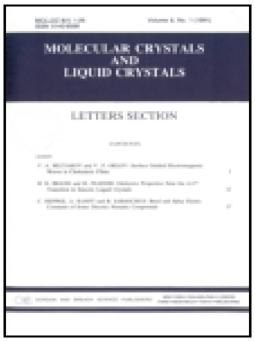

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Synthesis and Mesomorphic Properties of a Novel Ester Homologous Series: 4-(4′-*n*-Alkoxy Benzoyloxy) Benzyl Cinnamates

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A novel homologous series 4-(4′-n-alkoxy benzoyloxy) benzyl cinnamates was synthesized and studied with a view to understanding and establishing the relationships between mesomorphism and molecular structure. The novel series consists of eleven members with the commencement of mesomorphism from the pentyloxy homologue onwards to the hexadecyl homologue. The remaining homologues are nonmesomorphic. All the mesomorphic homologues exhibit smectogenic mesomorphism in addition to nematogenic mesomorphism for a definite temperature range in an enantiotropic manner, except for the pentyloxy homologue, which exhibits only a nematogenic mesophase in an enantiotropic manner without the formation of a smectic phase. The texture of the nematic mesophase is of the threaded or Schlieren type and the smectic mesophase is of the type A or C texture, as determined by a miscibility method. Transition temperatures were observed through an optical polarizing microscope equipped with a heating stage. The transition curves of a phase diagram predominantly behave in a normal manner, but partly deviated from normal behavior with the exhibition of an odd–even effect. Analytical and spectral data confirms the molecular structures of all homologues. The mesomorphic behavior of the present series is compared with other structurally similar known series. The mesomorphic temperatures vary between 106°C and 180°C. The mesophase length varies between 33°C to 53°C. Thus, the novel series is predominantly nematogenic and partly smectogenic with relatively high transition temperatures and is of a high melting type. The average thermal stabilities for smectic and nematic phases are 144.1°C and 171.4°C, respectively.

Keywords Enantiotropy; liquid crystals; mesomorphism; nematic; smectic

Introduction

The phenomenon of mesomorphism is not exhibited by all substances, but some substances, whose molecular structure generates favorable magnitudes of molecular rigidity and flexibility [1–3] to enable and maintain their molecular arrangement in a two-dimensional array in a floating condition, either in a layered manner by lamellar molecular packing in their crystal lattices or through a statistically parallel orientational order of molecules, can exhibit mesomorphic behavior. Cinnamic acids with an *n*-alkoxy terminal group and

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their ester derivatives [4,5] acquire suitable magnitudes of anisotropic forces of intermolecular attractions [6,7], as a consequence of favorable molecular rigidity and flexibility they can exhibit mesomorphism. Thus, the proposed investigation is planned to synthesize novel ester derivatives consisting of three phenyl rings bridged through $-COO-$ and $-CH{=}CH-COO-CH_{2-}$ central groups with a left *n*-alkoxy (–OR) terminal end group without any lateral or right terminal end group [8]. Hence, linear, lath-like molecules have sufficient end to end lateral intermolecular attractions to cause or to induce mesomorphism depending upon the molecular structure.

Experimental

Synthesis

4-Hydroxy benzoic acid was alkylated with appropriate alkylating agents (R–X) to prepare 4-*n*-alkoxy benzoic acids (A) by the modified method of Dave and Vora [9]. Component (B) viz. 4-hydroxy benzyl cinnamate was prepared by a usual established method [10,11] (m.p 150°C, yield 80%). Components A were converted to their acid chlorides using dry thionyl chloride. The acid chlorides were then condensed with a component B in dry cold pyridine [12,13] to prepare final ester derivatives of the novel homologous series. Final products were individually decomposed, filtered, washed, dried, and purified by EtOH until constant transition temperatures were obtained. The synthetic route to the novel homologous series is shown in scheme-1. 4-Hydroxy benzoic acid, 4-hydroxy cinnamic acid, benzyl alcohol, alkyl halides, MeOH, EtOH, KOH, toluene, sulfuric acid, thionyl chloride, and pyridine required for synthesis were used as received, except solvents, which were dried and purified prior to use.

Characterization

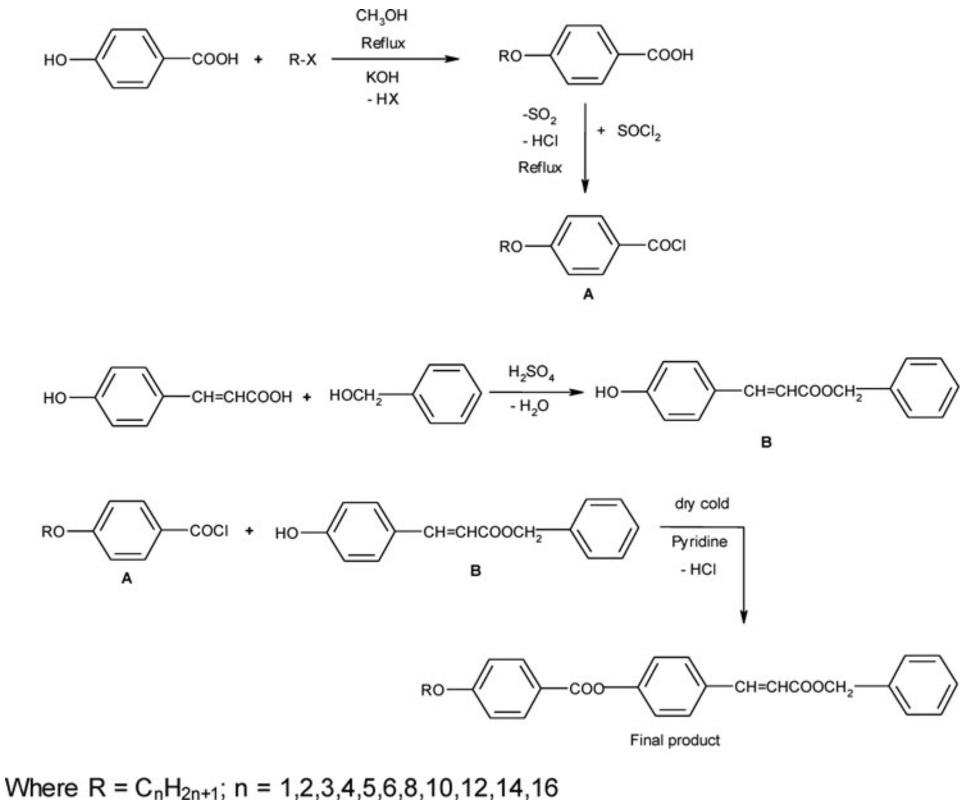
Some selected members of the present novel series were characterized by elemental analysis, infra red spectra, ^{1}HNMR spectra, and mass spectra. Microanalysis was performed using a Perkin-Elmer PE 2400 CHN analyzer, ^{1}HNMR spectra were recorded on Bruker spectrometer using DMSO solvent. Infrared spectra were recorded on a Perkin-Elmer spectrum GX spectrometer. Transition temperatures and the textures of the smectic and nematic mesophases as determined by a miscibility method were characterized by an optical polarizing microscope equipped with a heating stage. Thermodynamic quantity enthalpy change (ΔH) and entropy change ($\Delta S = \Delta\ \Delta H/T$) are qualitatively discussed.

Analytical Data (Table 1)

Spectral Data

^{1}HNMR in ppm for the decyloxy derivative: 1.23 (Long alkyl chain), 1.71 ($-CH_2-CH_2-O-$ of $C_{10}H_{21}$), 3.31 ($-OCH_2$ of $-OC_{10}H_{21}$ group), 3.9 ($-COO-CH_2-C_6H_5$), 6.3 and 6.83 (p-substituted phenyl ring), 7.85 (mono-substituted phenyl ring), 4.00 (trans $-CH{=}CH-$ group).

^{1}HNMR in ppm for the dodecyloxy derivative: 1.34 (Long alkyl chain), 1.72 ($-CH_2-CH_2-O-$ of $C_{14}H_{29}$), 3.3 ($-OCH_2$ of $-OC_{12}H_{25}$ group), 3.92



Scheme 1. Synthetic route to the series.

($-$COO$-$CH$_2-$C$_6$H$_5$), 6.3 and 6.9 (p-substituted phenyl ring), 7.8 (mono-substituted phenyl ring), 4.1 (trans $-$CH$=$CH$-$ group).

IR in cm^{-1} for the hexyloxy derivative: 696 and 733 (mono-substituted phenyl ring), 891 (p-substituted phenyl ring), 1200 (ether linkage of $-C_6H_{13}O-C_6H_4$), 1168, 1256, 1605 ($-$COO$-$ester group), 934 (trans $-$CH$=$CH$-$ group).

IR in cm^{-1} for the tetradecyloxy derivative: 695 and 732 (mono-substituted phenyl ring), 889 (p-substituted phenyl ring), 1198 (ether linkage of $-C_{14}H_{29}O-C_6H_4$), 1169, 1258, 1606 ($-$COO$-$ester group), 939 (trans $-$CH$=$CH$-$ group).

Mass Spectra

Mass spectra for the butoxy derivative: Molecular formula $C_{27}H_{26}O_5$; Calculated Molecular weight 430. Practical mass observed by Mass spectroscopy 430.

Texture of Nematic and Smectic Phase by Miscibility Method

Pentyloxy derivative → Threaded nematic

Hexadecyloxy derivative → Schlieren nematic and smectic C

Octyloxy derivative → Threaded nematic and smectic A

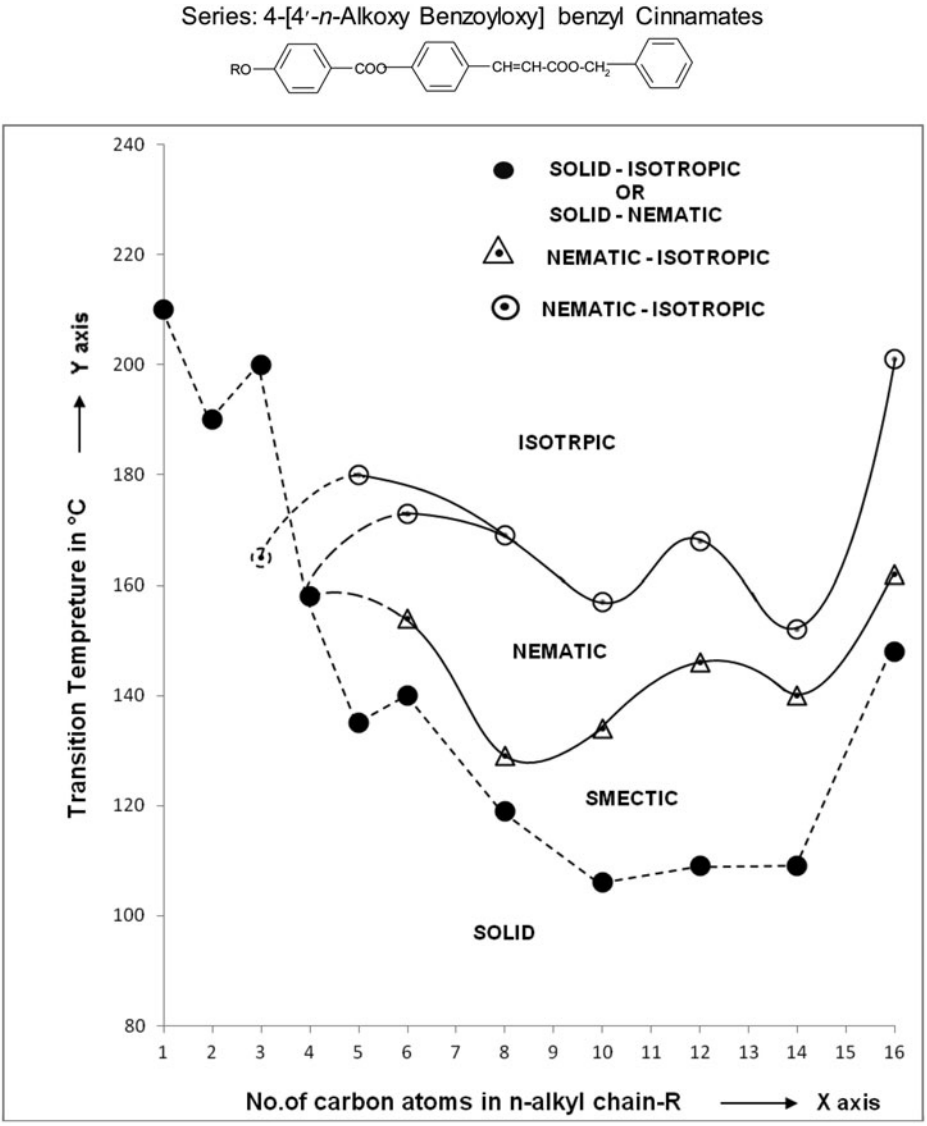


Figure 1. Phase behavior of series.

Results and Discussion

4-Hydroxy-benzyl cinnamate is nonmesomorphic, but on condensing with 4-*n*-alkxoy benzoic acids through their corresponding acid chlorides mesomorphic homologues are generated. The novel homologous series contains eleven members. The methyl to butyl homologues of the series are nonmesomorphic, while the pentyl to hexadecyl derivatives are mesomorphic. All the mesomorphic homologues, except for the pentyloxy homologue exhibit smectogenic and subsequently nematogenic character in enantiotropic manner, while only the pentyloxy homologue exhibits nematogenic character without the formation of a smectogenic mesophase. A phase diagram (Fig. 1) of the series is plotted for the transition temperatures (Table 2) as determined by an optical polarizing microscope equipped

Table 1. Elemental analysis for methoxy, ethoxy, and propoxy derivatives

		Elements % found (% calculated)	
Sr. No.	Molecular formula	C	H
1	$C_{24}H_{20}O_5$	74.35 (74.22)	5.26 (5.15)
2	$C_{25}H_{22}O_5$	74.51 (74.62)	5.31 (5.47)
3	$C_{26}H_{24}O_5$	74.98 (75.00)	5.79 (5.76)

with a heating stage, against the number of carbon atoms present in *n*-alkoxy chain of the *n*-alkoxy terminal end group. Smooth curves are drawn by linking like or related points for the solid–mesomorphic, solid–smectic, smectic–nematic, nematic–isotropic, or solid–isotropic transition points. Thus, the phase diagram consists of three transition curves viz. solid–mesomorphic or isotropic, smectic–nematic and nematic–isotropic. The solid–isotropic or mesomorphic transition curve follows a zigzag path of rising and falling values in the usual established manner. The smectic–nematic and nematic–isotropic transition curves adopt a partly descending tendency in a normal manner, but they deviate from normal behavior for very few members of the series. The nematic–isotropic transition curve exhibits an odd–even effect, while the smectic–nematic transition curve does not show an odd–even effect because the smectic mesophase is exhibited by the even numbered homologue derivatives. The nematic–isotropic transition curves for the odd and even members were extrapolated [14,15] to determine latent ability of propoxy and butoxy homologues to show mesomorphic behavior and to magnify odd–even effect for the nematic transition curve. The odd–even effect disappears from and beyond the octyloxy homologue. Analytical and spectral data of homologues matches with their molecular structures. The smectic mesophase length varies between 10°C to 37°C and nematic mesophase length varies between 12°C to 45°C. The average thermal stabilities for the smectic and nematic phases are 144.1°C and 171.4°C, respectively. The mesomorphic properties of the homologues vary

Table 2. Transition temperatures of series in °C

		Transition temperature in °C		
Compound no.	*n*-alkyl group ($-C_nH_{2n+1}$) (*n*)	*Sm*	*Nm*	Isotropic
1	1	—	—	210.0
2	2	—	—	190.0
3	3	—	—	200.0
4	4	—	—	158.0
5	5	—	135.0	180.0
6	6	140.0	154.0	173.0
7	8	119.0	129.0	169.0
8	10	106.0	134.0	157.0
9	12	109.0	146.0	168.0
10	14	109.0	140.0	152.0
11	16	148.0	162.0	201.0

Sm: Smectic *Nm:* Nematic

from homologue to homologue of the series under discussion. The series is predominantly nematogenic and partly smectogenic and of a high melting type.

The esterification process through acid chloride formation breaks the hydrogen bonding between two molecules of the 4-*n*-alkoxy benzoic acids and dimerization disappears. Nonmesomorphicity of the methoxy to butoxy homologues is attributed to their shorter *n*-alkyl chain, which normally strengthens intermolecular attractions to raise the crystallizing tendency of a substance, without formation of lamellar packing of molecules in their crystal lattices. Thus, such molecular packing is unable to withstand exposed thermal vibrations, and undergo abrupt breaking of their crystal lattices which, suddenly transform into isotropic liquid from solid crystalline state, at melting temperature with the absence of any sort of two-dimensional order in a floating condition. The molecules are randomly oriented in all possible directions with high order of disorder or entropy ($\Delta s = \Delta H/T$). Exhibition of a smectogenic and/or a nematogenic mesophase or mesophases by the pentyloxy to hexadecyloxy homologues is attributed to the disalignment of molecules at an angle less than 90° under observation through a hot stage polarizing microscope adopting either a sliding-layered or statistically parallel orientational-ordered arrangement of the molecules in a floating condition under the influence of heat. As heat is applied to a sample, the intermolecular attractive forces are weakened and as a consequence of favorable molecular rigidity and flexibility, the molecules under examination adopt sliding-layered arrangement due to the lamellar packing of molecules in their crystal lattices, which results in the appearance of a smectogenic mesophase from the hexyloxy to hexadecyloxy homologues. The pentyloxy homologue does not facilitate smectogenic behavior which is due to the absence of lamellar packing in their crystal lattices and hence, molecular arrangement adopts only a statistically parallel orientational order of molecules in floating condition. The nonmesomorphicity of the propoxy and butoxy homologues was examined by extrapolating the nematic–isotropic transition curves for odd (third) and even (fourth) homologues. The extrapolated transition curve for the propoxy homologue indicates 165°C as latent transition temperature (LTT) showing latent ability of the propoxy homologue to exhibit the nematic mesophase monotropically in an irreversible manner below its isotropic temperature. However, it is not realizable due to its high crystallizing tendency. The extrapolated transition curve for smectic and nematic merges into the isotropic point of 158°C for the butoxy homologue, which indicates that neither monotropic, nor enantiotropic nematic or smectic mesophase is possible to emerge. Moreover, extrapolated transition curves magnify the odd–even effect. The odd–even effect diminishes as the series is ascended and merges into each other at the octyloxy homologue. This odd–even effect disappears from and beyond the octyloxy homologue because the longer *n*-alkyl may coil or bend or flex or couple to lie with major axis of the core structure and follows as a single curve. Variations in mesomorphic properties from homologue to homologue in present or same series is attributed to the progressive addition of methylene units. Relatively higher transition temperatures of homologues and high degrees of mesomorphism may be attributed to the presence of the multiple bond in the vinyl cinnamate central group, and linear lath-like molecular geometry, which enhances molecular rigidity and flexibility, inducing higher thermal stability to maintain two dimensional array of molecules in a floating condition. Thus, the presently investigated novel series is predominantly nematogenic and partly smectogenic. The mesomorphic properties of the present series-1 are compared with other structurally similar known series *X* [16] and *Y* [17] as shown in Fig. 2.

Homologous series 1, *X* and *Y* have structural similarities and differences are clearly shown in Fig. 2. Mesomorphic behavior of the three series of homologues and the degree of mesomorphism will differ on the basis of differing molecular structure as a

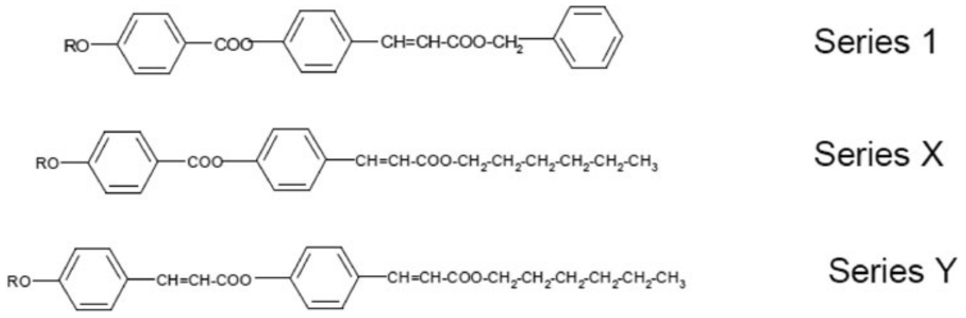


Figure 2. Structurally similar homologous series.

consequence of changing molecular rigidity and flexibility affecting magnitudes of intermolecular anisotropic forces of attractions. Table 3 as mentioned below shows the average thermal stability for smectic and nematic phases, the commencement of the smectic and nematic mesophases, and the group efficiency order.

Table 3 shows the mesomorphic behavior of the presently investigated novel homologous series 1 and series *X* and *Y* chosen for the comparative study. Series 1 and *X* are structurally similar for $RO{-}C_6H_4{-}COO{-}C_6H_4{-}CH{=}CH{-}COO{-}CH_2$–, whereas they differ from series *Y* from a central bridge linking the two phenyl rings by the $-CH{=}CH-$ unit from the common molecular part. The rest of the molecular part contained by series 1 is the aromatic phenyl ring while series *X* and *Y* contain commonly the $-CH_2{-}CH_2{-}CH_2{-}CH_2{-}CH_3$. Thus, the uncommon parts from series 1, *X*, and *Y* cause differences in magnitudes of intermolecular anisotropic forces of lateral and terminal attractions as a consequence of differing magnitudes of molecular rigidity and flexibility [12,13]. All the homologous of series 1, *X*, and *Y* exhibit smectic and then nematic mesophases, for definite temperature length, but their phase lengths are different due to their minor structural variation for the same homologue from series to series. Thermal stabilities for smectic and nematic of series 1 are more than for series *X*, but are less than for series *Y*. The relatively higher value of thermal stabilities of series 1 than series *X* is attributed to the additional phenyl ring substituted in place of the $-CH_2{-}CH_2{-}CH_2{-}CH_2{-}CH_3$ terminal chain, and of series *X* bonded to a middle common $-CH{=}CH{-}COO{-}CH_2$– unit. The difference of molecular rigidity induced by the aromatic phenyl ring of series 1 being more than the molecular flexibility induced by $-CH_2{-}CH_2{-}CH_2{-}CH_2{-}CH_3$ terminal chain. Conversely, the molecular flexibility

Table 3. Average thermal stability in °C

Series→	Series 1	Series *X*	Series *Y*
smectic-nematic Or smectic-isotropic commencement of smectic phase	144.1 ($C_6{-}C_{16}$) C_6	93.2 ($C_8{-}C_{14}$) C_8	179.6 ($C_7{-}C_{14}$) C_7
nematic-isotropic commencement of nematic phase	171.4 ($C_5{-}C_{16}$) C_5	136.4 ($C_6{-}C_{14}$) C_6	190.3 ($C_3{-}C_{14}$) C_3

induced by $-CH_2-CH_2-CH_2-CH_2-CH_3$ chain in the case of series *Y* exceeds the intermolecular attraction of the central bridge $-CH{=}CH-COO-$ in place of central bridge $-COO-$ of series 1. Thus, the central bridge which is $-CH{=}CH-COO-$ in case of homologous series *Y* and carboxylate $-COO-$ in the case of series 1 and *X* under comparison are closely similar to each other. Both these central bridges are comparable, though the vinyl carboxylate $-CH{=}CH-COO-$ has a greater length and causes more noncoplanarity due to a twist obtained as the oxygen atoms of the vinyl carboxylate group bump into the nonbonded adjacent hydrogen atoms of the aromatic rings. On account of these differences, the intermolecular lateral and terminal attractions of a homologous series *Y* exceeded those of homologous series 1 and *X*. Hence, smectic and nematic thermal stabilities of series *Y* are higher than series 1 and *X*. Earliest commencement of the nematic mesophase formation in case of series *Y* as compared to series 1 and *X* is related to the order of magnitudes of the anisotropic forces of intermolecular attractions caused by the varying molecular length to breadth ratio, and hence the variation of the ratio of the molecular polarity to polarizability. Alternation of transition temperatures, odd–even effect, and variations of mesomorphic properties from series to series for the same homologue or homologue to homologue in the same series is attributed to the changing methylene unit of end group.

Conclusions

- Group efficiency order derived on the basis of thermal stabilities for smectic and nematic mesophase formation is as under.
  Smectic:

$-CH{=}CH-COO-CH_2-(CH_2)_4-CH_3 >$
with
$-CH{=}CH-COO-$
$-CH{=}CH-COO-CH_2-C_6H_5 >$
with
$-COO-$
$-CH{=}CH-COO-CH_2-(CH_2)_4-CH_3$
with
$-COO-$

  Nematic:

$-CH{=}CH-COO-CH_2-(CH_2)_4-CH_3 >$
with
$-CH{=}CH-COO-$
$-CH{=}CH-COO-CH_2-C_6H_5 >$
$-CH{=}CH-COO-CH_2-(CH_2)_4-CH_3$

- Group efficiency order derived on the basis of early commencement of smectic and nematic is as under.

Smectic:

$$-\mathrm{CH{=}CH{-}COO{-}CH_2{-}C_6H_5} >$$
with
$$-\mathrm{COO}-$$
$$-\mathrm{CH{=}CH{-}COO{-}CH_2{-}(CH_2)_4{-}CH_3} >$$
with
$$-\mathrm{CH{=}CH{-}COO}-$$
$$-\mathrm{CH{=}CH{-}COO{-}CH_2{-}(CH_2)_4{-}CH_3}$$
with
$$-\mathrm{COO}-$$

Nematic:

−CH=CH−COO−CH2−(CH2)4−CH3 >
with
$$-\mathrm{CH{=}CH{-}COO}-$$
$$-\mathrm{CH{=}CH{-}COO}x\mathrm{{-}CH_2{-}C_6H_5} >$$
with
$$-\mathrm{COO}-$$
$$-\mathrm{CH{=}CH{-}COO{-}CH_2{-}(CH_2)_4{-}CH_3}$$
with
$$-\mathrm{COO}-$$

- The novel homologous series is predominantly nematogenic and partly smectogenic with considerable mesomorphic phaselength and higher-ordered melting type.
- Mesomorphism exhibition of a substance is very sensitive and susceptible to minute change in the molecular structure.
- Suitable magnitudes of molecular rigidity and flexibility play an important role in inducing mesomorphism in a substance.
- Variations in mesomorphism from homologue to homologue in the same series is due to the sequential addition of methylene unit or units, to the *n*-alkyl chain of a left *n*-alkoxy end group.
- Absence of lamellar packing of molecules in the crystal lattices hinders the smectogenic property.

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