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Mesogenic cinnamates with a substituted ethyl terminal chain*

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Two new mesogenic homologous series of liquid crystalline cinnamates with substituted ethyl tails, β -methoxyethyl [4-(4'-*n*-alkoxycinnamoyloxy)benzoates (**I**) and β -chloroethyl [4-(4'-*n*-alkoxycinnamoyloxy)benzoates (**II**), have been synthesized and characterized by a combination of elemental analysis and standard spectroscopic methods. In series **I**, lower-chain members exhibit nematic mesophase, middle members exhibit enantiotropic nematic as well as smectic A (SmA) mesophases, whereas higher members exhibit only an enantiotropic SmA mesophase. In series **II**, methoxy to *n*-butyloxy derivatives exhibit a monotropic nematic mesophase. The SmA mesophase commences from *n*-propyloxy derivative as monotropic and persists up to the last member synthesized. The mesomorphic properties of present series were compared with each other and with a structurally related mesogenic homologous series to evaluate the effects of substituted ethyl tail and cinnamoyloxy central linkage on mesomorphism.

1. Introduction

Terminal groups present in a molecule have their own importance because of their polarity. It has been found that terminally substituted compounds exhibit more stable mesophases than do non-substituted mesogenic compounds. Any terminal group, which increases the molecular polarizability, without significantly increasing the molecular breadth, increases the thermal stability of the resulting mesophase.

A number of homologous series of mesogenic esters with normal and branched terminal alkyl chains have been reported previously [1–3]. Cinnamates esters have been well known for over 30 years and extensive literature exists [1–14]. Vora and Rajput [15] reported that binary mixtures of cinnamate esters exhibit a wide range of smectic and nematic mesophases. Sadashiva *et al.* [16] reported the synthesis and mesomorphic properties of some esters of *trans*-4-*n*-alkoxycinnamic acid and *trans*-4-*n*-alkoxy- α -methylcinnamic acid with branched-chain alkyl tails exhibiting ferroelectric and antiferroelectric phases. Our literature survey indicated that, compared with mesogenic esters with normal and achiral-branched terminal alkyl chains, liquid crystalline esters having

chains containing differing kinds of atoms are very rare. To distinguish these from branched alkyl groups, we will refer to them as broken terminal alkyl chains. Weygand *et al.* [17] have studied the alkyl chain $\text{CH}_3\text{OCH}_2\text{O}-$, which combines two ether functions as a terminal substituent. Rather few compounds have been examined, but the data shows that mesomorphic properties disappear entirely or compounds that have lower nematic thermal stabilities than the analogous compounds containing the $\text{CH}_3\text{CH}_2\text{CH}_2\text{O}-$ group. Liquid crystalline dimers comprising cholesteryl esterimide groups linked via oligo(ethylene oxide) chains, as well as homologous mesogenic series of cholesteryl esterimides with oxyethylene and methylene tails, have also been reported [18, 19]. Chiang *et al.* [20, 21] studied the effects of ethoxyethoxy (EO) and butoxyethoxyethoxy (BO) chains on mesomorphism. They observed that the BO-containing liquid crystals have greater SmC* phase stability than the corresponding EO-containing materials. Recently, Wu and Lai [22] reported the synthesis and ferroelectric properties of new chiral liquid crystals derived from (*S*)-lactic acid with alkoxyethanols. We have reported methoxyethyl and ethoxyethyl esters of 4-(4'-*n*-alkoxybenzoyloxy)benzoic acids [23a] as well as methoxyethyl *trans*-4-(4'-*n*-alkoxybenzoyloxy)- α -methylcinnamates [23b]. All three homologous mesogenic series exhibited smectic A (SmA) mesophases at ambient temperatures and the binary mixtures of such compounds [23c] exhibited room temperature smectogenic properties

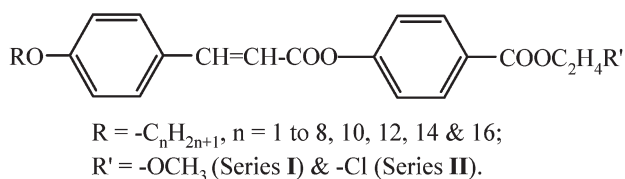
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with a wide range of temperatures. Recently we reported [24] a homologous series containing an ethoxyethyl chain and based on three phenyl rings and ester and azomethine central linkages, which exhibited nematic and/or smectic mesophases.

In order to further study the effect of broken alkyl terminal chains, four mesogenic homologous series of azoesters having methoxyethyl and ethoxyethyl terminal chain have been synthesized [25, 26]. A literature survey also reveals that mesogenic homologous series with chloroethyl tail are very few. Vora and Patel [27] have reported the mesogenic homologous series with chloroethyl tail. We have also reported [28] two mesogenic homologous series of Schiff's base ester with chloroethyl tail, as well as the dielectric relaxation and anisotropic studies of such smectogen [29].

In continuation of our work on substituted ethyl tails, we have synthesized two new mesogenic homologous series of cinnamates with substituted ethyl tails having the general structural formula shown in scheme 1, and



Scheme 1. General formula of cinnamates with substituted ethyl tails.

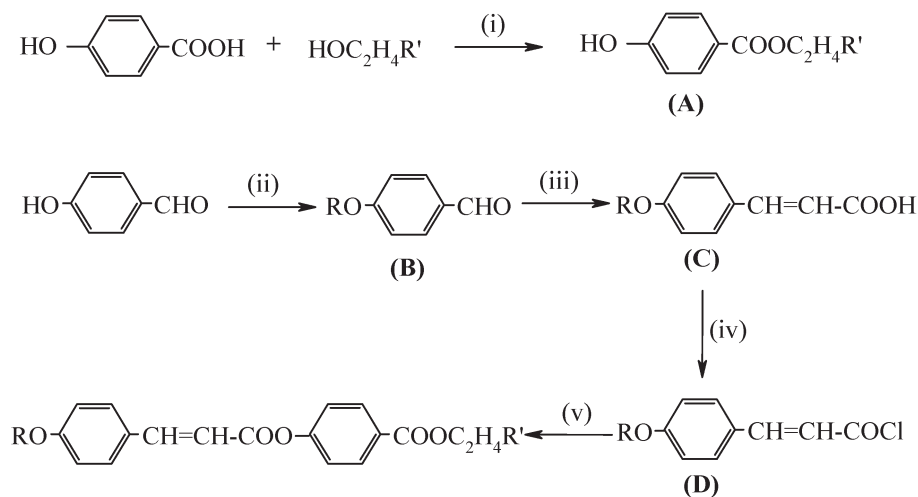
have evaluated the effect of cinnamoyl central linkage and substituted ethyl tail on mesomorphism.

2. Experimental

2.1. Synthesis

4-Hydroxybenzoic acid, 4-hydroxybenzaldehyde, malonic acid, piperidine, concentrated H_2SO_4 , $SOCl_2$, chloroethanol, methoxyethanol, the appropriate *n*-alkyl halides, and anhydrous K_2CO_3 were used as received. Solvents were dried and distilled before use. Microanalysis of the compounds was performed on a Coleman carbon-hydrogen analyser, and the values obtained are in close agreement with those calculated. IR spectra were determined for KBr pellets, using a Shimadzu IR-408 spectrophotometer. 1H NMR spectra were obtained with a Perkin-Elmer R-32 spectrometer using tetramethylsilane (TMS) as internal reference standard. The chemical shifts are quoted as δ (ppm) downfield from the reference; $CDCl_3$ was used as solvent for all the compounds. The phase assignments and transition temperatures were determined by thermal polarizing optical microscopy using a polarizing microscope (Leitz Laborlux 12 POL) equipped with a heating stage. The enthalpies of transitions, reported in $J g^{-1}$, were measured on a Mettler TA-4000 system, at a scanning rate of $5^\circ C min^{-1}$. The instrument was calibrated using pure indium as a standard.

The synthetic route to compounds of series I and II is illustrated in scheme 2.



Reagents and conditions: (i) Conc. H_2SO_4 (ii) $R'Br$, Anhyd. K_2CO_3 , Dry acetone (iii) Malonic acid, Pyridine, Piperidine (iv) Excess $SOCl_2$ (v) **A** in dry pyridine, Cold aq. HCl

Scheme 2. Synthetic route to series I and II compounds.

β -Methoxyethyl 4-hydroxybenzoate and β -chloroethyl 4-hydroxybenzoate (**A**) were synthesized by the esterification of 4-hydroxybenzoic acid with β -methoxyethanol and β -chloroethanol, respectively, as described previously [23]. 4-*n*-Alkoxybenzaldehydes (**B**) were prepared using the method of Vyas and Shah [30]. *trans*-4-*n*-Alkoxybenzaldehydes (**C**) were synthesized from the corresponding 4-*n*-alkoxybenzaldehydes and malonic acid following the method reported by Gray and Jones [31]. *trans*-4-*n*-Alkoxybenzoyl chlorides (**D**) were prepared by treating corresponding acids with excess of thionyl chloride under reflux for 2 h [25].

2.2. Synthesis of series I and series II compounds

Dry β -methoxyethyl (for series I) or β -chloroethyl (for series II) 4-hydroxybenzoate (15 mmol) was dissolved in dry pyridine (10 ml) and a cold solution of an appropriate *trans*-4-*n*-alkoxybenzoyl chloride (15 mmol) in dry pyridine (10 ml) was added slowly while stirring in an ice bath. The mixture was allowed to stand overnight at room temperature before being acidified with cold 1:1 aqueous hydrochloric acid. The product obtained was extracted by ether. The ethereal extract was washed successively with water (2 \times 30 ml),

saturated solution of sodium bicarbonate (2 \times 50 ml) and water (2 \times 30 ml) before being dried over Na₂SO₄. The residue obtained on removal of solvent was separated by chromatography on silica gel using petroleum ether (60–80°C)/ethyl acetate (95/5) as eluent. Removal of solvent from the eluate afforded a solid material, which was crystallized repeatedly from methanol. The purities of the synthesized compounds were checked by thin-layer chromatography (Merck kieselgel 60F₂₅₄ pre-coated plates). The melting points and transition temperatures are recorded in table 1.

The elemental analyses of all the compounds of both series were found to be satisfactory. Typical spectral data of a representative member, the *n*-tetradecyloxy derivative, of both series I and series II are as follows.

Series I: IR ($\nu_{\max}/\text{cm}^{-1}$): 3075, 2917, 2851, 1738 (–COO–), 1716, 1630 (–CH=CH–) 1604, 1512, 1434, 1281, 1112, 824. ¹H NMR (300 MHz): δ 0.88 (t, 3H, CH₃), 1.20–1.40 (m, 22H, 11 \times CH₂), 1.80 (quant., 2H, Ar–O–C–CH₂), 3.8–3.95 (m, 7H, 5H of CH₂OCH₃ and 2H of Ar–O–CH₂), 4.05 (t, 2H, COOCH₂), 6.45 (d, 1H, Ar–CH=), 6.93 (d, 2H, ArH), 7.26 (d, 2H of ArH), 7.55 (d, 2H, ArH), 7.85 (d, 1H, =CH–COO), 8.08 (d, 2H, ArH). Series II: IR ($\nu_{\max}/\text{cm}^{-1}$): 3030, 2918, 2850, 1722 (–COO–), 1631 (–HC=CH–), 1602, 1511, 1471, 1258,

Table 1. Transition temperatures (°C) of the series I and II compounds.

Compound	<i>n</i>	Cr		SmA		N		I
Series I								
1	1	•	155	–		(•	147)	•
2	2	•	145	–		•	152	•
3	3	•	106	–		•	142	•
4	4	•	95	•	101	•	140	•
5	5	•	97	•	116	•	138	•
6	6	•	95	•	124	•	137	•
7	7	•	95	•	127	•	134	•
8	8	•	84	•	124	•	128	•
9	10	•	85	•	130	–		•
10	12	•	94	•	133	–		•
11	14	•	95	•	134	–		•
12	16	•	94	•	126	–		•
Series II								
13	1	•	102	–		(•	87)	•
14	2	•	118	–		(•	103)	•
15	3	•	107	(•	100)	(•	101)	•
16	4	•	114	(•	107)	(•	113)	•
17	5	•	104	•	111	–		•
18	6	•	101	•	113	–		•
19	7	•	90	•	114	–		•
20	8	•	82	•	119	–		•
21	10	•	83	•	122	–		•
22	12	•	78	•	125	–		•
23	14	•	91	•	126	–		•
24	16	•	86	•	122	–		•

(•)=monotropic value; Cr=crystalline solid; SmA=smectic A phase; N=nematic phase; I=isotropic liquid phase; •, phase exists; –, phase does not exist.

1111, 831, 687 (–Cl), 564. ^1H NMR (300 MHz): δ 0.88 (t, 3H, CH_3), 1.20–1.40 (m, 22H, $11 \times \text{CH}_2$), 1.81 (quant., 2H, Ar–O–C– CH_2), 3.83 (t, 2H, CH_2Cl), 4.00 (t, 2H, Ar–O– CH_2), 4.58 (t, 2H, COOCH_2), 6.48 (d, 1H, Ar–CH=), 6.92 (d, 2H, ArH), 7.26 (d, 2H, ArH), 7.52 (d, 2H, ArH), 7.82 (d, 1H, =CH–COO), 8.11 (d, 2H, ArH).

3. Results and discussion

The liquid crystalline phases were observed using a polarizing optical microscope. Thin-film samples were obtained by sandwiching them between a glass slide and cover slip. All the compounds of series **I** and series **II** exhibited mesomorphism. On cooling, isotropic liquids of series **I** compounds with $n \leq 8$ formed small droplets that coalesced to the classical schlieren texture of the nematic phase (figure 1 a) and *n*-butyloxy to *n*-octyloxy derivatives, on further cooling, exhibited focal-conic texture characteristic of a SmA mesophase (figure 1 b). On cooling, isotropic liquids of the higher homologues of series **I** ($n=10$ –16) gave only focal-conic texture characteristic of SmA mesophase. For series **II**, on cooling the isotropic liquid, lower members ($n \leq 4$) exhibit schlieren texture of the nematic phase and *n*-propyloxy and *n*-butyloxy derivatives, on further cooling, exhibited focal-conic texture characteristic of a SmA mesophase (figure 1 c). Middle and higher homologues ($n=5$ –16) of series **II** exhibited only focal-conic texture characteristic of SmA when cooled from the isotropic liquid. The SmA phase shows a focal-conic fan and isotropic textures under homogeneous (figures 1 b

and 1 c) and homeotropic (figure 1 d) alignments under shearing, respectively. There was no fundamental difference in the textures through both series of derivatives.

In the present study, the enthalpies of transition of a few derivatives in series **I** and **II** were measured by differential scanning calorimetry (DSC); the results are recorded in table 2.

All twelve compounds synthesized in series **I** exhibit mesomorphism. When the terminal attractions are more pronounced, the nematic mesophase prevails. In this series, the first three members show a purely nematic mesophase. The overall polarizability is enhanced with the increase of alkyl chain length as the series is ascended, i.e. as n increases. The ratio of terminal attractions to lateral attractions decreases as the series is ascended; the net result is that the lateral attractions gain predominance over the terminal attractions. The molecules under such conditions will adhere, strictly maintaining the layered structure, although they might partially acquire a fluid condition. Under such situations, the layered smectic structure is maintained, giving rise to a smectic mesophase. As the temperature is further increased, the strict adherence of the molecules in the layered form is weakened due to increased thermal vibrations. The molecules slowly acquire a more fluid nature, although they do not abruptly acquire any randomness. At a definite temperature, the layered structure breaks down, but end-to-end parallel orientation is still maintained, giving rise to more fluidity and a nematic threaded texture to the molecules. Thus, polymorphism is exhibited from the fourth member up to the eighth member of the homologous series. From the tenth member onwards, a purely SmA mesophase is exhibited. The overall polarizability of the molecules is enhanced as the chain length of the alkyl chain at one end increases, contributing to the exhibition of smectic texture. However, the ratio of terminal to lateral attractions is also low that at a certain stage the thermal vibrations break down the orientation of molecules without giving them a chance for adopting a nematic orientation. Thus, only a SmA mesophase prevails from the *n*-decyloxy derivative.

Figure 2 shows the plot of transition temperatures against the number of carbon atoms in the alkoxy chain, from which it can be noticed that the crystal–mesophase/isotropic transition temperatures decrease sharply with an initial increase in the length of terminal alkoxy tail, which gives pronounced enantiotropic behaviour from the second member in the series **I**. The nematic–isotropic (N–I) transition temperatures also decrease with increase in the number of carbon

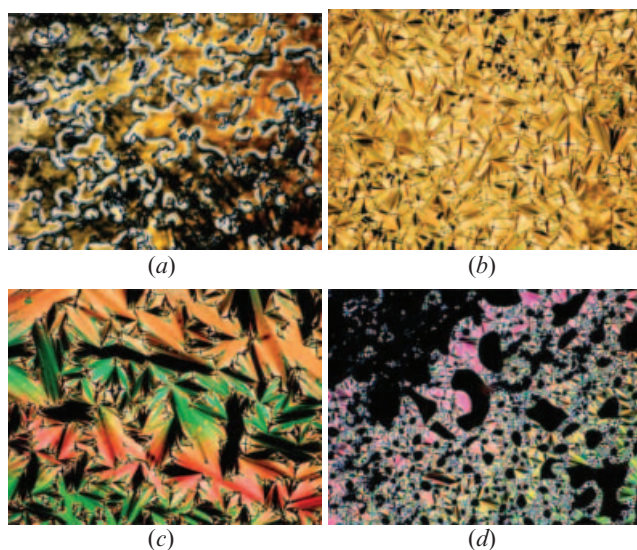


Figure 1. Optical textures of (a) compound **4** (120°C), (b) compound **4** (97°C) and (c) compound **16** (104°C). (d) Homeotropic alignments of compound **16** (104°C).

Table 2. DSC data for compounds of series **I** and series **II**.

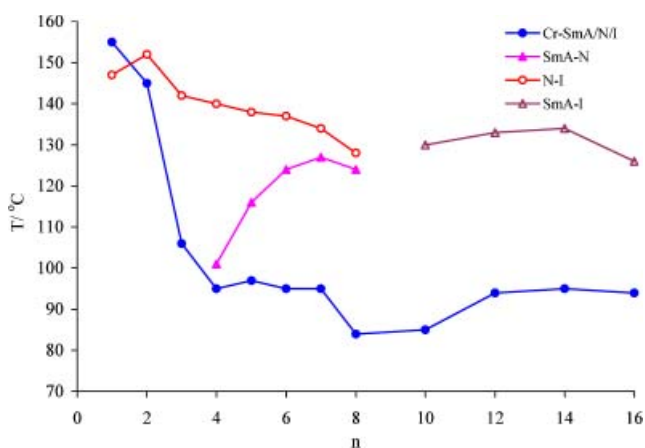
Series	Compound	Transition	Peak temp./°C	$\Delta H/J\ g^{-1}$	$\Delta S/J\ g^{-1}\ K^{-1}$
I	7	Cr–SmA	97.5	103.7 ^a	0.2799
		SmA–N	129.2	6.6	0.0164
		N–I	135.8	2.1	0.0051
	8	Cr–SmA	82.5	60.5 ^a	0.1702
		SmA–N	122.9	7.8	0.0197
		N–I	–	^b	–
II	17	Cr–SmA	105.7	54.98 ^a	0.1454
		SmA–I	111.6	8.8	0.0229
	19	Cr–SmA	93.2	56.94 ^a	0.1555
		SmA–I	117.0	11.2	0.0287

^aTotal enthalpy including any other Cr–Cr transition.

^bThe enthalpy could not be measured.

atoms in the alkoxy chain and exhibit the usual odd–even effect for the first three derivatives, whereas it exhibits a tendency for rising SmA–N as well as SmA–I transition temperature in the ascending series, which levels off slightly in the middle and higher homologues, respectively.

All twelve compounds synthesized in series **II** also exhibit mesomorphism. Methoxy to *n*-butyloxy derivatives exhibited a monotropic nematic mesophase. The SmA mesophase commences from the *n*-propyloxy derivative as a monotropic phase and persists up to the last homologue synthesized. Thus, *n*-propyloxy and *n*-butyloxy derivatives exhibited monotropic SmA as well as monotropic nematic mesophases. The plot of transition temperatures against the number of carbon atoms in the alkyl chain (figure 3) reveals a tendency for N–I transition temperatures to increase with the usual odd–even effect for lower members, i.e. the N–I transition temperatures are altered with odd and even number of carbon atoms in the alkyl chain. The SmA–N and SmA–I transition temperatures exhibit a tendency to increase that levels off for SmA–I transition

Figure 2. The phase behaviour for series **I** compounds.

temperature for last homologue synthesized. The SmA–I transition temperatures also exhibited little odd–even effect. The tendency of rising transition curve has been considered to be a characteristic of low-melting mesogenic homologous series.

Table 3 provides a comparison of smectic mesophase range, transition temperatures and molecular structure of representative compounds **10** and **22** ($n=12$) of the present series **I** and **II**, respectively, as well as structurally related compounds **A** [32], **B** [23], **C** [27], **D** [33], **E** [25], **F** [28] and **G** [34]. The smectic range of compound **10** is about 39°C, whereas that of compound **22** is 47°C. However, the SmA–I transition temperature for the compound **10** is little higher (8°C) when compared with that of compound **22**. The molecular structure of compound **10** differs from compound **22** only at the one terminus. Compound **10** has a methoxyethyl tail, whereas compound **22** has a chloroethyl tail at the same end. Gray [35] explained that

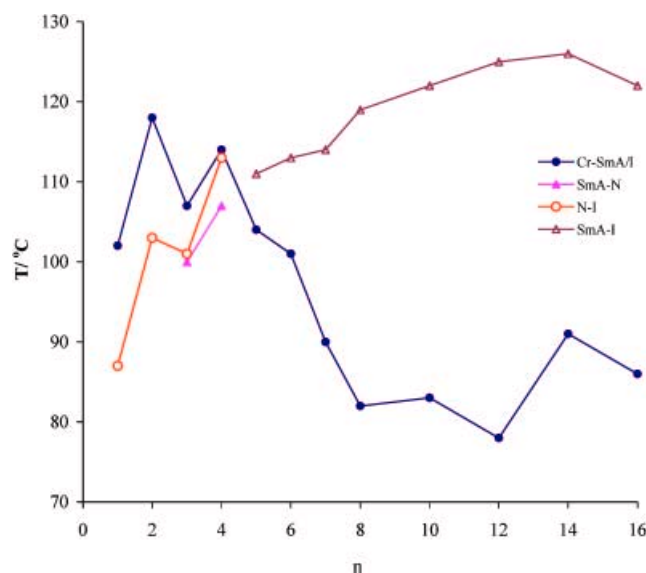
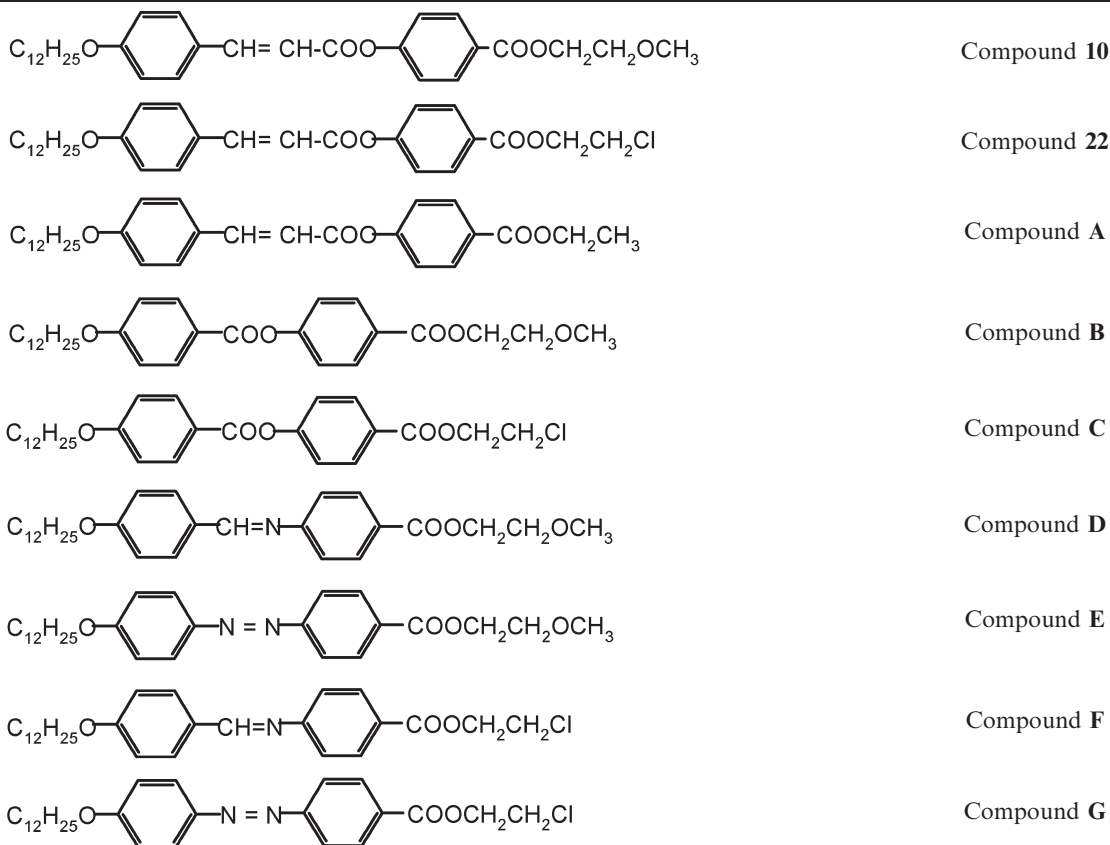
Figure 3. The phase behaviour for series **II** compounds.

Table 3. The smectic mesophase range, phase transition temperatures and comparative molecular structure of compounds **10**, **22** and **A–G**.

Compound	Mesophase range/°C	SmA–I/°C	Commencement of smectic phase
10	39	133	C ₄
22	47	125	C ₃
A	45	120	C ₂
B	29	71	C ₅
C	28	87	C ₆
D	10	68	C ₅
E	07	79	C ₄
F	38	104	C ₅
G	30	102	C ₄



a compound that requires more thermal energy to disorganize its parallel molecular arrangement in the nematic phase is more thermally stable. As can be seen in table 3, more thermal energy has to be supplied to disorganize the parallel molecular arrangement of the smectic melt of compound **10**, evidenced by the fact that the SmA–I transition temperature is higher by 8°C than that for the compound **22**. The higher smectic–isotropic transition temperatures of compound **10** may be due to the longer tail at one end, which may increase the overall polarizability of the molecules of compound **10** compared to compound **22**. The range and thermal stability of the

mesophase is a more important factor in relating mesomorphic behaviour to chemical constitution, since the chemical grouping gives rise to intermolecular attractions, which in turn determine the mesophase range and thermal stability. One should remember too that the length of the mesophase is determined partly by the Cr–SmA transition temperatures.

Table 3 shows that the SmA–I transition temperatures of compound **10** and **22** are higher by 13°C and 5°C, respectively, than that of compound **A**. This is understandable since a H atom of the terminal ethyl chain of compound **A** is replaced by the more polar –

OCH₃ and –Cl groups in molecules of compound **10** and **22**, respectively.

Table 3 also shows that the width of smectic mesophase and the SmA–I transition temperature of compound **10** is higher by 10°C and 62°C, respectively, compared with that of compound **B**. The molecules of compound **10** and compound **B** differ only at the central linkages. The molecules of compound **10** have cinnamoyloxy (–CH=CH–COO–) central linkages, whereas compound **B** has an ester (–COO–) central linkage. Gray [35] explained that the addition of double bond to the system increases the length and polarizability of rod-like molecules. Therefore, the greater smectic mesophase range and thermal stability of series **I** can be explained in terms of the greater molecular length and polarizability of the molecule resulting from additional –CH=CH– units in the central linkage. This is also reflected by compound **22** and compound **C**, where the smectic mesophase range and SmA–I transition temperature of compound **22** are higher by 19°C and 38°C, respectively, compared with those of compound **C**.

The smectic mesophase range of compound **10** is higher by 29°C and 32°C compared with compound **D** and compound **E**, respectively (table 3). In addition, the SmA–I transition temperature of compound **10** is also higher by 65°C and 54°C compared with compound **D** and compound **E**, respectively. The molecules of compound **10**, **D** and **E** differ only at the central linkages. Compound **10** has a cinnamoyloxy central linkage, whereas compounds **D** and **E** have azomethine and azo central linkages, respectively. As discussed above, cinnamoyloxy (–CH=CH–COO–) central linkages increase the length and polarizability of the rod-like molecules, which are responsible for the higher transition temperatures and greater mesophase length as compared with compounds **D** and **E**. For the same reasons the mesophase length of compound **22** is higher by 9°C and 17°C compared with compound **F** and compound **G**, respectively, and the SmA–I transition temperature of compound **22** is also higher by 21°C and 23°C, respectively.

4. Conclusions

In this article we have presented the synthesis and characterization of two new mesogenic homologous series of liquid crystalline cinnamates with a substituted ethyl tail. Series **I** with a methoxyethyl tail has higher thermal stabilities compared with series **II** with a chloroethyl tail. Both series with a cinnamoyloxy central linkage are more stable compared with the structurally related series containing ester, azomethine or azo central linkages due to the greater molecular length and polarizability of the molecule resulting from additional –CH=CH– units in the central linkage.

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