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Mesomorphism dependence on heterocyclic tail

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ABSTRACT

A novel homologous series of liquid crystal materials containing a heterocyclic ring was synthesized with a view to understanding and establishing the relation between liquid crystal (LC) properties and a molecular structure; and with a view to curing skin-related diseases following biological activity studies. The series consist of 13 (C₁ to C₁₈) members. All the members are liquid crystals. The C₄ to C₁₈ members are smectogenic of which C₄ and C₅ are monotropic and remaining members (C₆ to C₁₈) are enantiotropic in nature; whereas all C₁ to C₁₈ members are only nematogenic. Hence the C₁, C₂, C₃ members are only nematogenic. Transition temperatures were determined by an polarizing optical microscope equipped with heating stage. Analytical and spectral data confirmed the molecular structures of homologues. It is a middle ordered melting type series. LC properties of a series are compared with the structurally known series.

KEY WORDS

Liquid crystals; Mesogen; Mesomorphism; Nematic; Smectic

Introduction

Thermal and lyotropic liquid crystalline states of matter have proved their ability to benefit mankind since their discovery in 1888[1]. All scientific and technological researchers have contributed to the LC state with different aims and objectives [2,3,4,5,6,7,8,9,10,11]. Novel substances are required by a wide variety of researchers. Therefore present investigation is aimed to synthesize novel thermotropic LC substances through a homologous series and to establish the effect of molecular structure on liquid crystal properties and the degree of liquid crystallinity [12,13,14]. Many homologous series and novel LC substances have been reported to date [15,16,17,18,19,20,21]. The novel homologous series consists of two phenyl rings and one heterocyclic ring bonded through –CH–CH–COO– and –CO–CH=CH– central bridges with changing n-alkoxy terminal end group. LC properties of series will be evaluated and comparative study with structurally similar homologous series will be carried out in terms of molecular rigidity and flexibility [22,23,24,25]. Novel LC compounds may be biologically active against skin diseases.

Experimental

Synthesis

4-*n*-Alkoxybenzaldehydes were synthesized by refluxing 4-hydroxybenzaldehyde (1 equiv.) with corresponding n-alkyl bromides (1 equiv.) in the presence of potassium carbonate

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(1 equiv.) and acetone as a solvent [26]. The resulting 4-*n*-alkoxybenzaldehydes were reacted with malonic acid (1.2 equiv.) in the presence of 1–2 drops piperidine as catalyst and pyridine was used as the solvent to yield corresponding trans 4-*n*-alkoxy cinnamic acids (A) [27]. 3-(2-Thiophenyl)-1-(4-hydroxyphenyl) 2-propen-1-one (B) was prepared by an established method [28] M.P.170–172°C, Yield- 64.8%. Coupling of compounds A and B is done by Steglich esterifications to yield α -4(4'-n-alkoxy cinnamoyloxy) benzoyl- β -(2-thiophenyl) ethylene [29].

The synthetic route to the novel homologous series of ethylene derivatives is shown in scheme 1.

Characterization

Some of members of the novel series were characterized by elemental analysis (Table 1), Infrared spectroscopy, ¹H NMR spectra and mass spectroscopy. Microanalysis was performed on Euro EA Elemental Analyzer. IR spectra were recorded on Shimadzu FTIR Model IRAffinity-1S (MIRacle 10), ¹HNMR spectra were recorded on Bruker spectrometer using CDCl₃ as a solvent and mass spectra were recorded on Shimadzu GC-MS Model No.QP-2010. The liquid crystal behavior and the type of textures were determined by miscibility method on microscopic observations.

		Elements %Found		Elements %Calculated			
Sr. No.	Molecular formula	С	Н	0	С	Н	0
1	C ₂₄ H ₂₀ O ₄ S	71.21	5.01	15.85	71.27	4.98	15.82
2	$C_{28}^{-1}H_{28}^{-0}O_{4}^{-1}S$	73.09	6.08	13.85	73.02	6.13	13.89
3	C ₃₆ H ₄₄ O ₄ S	75.55	7.71	11.12	75.49	7.74	11.17

Table 1. Elemental analysis for (1) Ethyloxy (2) Hexyloxy (3) Tetradecyloxy derivatives.

Analytical data

Spectral data

¹HNMR in ppm for butyloxy derivative

0.89–0.93 (t,3H,–CH₃ of $-OC_4H_9$ group), 1.38–1.53 (m,2H,CH₃–<u>CH₂–</u>), 1.71–1.88 (p,2H,–<u>CH₂–</sub>CH₂–O–), 3.91–3.94 (t,2H,–<u>CH₂–O–</u>), 7.75–7.79 (d,1H,–<u>CH</u>=CH–COO–), 6.39–6.43 (d,1H,–CH=C<u>H</u>–COO–), 7.45–7.47 (d,1H,–CO–C<u>H</u>=CH–), 7.86–7.90 (d,1H,–CO–CH=C<u>H</u>–), 7.45–7.98 (3H, thiophene ring), 7.45–7.47 & 7.98–8.01 (4H, middle phenyl ring), 6.84–6.86 & 7.34–7.36 (4H, phenyl ring with alkoxy chain). The NMR data are consistent with the molecular structure.</u>

¹HNMR in ppm for octyloxy derivative

0.79–0.83(t,3H,-CH₃ of $-OC_8H_{17}$ group), 1.21–1.27 (m,8H,CH₃–<u>CH₂-CH₂-CH₂-CH₂–<u>CH₂-</u>, 1.35–1.42 (p,2H,-<u>CH₂-CH₂-CH₂-CH₂-O-), 1.69–1.76 (p,2H,-<u>CH₂-CH₂-O-), 3.90–3.94</u> (t,2H,-CH₂-O-), 7.36–7.30 (d,1H, -C<u>H</u>=CH-COO-), 6.40–6.44 (d,1H, -CH=C<u>H</u>-COO-), 7.01–7.03 (d,1H, -CO-C<u>H</u>=CH-), 7.18–7.25 (d,1H, -CO-CH=C<u>H</u>-), 7.75–8.01 (3H, thiophene ring), 7.27–7.36&7.99–8.01 (4H, middle phenyl ring), 6.84–6.86 & 7.75–7.79 (4H, phenyl ring with alkoxy chain). The NMR data are consistent with the molecular structure.</u></u>

IR in cm⁻¹ for propyloxy derivative

3072(C–H str. of alkene disubstituted), 2933 & 2872 (C–H str. of (–CH₂–)_n group of –OC₃H₇), 1720 (C=O str. of carbonyl carbon of ester group), 1653 (C=O str. of α , β unsaturated ketone), 1653 & 1629 (C=C str. of alkene), 1593,1573 & 1512 (C=C str. of aromatic ring), 1332& 1286 (C–H bending of alkene disubstituted), 1251 (C–O str. of ether linkage), 1128 (C–O str. of ester group), 995,968 & 875 (C–H bending of alkene). The IR data are consistent with the molecular structure.

IR in cm⁻¹ for heptyloxy derivative

3072 (C–H str. of alkene disubstituted), 2956 & 2854 (C–H str. of $(-CH_2-)_n$ group of – OC_7H_{15}), 1720 (C=O str. of carbonyl carbon of ester group), 1654 (C=O str. of α,β unsaturated ketone), 1631 (C=C str. of alkene), 1593,1573 &1506 (C=C str. of aromatic ring), 1332, 1303 & 1288 (C–H bending of alkene disubstituted), 1253 (C–O str. of ether linkage), 1130 (C–O str. of ester group), 964,873& 817 (C–H bending of alkene). The IR data are consistent with the molecular structure.

Mass spectra of pentyloxy derivative

m/*z* (rel.int%): 446 (M)⁺ ,217,201,171,147,119,91,65

Mass spectra of dodecyloxy derivative

m/*z* (rel.int%): 544(M)⁺ ,315,230,201,164,147,119,91,55

Results and discussion

Trans 4-*n*-alkoxy cinnamic acid, on linking with 4-hydroxy chalconyl derivative with thiophene heterocyclic ring yielded mesomorphic homologues. Smectic mesophase commences

		Transition temperatures in ^o C		
Compound No.	R=n-alkyl chain C_nH_{2n+1}	Sm	Ν	lsotropic
1	1	_	151.6	158.2
2	2	-	145.2	167.8
3	3	-	161.9	166.3
4	4	(145.2)	160.4	165.8
5	5	(142.3)	147.6	157.4
6	6	135.8	140.9	153.0
7	7	132.7	135.2	153.4
8	8	126.2	137.6	152.2
9	10	124.4	136.8	148.1
10	12	122.0	132.3	146.8
11	14	121.2	130.0	144.3
12	16	122.9	127.5	140.6
13	18	119.7	122.6	132.3

Table 2. Transition temperatures in °C.

Sm, Smectic; N, Nematic; () indicate monotropy

from C_4 homologue, whereas nematic mesophase commences from very first member of a series. Thus, mesomorphism is exhibited by all the members of the present novel series of ethylene derivatives. Transition temperatures (Table 2) as determined by an optical polarizing microscope equipped with a heating stage were plotted versus the number of carbon atoms present in n-alkyl chain of left flexible unit -OR, and then like or related points were linked to draw the Cr-M, N-I, Sm-N transition curves showing phase behaviors of novel series in a phase diagram (Fig. 1). Analytical and spectral data confirmed the structures of novel molecules. Cr-M transition curve adopted zigzag path of rising and falling tendency from C_1 to C_4 homologues and then zigzag path extended in negligible proportion with overall descending tendency as series is ascended up to C_{18} homologue. Sm-N transition curve for odd and even member rises and then passing through maxima at the C_8 homologue and descends up to last homologue. Odd–even effects is observed for the curve up to C_9 homologue and then, descended as a single curve up to C_{18} member of a series. N-I transition curves



Figure 1. Phase Behavior of Series.

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for odd and even homologues of a series merge into each other at the C7 homologue and then, descended as a single curve up to C_{18} member of a series. Hence N-I transition curve exhibited odd-even effect. Thus, Cr-M, Sm-N and N-I transition curves behave in normal manner, with overall descending tendency of transition temperatures. Thermal stability for smectic and nematic are 132.86°C and 152.78°C, respectively; whose total (Sm+N) mesomorphic phase length varies from 4.4 °C to 26.0 °C. The present series is partly smectogenic and fully nematogenic whose transition or melting temperatures vary between 119.7°C and 167.8°C. LC properties vary from homologue to homologue in the same series with alternation of transition temperatures. As series is ascended from homologue to homologue in the same series, by progressive sequential addition of methylene unit, the molecular lengths, length to breath ratio, molecular flexibility, intermolecular end to end and lateral attractions, permanent dipole moment across the long molecular axis, dipole-dipole and electron-electron interactions, dispersion forces; molecular polarity and polarizibility etc. increases or alters as a consequence of changing molecular flexibility keeping molecular rigidity unchanged. The above alterations from homologue to homologue undergo in present novel series in such a manner that, the magnitudes of anisotropic forces become suitable or fittest to cause either only nematic mesophase formation or nematic mesophase formation in addition to smectic mesophase formation which become possible and favorable. Thus, entire series from C_1 to C₁₈ homologues exhibited mesomorphic property with different degree of mesomorphism and different magnitudes of same property like transition temperature. The exhibition of oddeven effect and alternation of transition temperatures are attributed to the addition of methylene unit or units in sequential manner at the *n*-alkyl chain bonded to phenyl ring through oxygen atom. The disappearance of odd-even effect after merging of curves for odd and even homologues is attributed to the coiling or flexing or bending or coupling of longer *n*-alkyl chain with the principal axis of the core structure. The molecules of homologues are disaligned at an angle ninety or less than ninety degree on the plane of floating surface and resist the exposed thermal vibrations accordingly for definite range of temperature, depending upon the magnitudes of thermal resistivity offered by individual homologue due to their individual combine effect of molecular rigidity and flexibility i.e. energy stored in a molecule in terms of enthalpy (Δ H) to facilitate and stabilize nematic or/and smectic mesophase formation for definite phase length range. Thus, present series is partly smectogenic and fully nematogenic with middle ordered melting type.

Some mesomorphic properties of presently investigated novel mesogenic series-1, are compared with the structurally similar other known series-X [30] and Y [30] as mentioned in Fig. 2.

Homologous series-1 of present investigation and the homologous series-X and Y selected for comparative study are identical with respect to two phenyl rings, two central bridges



Figure 2. Structurally similar series.

Series \rightarrow	1	Х	Y
Smectic-Isotropic or Smectic-Nematic	132.86 (C ₆ -C ₁₈)	132.25 (C ₅ -C ₁₀)	$\begin{array}{c} 159.7 \ (C_5 - C_8) \\ C_5 \\ 174.1 \ (C_5 - C_{14}) \\ C_5 \\ 18.0 \ (C_{10}) \ to \ 45.0 \ (C_6) \end{array}$
Commencement of smectic mesophase	C ₄	C ₅	
Nematic-Isotropic	152.78 (C ₁ -C ₁₈)	171.5 (C ₅ -C ₁₆)	
Commencement of nematic phase	C ₁	C ₅	
Total Mesophase length range (Sm+N)	4.4(C ₃) to 26.0(C ₈)	34.0(C ₅) to 76.0 (C ₆)	

Table 3. Average thermal stabilities in °C.

viz. -CH=CH-COO- and -CO-CH=CH- or -CH=CH-CO- as well as left n-alkoxy terminal end group for the same homologue from series to series, but their tail groups

•NO₂ and OCH₃ are different. Therefore, variations in viz. mesomorphic properties and the degree of mesomorphism are varied with the changing, combined effects of molecular rigidity and flexibility as emerged from changing features of series-1, X and Y respectively. Following Table 3 represents some mesomorphic properties of series-1, X and Y under comparison.

Table 3 indicates that,

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- Homologous series-1, X and Y are smectogenic in addition to nematogenic in character.
- Smectic and nematic properties of series-1 commences earlier from C₄ and C₁ homologues respectively; whereas smectic and nematic properties for both homologues series-X and Y commences equally from C₅ homologue.
- Smectic thermal stability of series-1 is (132.86°C) same as series-X (132.25°C) and the lower than a series-Y (159.7°C).
- Nematic thermal stability of series-1 (152.78°C) is lowest among the series under comparison and the same is highest (174.1°C) for series-Y and that of series-X is intermittent (171.5°C) to series-1 and Y.
- Odd-even effects are observed for Sm-N and N-I transition curves for all the series under comparison.
- Mesophase length ranges are in the decreasing order as series-X > series-Y > series-1.

The suitable magnitudes of anisotropic forces of intermolecular end to end and/or lateral attractions as a consequences of favorable molecular rigidity and flexibility as occurred from molecular structure which induces smectic or/and nematic mesophase or mesophases. -NO₂ and -OCH₃ groups bonded to phenyl ring are highly nematogenic polar and polarizable groups and their end to end attractions are relatively more than a thiophene unit of series-1. Therefore, disallignment of molecules on the plane of floating surface for the same homologue from series to series and from homologue to homologue in the same series differs under the influence of exposed thermal vibrations. Thus, resistivity towards exposed thermal vibrations occurs in different degree of temperatures to maintain either statistically parallel orientational order of molecules or/and focal conic layered or sliding layered arrangement of molecules. The combine effect of end group polarity and polarizibility in combination with molecular polarity and polarizibility operates mesomorphic tendency. The -OCH₃ group is penta atomic and -NO₂ group is triatomic generates polarizibility for the same homologue in varying proportions which reflects in intermolecular end to end and lateral attractions. Therefore, nematic thermal stability of series-X and Y are nearly equivalent (171.5°C and 174.1°C) but exceeds by 15°C to 20°C units to the nematic thermal stability of series-1. However smectic thermal stability of series-1 and X (132.86°C and 132.25°C) is equivalent, but it is about 28°C less than a series-Y (159.7°C). The smectic and nematic mesophases of series-1 commences (C_4 and C_1) earlier than the series-X and Y (from C_5) because, the extent of

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molecular noncoplanarity vary from series to series for the same homologue due to differing magnitudes of molecular flexibility. Almost equipolar and equipolarizable $-C_6H_4.NO_2$ and $-C_6H_4.OCH_3$ units of series-X and Y undergo almost equal degree of molecular noncoplanarity. Hence, smectic and nematic mesophases commences equally from same C_5 member of the both series. None of the functional group like $-NO_2$ or $-OCH_3$ is attached with thiophene ring of a series-1, which reduced the molecular flexibility and the extent of molecular noncoplanarity to cause early commencement of smectic mesophase formation earlier by one homologue and the commencement of nematic mesophase formation earlier by four homologues. The energy stored in a molecule (Δ H) varies from homologue to homologue in the same series and for the same homologue from series to series whose resistivity against externally exposed thermal vibrations differs in proportionate magnitudes. Hence, the transition temperatures, degree of mesomorphism and the extent to which type of mesophase facilitated or hindered is dependent.

Conclusion

- A novel ester homologous series of thirteen members (C_1 to C_{18}) are synthesized to understand the effect of heterocyclic tail on mesomorphism. Series is partly smectogenic and fully nematogenic as derived from trans *n*-alkoxy cinnamic acids. Its degree of mesomorphism is relatively low and it is middle ordered melting type.
- The group efficiency order derived for smectic and nematic on the basis of (i) thermal stability (ii) commencement of mesophase and (iii) the total mesophase length are as under.

C

(i) Smectic

$$-C_6H_4OCH_3 > \text{NO}_2$$
 = $-C_6H_4.NO_2$

Nematic

$$-C_6H_4OCH_3 > -C_6H_4.NO_2 > \text{N}$$

(ii) Smectic

$$\text{S} > -C_6H_4OCH_3 = -C_6H_4.NO_2$$

Nematic

$$\text{ C} > -C_6H_4OCH_3 = -C_6H_4.NO_2$$

(iii) Smectic+ Nematic

$$-C_6H_4.NO_2 > -C_6H_4OCH_3 > \text{Image}$$

• Liquid crystal properties and the degree of mesomorphism are very sensitive and susceptible to molecular structure as a consequence of molecular rigidity and flexibility.

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- Present LC materials may be useful for the study of binary system, for the LC devices workable between 100°C and 147°C.
- LC material of present investigation may be useful for Dermatological treatment, if studied properly by researchers working in medical or pharmaceutical fields.

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