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Bent liquid crystal compounds containing heterocyclic: Synthesis and Characterization

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Abstract:

This article reported the synthesis and characterization of new bent-shape liquid crystals compounds containing pyrimidine as a core unit and a 5H-thiazolo[3,4-b] [1, 3,4] thiadiazols ring with an amid group. This study includes reaction 5,5-diethylpyrimidine-2,4,6(1H,3H,5H)-trione and two moles of chloroacetylchloride in *N*, *N*-dimethyl formamide (DMF) and triethylamine (TEA) to product compound [I], then reacted the later compound with two moles of 4-hydroxybenzaldehyde to yield compound [II] then synthesis compound [III] by reaction compound[II] , thiosemicarbazide and mercaptoacetic acid with sulfuric acid concentration, then the compound [III] reaction with deferent acid chlorides to product amid compounds [IV-VII]. The compounds were identified by melting points, FTIR and ^{1}HNMR spectroscopy. The esomorphic behavior was examined using Differential scanning calorimetry (DSC) and a polarized optical microscope.

Keywords: Bent liquid crystal, heterocyclic, Nematic phase; Smectic phase.

مركبات بلورية سائلة منحنية تحتوي حلقة غير متجانسة: تخليق وتوصيف

مريم ناصر لازم ، نسرين حسين كرم
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مستخلص:

-تشمل هذه المقالة تخليق وتوصيف مركبات جديدة من البلورات السائلة ذات الشكل المنحني تحتوي على البيريميدين كوحدة مركزية وحلقة 5H-ثيازولو[3,4-b][1,3,4]ثياديازول مزودة بمجموعة أميد. تشمل هذه الدراسة التفاعل بين 5,5-ثنائي إيثيل بيريميدين2,4,6-(1H,3H,5H)-تري ون ومولين من كلوروأسيتيل كلوريد في ثنائي ميثيل فورماميد (DMF) مع ثلاثي إيثيل أمين (TEA) لإنتاج المركب [I]، ثم تفاعل المركب الناتج مع مولين من 4-هيدروكسي بنزالدهيد للحصول على المركب [II]. بعد ذلك، تم تخليق المركب [III] من خلال تفاعل المركب [II] مع ثيوسمكربازيد وحامض الميركابتوأسيتك بوجود حامض الكبريتيك المركز، ثم تم تفاعل المركب [III] مع كلوريدات الحامض مختلفة لإنتاج مركبات أميد [IV–VII].

-تم تحديد هوية المركبات باستخدام درجه الانصهار، وأطياف الأشعة تحت الحمراء (FTIR)، وأطياف الرنين المغناطيسي النووي للهيدروجين (^{1}HNMR). كما تم فحص السلوك الميزومورفي للمركبات باستخدام المسح الحراري التفاضلي (DSC) والمجهر الضوئي المستقطب.

Introduction

It is possible to think of the liquid crystal state as a fourth (real) state of matter, with qualities halfway between those of the solid and liquid states it borders. The formation of liquid crystals (LCs) between the solid crystal and isotropic liquid phases the process of generating the liquid crystals (LCs) by combining the solid crystal and isotropic liquid phases is determined by the structure and geometry of molecules, which are important structural features of LCs [1]

There are Lyotropic liquid crystals are one of the two main kinds of liquid crystals. and thermotropic liquid crystals, Thermotropic behavior is caused by three basic molecular shapes: long rods, discs, and molecules with a bend (typically 120°) at the bent-core liquid crystals' molecular core [2].

LCs are valuable materials with a wide range of uses [3-5] . Several compounds synthesis including thermotropic LC molecules[6,7].

The task of designing new compounds as functional materials typically entails investigating novel heterocycles and aromatic systems. Furthermore, interactions molecular entities dependent by generating electrostatic and supramolecular shape-, the presence of a heterocyclic molecular system raises the molecular dipole and provides a certain degree of molecular polar aggregation[8-11]. As a component of our research, this study [12-18]

The shape of the liquid crystal-forming building blocks has an enormous impact on the way they arrange into different phases For instance, while simple rod-like molecules just form a handful of liquid crystal phases curved or banana-shaped molecules form more than fifty. These 'banana phases' were discovered almost 30 years ago and their spontaneous chirality and polarity have initiated a true 'banana-mania' in the field of liquid crystals [19].

The unique combination, leads LCs to exhibit a range of anisotropic physical properties, such as direction-dependant optical and mechanical properties e.g. optical modulators or tough LC spider silk fibers. Furthermore, LCs can be easily deformed and respond to external stimuli, such as electric fields or shear stresses [20], making them readily used in range of material processes, and in numerous dynamic processes in living organisms.

To prepare heterocyclic LC, we systematically synthesized bent liquid crystal compounds containing heterocyclic and investigated how they behave at transition phase the transition behavior To produce mesophases and the terminal substituent in the mesogenic behavior, we present an effective design of molecules.

Additionally, measurements were made to determine the influence of skeleton structure molecules as well as their ability to initiate the LC mesophases of the synthesized compounds.

Experimental

Materials and techniques

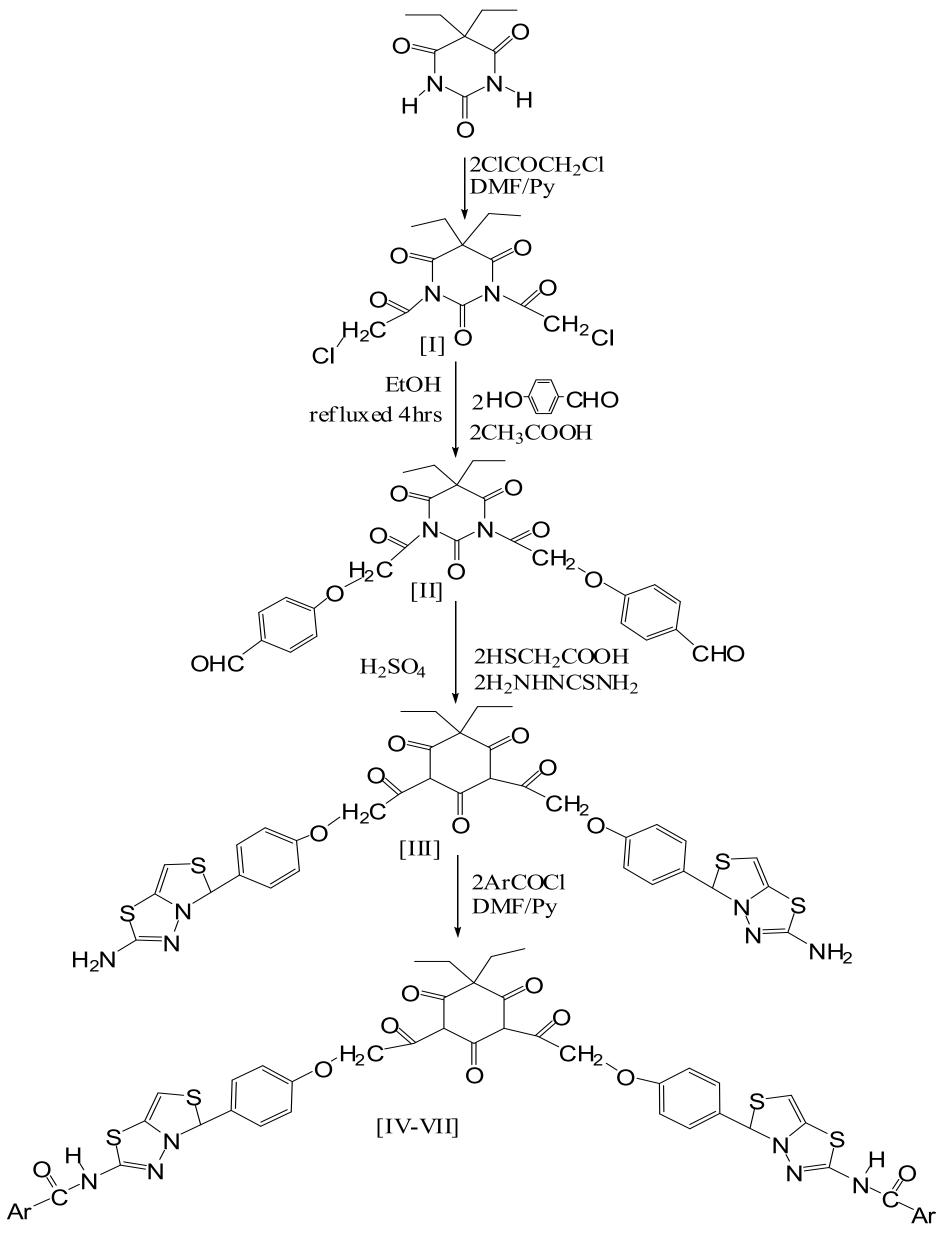
The materials of this study were provided from Merck Co., Fluka, and Aldrich.

Fourier Transform Infrared spectrum measured by SHIMADZU (IR Spirit-x series), range from 600 through 4000cm^{-1}. ^{1}H-NMR spectra analyzed by Bruker, 400 MHz in ppm (δ), the solvent materials DMSO-d6 were used to dissolve the materials. Mass spectrum were measured using Agilent model: 5975C USA. The melting point checked by Gallen Kamp technology. Using a model of a polarized optical microscope, the mesophase , the transition of the temperatures and textures were determined.

PW-BK 5000PR. . DSC measurements were taken using the Q600, a German-made device with an average heat rate of 5 degrees Celsius per minute

Synthesis

The synthetic pathway described in Scheme 1 was followed in the synthesis of the novel compounds.



Ar=4-OCH3C$_6$H$_4$, C$_6$H$_5$, CH$_3$, CH$_2$Cl

Scheme 1.Synthesis compounds [I-VII]

Preparation of compound[I]

5,5-diethylpyrimidine-2,4,6(1H,3H,5H)-trione (0.184 g, 0.001 Mol) in DMF (2 mL) was combined with pyridine (1mL) and chloroacetylchloride (0.226 g, 0.002 Mol) agitated in the bath of ice for three hrs [21]. After cooling adding the components to ice water, filtration then dried and recrystallized in ethanol. Reddish color brown , 74% yield, m.p. 125-126°C.

FTIR (ν /cm^{-1}): 2970,2879 (C-H aliph.), 1768-1672(C=Ogroups), 798(C-Cl). ^{1}H-NMR (400 MHz, DMSOd_6) (ppm): 4.41(s, 4H, 2COCH$_2$Cl), 2.15-1.25 (q,4H, 2CH$_2$CH$_3$), and 0.97-0.80 (t, 6H, 2CH$_3$)

Synthesis of compound [II]

The 4-hydroxybenzaldehyde(0.244 g, 0.002 mol) was mixed with compound [I] (0.337g, 0.001mol) and sodium acetate fused (0.164 g, 0.002mol) in 5 mL of ethanol, and then refluxed for four hours[22]. After that, it was cooled by cold water. The solid was then filtered, separated from ethanol, dried, and then recrystallized. The molecular formula: $C_{26}H_{24}N_2O_9$, Yield 78 %, Color off white, m.p.= 102-105 °C

FTIR (ν /cm^{-1}): 3046 (Ar-H), 2970, 2829, (C-H aliph), 2756(C-H aldehyde), 1763-1664 (C=O groups),1592 (C=C Ar), 1213 (C-O). ^{1}HNMR (400 MHz, DMSO-d6) δ(ppm):0.73-0.78(t,6H,2CH$_3$)1.79-1.92(q,4H,2CH$_2$),3.75(s,4H, 2CHO),6.92-7.80(m,8H,Ar-H),9.80(s,2H,2CHO).m/z=360,299,185,155 (base peak),126,106,98,77,65,54

Synthesis of compound [III]

Compound [II] (0.51g, 0.001 mol), as well as mercaptoacetic acid (0.184 g, 0.002 mol) were stirred for 20 to 25 minutes. Afterward, thiosemicarbazide (0.182 g, 0.002 mol) was added, and after cooling, 15 mL H_2SO_4 con. was added in portions. For 24 hours,the mixture was stored in the freezer. 40 grams of crushed ice were then added, along with 40% aqueous sodium hydroxide, to produce

mol) was added, and after cooling, 15 mL H_2SO_4 con. was added in portions. For 24 hours, the mixture was stored in the freezer. 40 grams of crushed ice were then added, along with 40% aqueous sodium hydroxide, to ~~produce~~ obtain pHis7-8. The solid filtered[23], then recrystallized via ethanol.

Molecular formula $C_{32}H_{30}N_8O_7S_4$, 80 %yield, yellow color, m.p.= 192-194 °C

FTIR (ν /cm^{-1}): 3356-3182(NH$_2$) 2970,2832 (C-H aliph), 3054 (Ar-H), 1764-1670(C=O groups), 1620 (C=N), 1588 (C=C aromatic), 1235(C-O)

General procedures of synthesis amid compounds [IV]-[VII]

Dissolved compound [III] (0.734 g ,0.001 mol) in ice bath and (pyridine 1 mL and DMF 2 mL) with different acid chlorides(anisole chloride, benzoyl chloride, acetyl chloride and chloroacetylchloride) was added, the mixture was stirred for 3 hrs at room temperature , then added 10% HCl the solid filtered then recrystalized by ethanol

Data of compound [IV]

Molecular formula: $C_{48}H_{42}N_8O_{11}S_4$, Yield 64 %, Color yellow, m.p.= 136_139 °C

FTIR (ν /cm^{-1}): 3200(NH),3050 (Ar-H)2969-,2818 (C-H aliph),1764-1677(C=O), 620 (C=N),1599(C=C Ar),1207 (C-O),

Data of compound [V]

$C_{46}H_{38}N_8O_9S_4$, 65 % yield, brown powder, m.p.= 90-93 °C

FTIR (ν /cm^{-1}):3158-NH, 3067 (Ar-H),2971,2816(C-H aliph.),1763-1658(C=O and C=N),1599(C=C Ar),1236(C-O), ^{1}HNMR (400 MHz, DMSO-d6) δ(ppm): 0.72-0.77(t,6H,2CH3),1.78-1.99(q,4H,2CH2), 2.89(s,2H,2OCH$_2$),3.89(s,2H,2S-CH-N),3.88(s,2H,2S-CH=),6.79-8.62(m,18H,Ar-H),8.64(s,2H,NH)

Data of compound [VI]

Molecular formula: $C_{36}H_{34}N_8O_9S_4$, Yield 62 %, Color brown, m.p. gummy

FTIR (ν /cm^{-1}): 3170 (NH), 3066(Ar-H), 2970,2800 (C-H aliph), 1752-1651(C=O andC=N),1602 (C=C Ar),1294 (C-O)

Data of compound [VII]

Molecular formula: $C_{36}H_{32}Cl_2N_8O_9S_4$, Yield 74 %, Color yellow, m.p.= 280-282 °C

FTIR (ν /cm^{-1}):3220 (NH),3072 (Ar-H),2970,2879 (C-H aliph),1764-1674 (C=O group),1637 (C=N),1598 (C=C Ar),1231 (C-O),744(C-Cl)

Results and discussion

Outlined in scheme 1,the synthesis process all compounds were prepared. The analysis spectroscopy gave satisfactory results for the proposed structures of synthesized compounds, the FTIR spectrum, mass spectroscopy and ^{1}H-NMR to confirm the synthesis process. The compound [I] was prepared by reacting of 5,5-diethylpyrimidine-2,4,6(1H,3H,5H)-triune with two moles of chloroacetylchloride in DMF and pyridine.

Using two moles of 4-hydroxybenzaldehyde and in medium of fused sodium acetate in ethanol reacted with compound [I], the aldehyde compound [II] were synthesized. Two absorption stretching bands on at 2756 cm^{-1} and the second at 1213 cm^{-1} that belong to group C-H aldehyde and C-O group respectively have been showed by

Figure 2’s mass spectrum of compound [II] revealed many peaks that were ascribed to the molecule’s fragmentation. This spectrum indicates a base peak at m/z=155, and the other peaks at m/z=360,299,185, 126,106,98,77,65 and 54. The most important distinctive fragmentation characteristic compound [II] were depicted in Scheme 2.

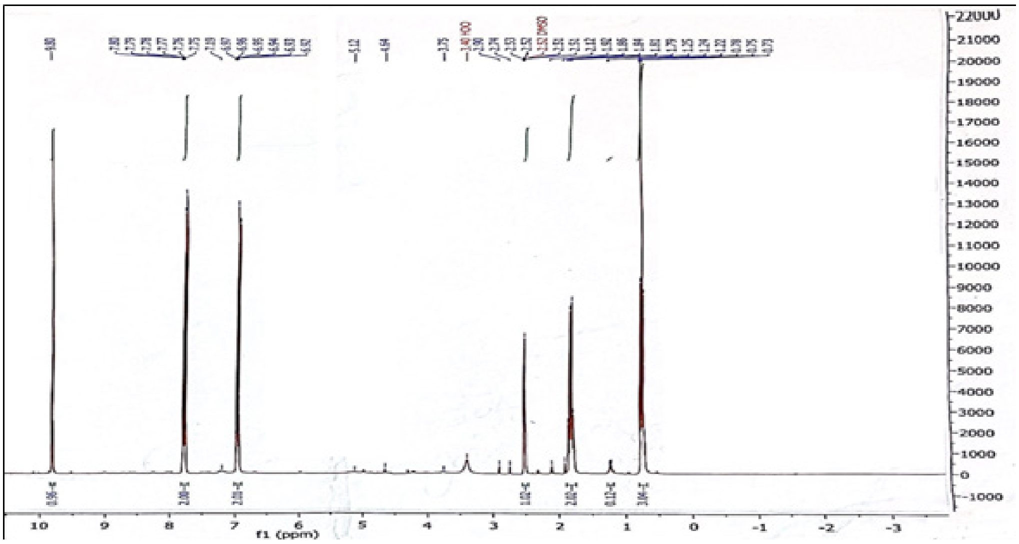


Figure 1: The^{1} H-NMR spectrum of compound [II]

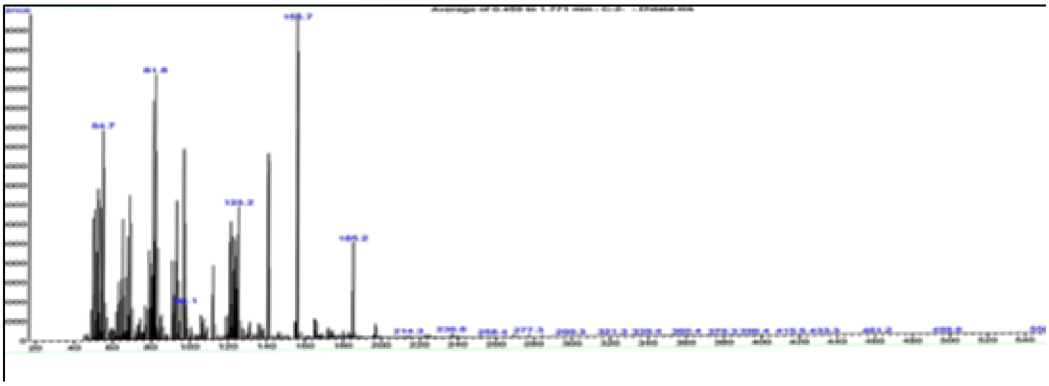
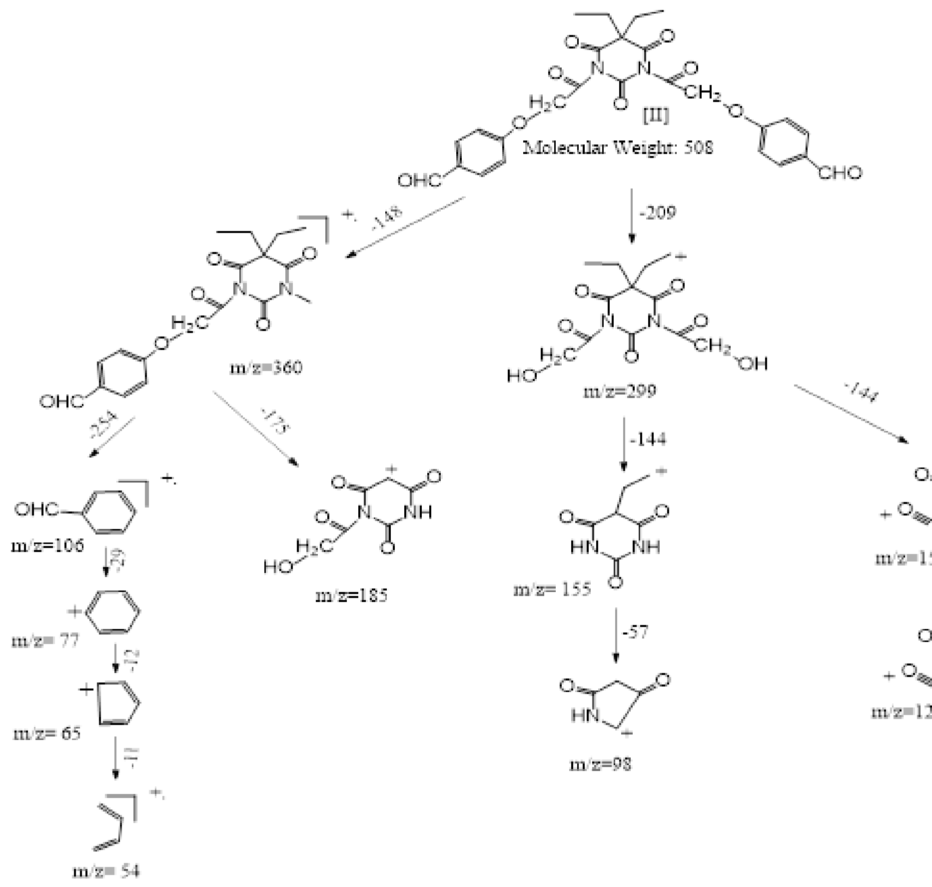


Figure 2: The Mass spectrum of compound [II]



Scheme2: Mass fragments for compound [II]

formation heterocyclic compound [III] can be formed through the process of cyclization of aldehyde derivative [II], mercaptoacetic and thiosemicarbazide followed by acidification with concentrated sulfuric acid [III]. FTIR of [III] display two n stretching bands of asymmetric and symmetric, the first stretching vibration appears at 3356 through 3182 $cm^{-1}$for NH_2 groups appears between and the second stretching band happenat 1620 cm^{-1} may be for the(C=N). Resulting compounds [IV-VII] where obtained from reaction compound [III] with different acid chlorides (anisole chloride, benzoyl chloride, acetyl chloride and chloroacetylchloride, respectively. The characteristic absorption bands in FTIR spectra showed disappeared bands of the NH_2 groups in the starting material and showed bands in the range at (1677-1651 cm^{-1}) corresponding to group C=O. For compound[V], the ^{1}HNMR spectrum in Figure 1 revealed signals at 0.72-0.77, 1.78-1.99, 2.89, 3.89, 3.88, 6.79-8.62 and 8.64 ppm for (t,6H,2CH3), (q,4H,2CH2), (s,2H,2OCH$_2$), (s,2H,2S-CH-N), (s,2H,2S-CH=), (m,18H,Ar-H) and (s,2H,NH) group, respectively .

Mesomorphic behaviors

The mesophase transitions in the synthesized compounds were investigated. The studies were carried out, in sequence, by means of using differential scanning calorimetry (DSC) and polarized light optical microscopy (POM) by which the phase transition temperatures were determined. The optical textures of the synthesized compounds' mesophases, as detected by POM, were used to the Dierking [24] and Goodbye [25] classification systems. Table 1.

Shows the Phase transition temperature of compounds [IV-VII]

Table 1. Compounds [IV-VII] phase transition temperature of

Compound No.	**Phase transition**
[IV]	$\mathrm{Cr} \overset{103}{\rightleftharpoons} \mathrm{N} \overset{220}{\rightleftharpoons} \mathrm{I}$
[V]	$\mathrm{Cr} \xrightarrow{45} \mathrm{Cr}$ $\mathrm{Cr} \xrightarrow{162} \mathrm{N} \xrightarrow{269} \mathrm{I}$
[VI]	$\mathrm{Cr} \longrightarrow \mathrm{I}$
[VII]	$\mathrm{Cr} \overset{66}{\rightleftharpoons} \mathrm{SmA} \overset{191}{\rightleftharpoons} \mathrm{N} \overset{258}{\rightleftharpoons} \mathrm{I}$

(Crystalline Phases; Cr/ smectic A; SmA /nematic phase ; N/ isotropic liquid; I)

Compound [IV] showed purely nematic phase at 129 ^{o}C as in Fig.3

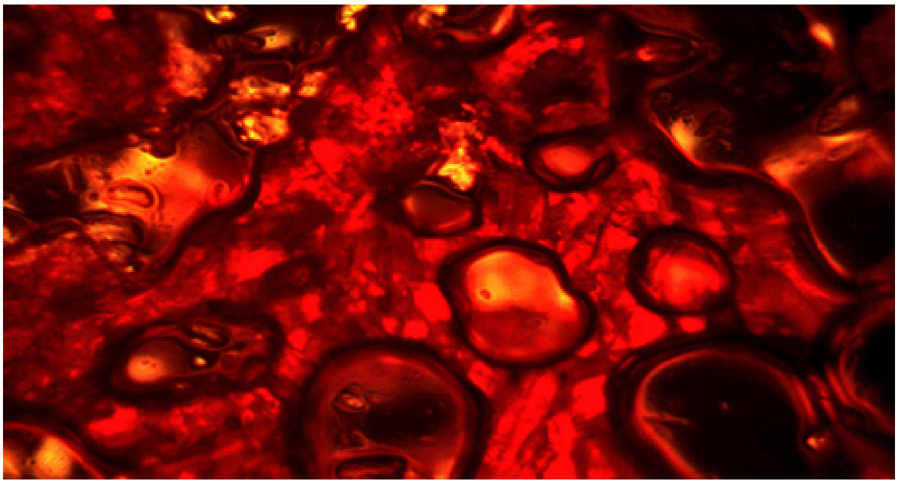


Figure 3 textures of droplets nematic phase for [IV] at 129 °C

Compound [V] display only the nematic phase as in Fig.4 on left forthe textures of droplets nematic phase and on right nematic phase for compound [V] on cooling. While the compound [VI] doesn't show liquid crystal properties .

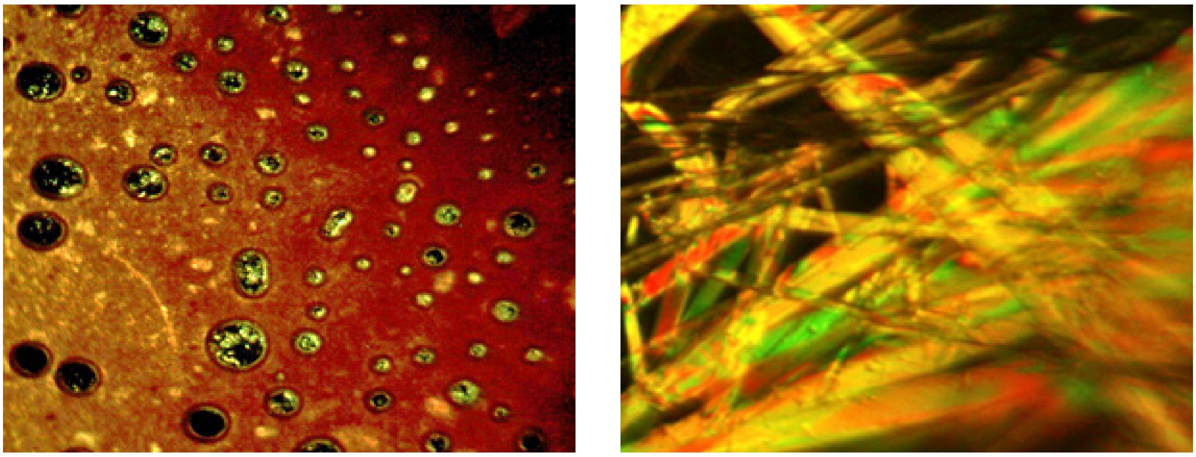


Figure4 optical textures of cross polarizing
(a) droplets nematic phase of [V] at 112 °C
(b) nematic phase of [V] at118 °C on cooling

Fig.5 shows that the compound[VII] express nematic phase and dimorphism enantiotropic smectic A (SmA) in the right and left of the figure respectively.

Exhibit [VII] smectic A (SmA) based nematic phases , in Fig. 5on left of a texture SmA and ~~on~~ the right for droplets nematic phases.

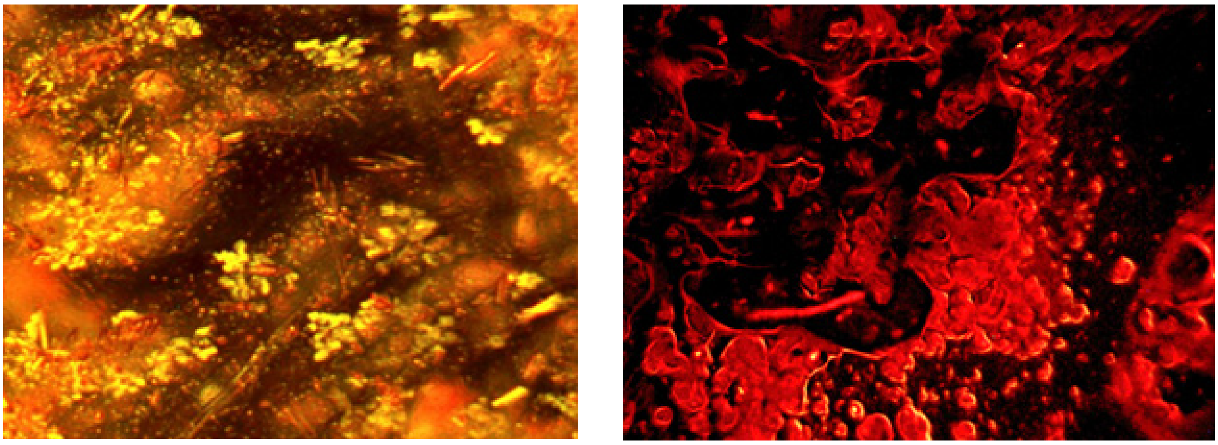


Figure5 Cross polarizing optical textures of (a)on left SmA phase for compound [VII] at 88 °C (b) on right droplets nematic phase for compound [VII] at 250 °C

Fig. 6 DSC of [V]

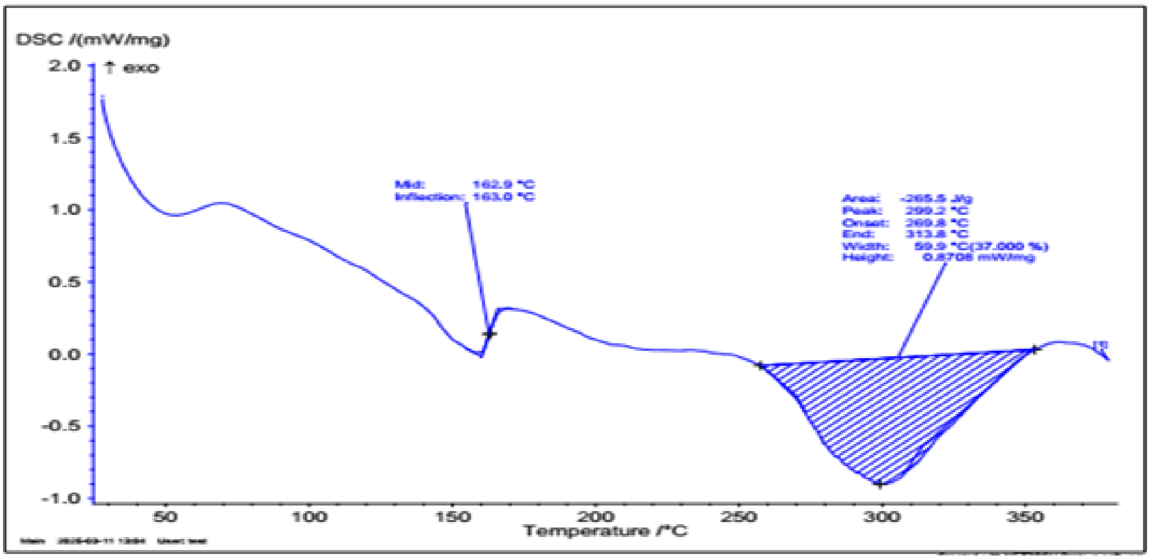


Figure 6: DSC thermogram for compound [V]

Except for end groups on both sides of the core, compounds [IV] through [VII] have an identical chemical skeleton of pyrimidine trione core. Appearance liquid crystalline properties for compounds [IV] and [V] may be explained by the presence of phenyl groups in these compounds and causing increase in the terminal interaction as well as the core's polarizability and stiffness the alkoxy group terminal length which is causes the terminal/lateral interaction force ratio is the main cause te explain the occurrence of the methoxy group in [IV] compound, which interferes with ordering of liquid crystalline ordering. With percentage increased , showed mesophases that are less ordered (nematic mesophase) [26]. The compound [VI] with end alkyl group (CH_3) do not reveal any liquid crystalline behavior this behavior is consistent with what has been reported in the literature for this form of mesophase [12]. Methyl groups influence liquid crystal formation by impacting the molecular interactions and shape anisotropy of the mesogenic units, ultimately affecting the type and stability of the liquid crystal phase. They can either stabilize or destabilize the mesophase depending on their position and the overall molecular structure. The shape and flexibility of a molecule play a crucial role in its ability to self-organize into a mesophase Methyl groups in liquid crystal (LC) molecules can significantly influence their mesomorphic properties, by disrupting the molecular order necessary for liquid crystal formation and may not display liquid crystalline behavior.

The compound [VII] showed SmA and nematic phases this behavior my by attributed effect of the chloro end-group on liquid crystal phases and this agreement with another study [27]

According data of phase transition temperature of the bent-core LCs compounds [IV],[V] and[VII] showed the transition temperatures and stability of nematic phase of compounds [IV],[V] is more stable than compound[VII], this behavior may be attributed to the presence of phenyl ring in compounds [IV],[V] and absence in compound [VII]

In addition to when compared these compounds [IV],[V] and[VII] with compound in another study [17] which containing same center core, found all compounds in this work showed liquid crystal phases with transition temperatures and stability more than compound of study [17] this behavior may be attributed to the presence thiazolo [4,3-b][1,3,4]thiadiazol unit with amide as a terminal group.

Conclusions

The mesomorphic and synthesis behavior of new bent compounds which contain 5-diethylpyrimidine 2,4,6(1H,3H,5H)-trione as a core unit and comprising different substituent end groups have been described. Using differential scanning calorimetry(DSC)and Polarized optical microscope (POM) ~~used~~ to examine the mesomorphic behavior. The compounds [IV]and [V] showed complete nematic state, at tha same time [VI] compound does not have any LC behavior. On the other hand [VII]compound shows nematic phases and enantiotropic smectic A (SmA) dimorphism. The molecule geometrical shape can effecting the mesogens properties (phase transition, type of mesophase, temperature…. etc.). Geometric molecule, influencing type of mesophase, transition temperatures, and other properties of mesogens. This indicates t acutance LC feature synthesized compound.

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