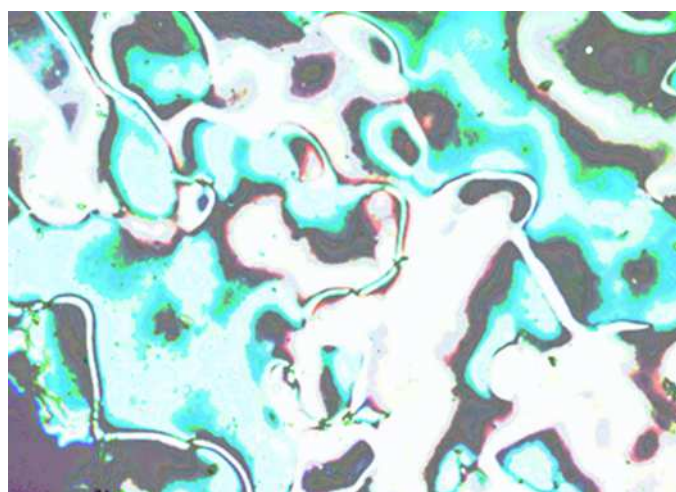


Figure 4. Cross polarizing Optical textures of the thread-like nematic mesophase obtained on cooling (Magnification 200X) for compound [IV]₇ at 155°C.

4. Conclusions

Different symmetrical and non-symmetrical mesogenic homologous series of terminal alkoxy substituent (series [II]_n, [III]_n and [IV]_n) incorporating thiazole ring were synthesized. All compounds of the first and the second series exhibited liquid crystalline properties with anantitropic nematic mesophase. All compounds of the third series [IV]_n also show mesomorphic behaviour, except the last homologue which simply changed to isotropic liquid. The mesomorphic behaviour has been analyzed in terms of structural property relationship and compared with other homologous series of comparable structural formula.

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