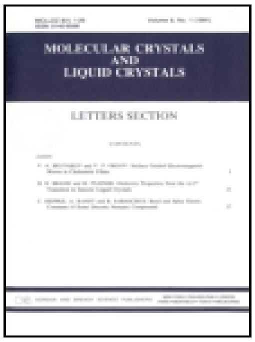

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Molecular flexibility dependence mesomorphism

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ABSTRACT

A novel series of chalconyl homologue derivatives with $RO.C_6H_4.COO.C_6H_4.CO.CH{:}CH\text{-}C_6H_4.N.(CH_3)_2$ (Para) have been synthesized and thermotropically studied with a view to understanding and establishing the effect of molecular structure on mesomorphic behavior of the novel homologues. The series consists of thirteen homologues (C_1 to C_{18}). C_7 to C_{18} homologues are smectogenic, C_4 to C_{18} nematogenic and C_1 to C_3 nonmesomorphic. Thus, novel chalconyl series is predominantly nematogenic and partly smectogenic. Transition temperatures and textures of mesophases were determined using an polarizing optical microscope (POM) equipped with a heating stage. Analytical and spectral data confirmed molecular structures of homologues. Phase transition curves showing phase behavior in a phase diagram behaved in normal manner. Thermal stability for smectic and nematic are relatively low at 59.6 °C and 76.6 °C respectively whose, smectogenic and nematogenic phase length vary from 12 to 16 °C and 06 to 20 °C, respectively. Mesomorphic behaviors of the present series are compared with a structurally similar known homologous series.

KEYWORDS
Mesomorphism; smectic; nematic; monotropic; thermotropic

Introduction

The study of the liquid crystalline state(LC) [1] is a multidisciplinary subject useful to mankind and industrial applications [2–7] due to unique properties of flow as liquid and optical properties as crystals. Research on the LC state is planned with a view to understanding and establishing the effect of molecular structure on LC properties [8–11] as a consequence of molecular rigidity and flexibility [12–15]. This study will include synthesis of novel homologous LCs (thermotropic) of chalconyl derivatives in order to provide novel LC materials to various research groups. The novel substances were characterized by analytical and spectral data. LC properties were studied by using an optical polarizing microscope equipped with a heating stage. Several ester-chalconyl series have been reported to date [17–21]. The proposed investigation will include thermotropic properties and the comparative study of novel homologues with structurally similar series to derive group efficiency order.

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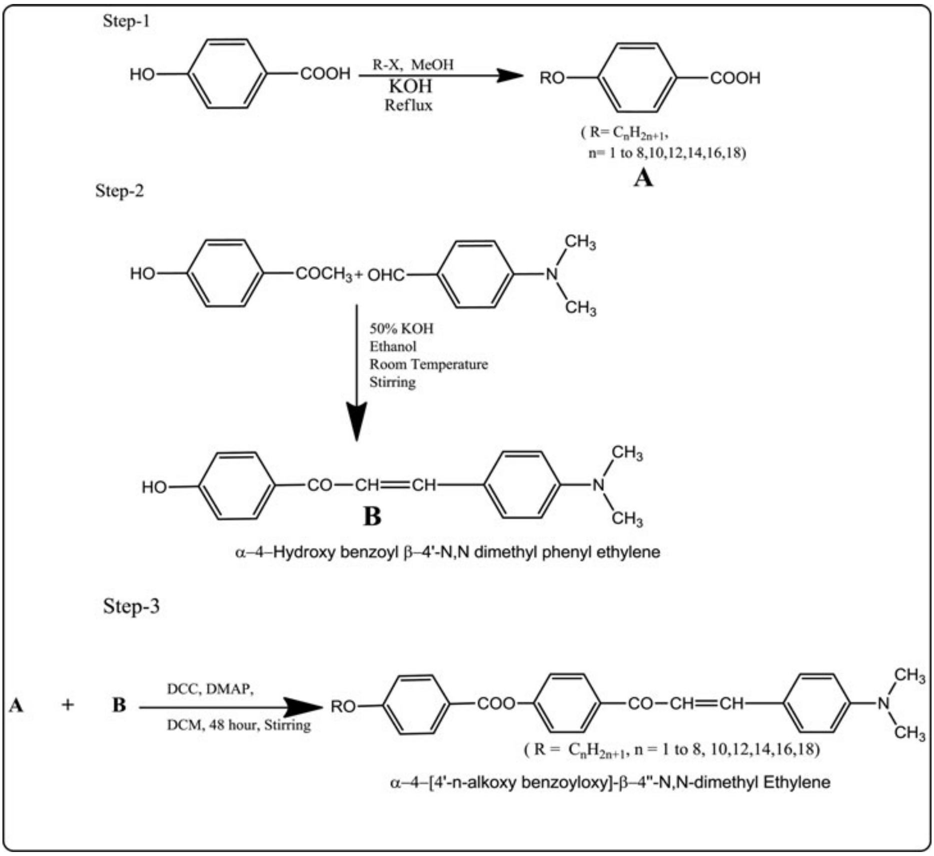


Scheme 1. Synthetic route to the Series-1.

Reaction scheme

Characterization

Representative homologues of a series were characterized by elemental analysis, Infrared spectroscopy, ^{1}H NMR spectra. IR spectra were recorded by Perkin-Elmer spectrum GX, ^{1}HNMR spectra were recorded on Bruker using $CDCl_3$ as solvent. Microanalysis was performed on Perkin-Elmer PE 2400 CHN analyzer (Table 1). Transition temperature and LC properties (Textures) were determined using an optical polarizing microscopy equipped with heating stage. Texture of some homologues for mesophase phase were determined by POM image (Fig. 1) and miscibility method (Table 2). Thermodynamic quantities enthalpy (ΔH) and entropy ($\Delta S = \Delta H/T$) are qualitatively discussed.

Table 1. Elemental analysis for Ethoxy, Pentyloxy, Dodecyloxy, Tetractadecyloxy Derivatives.

		% Elements found			% Elements theoretical		
Sr. No.	Molecular formula	C	H	N	C	H	N
1	$C_{26}H_{25}O_4N$	75.04	5.97	3.28	75.18	6.02	3.37
2	$C_{29}H_{31}O_4N$	75.98	6.71	2.98	76.14	6.78	3.06
3	$C_{36}H_{45}O_4N$	77.01	8.05	2.48	77.83	8.10	2.52
4	$C_{38}H_{49}O_4N$	78.14	8.32	2.37	78.21	8.40	2.40

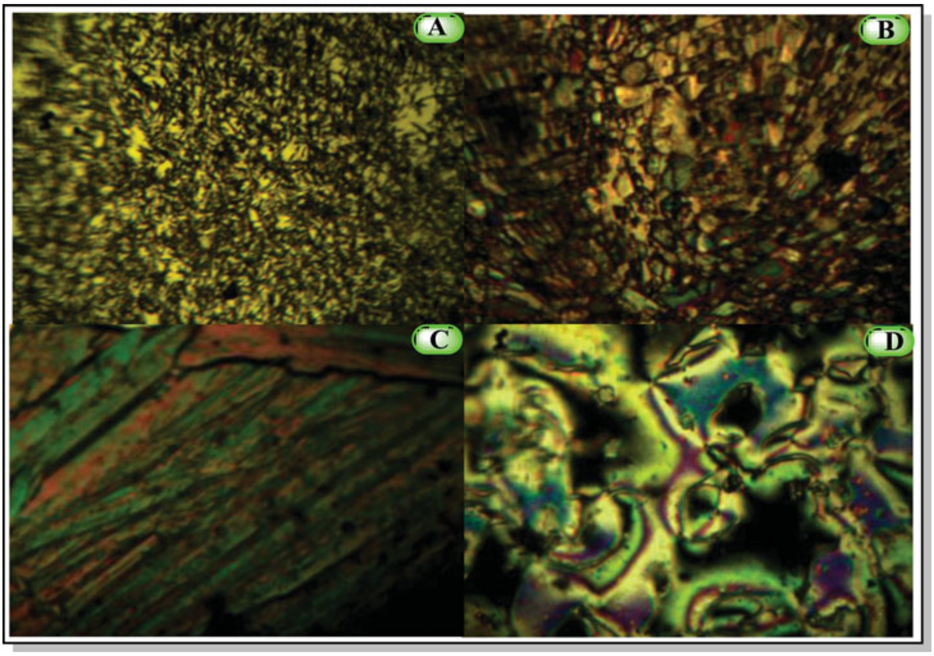
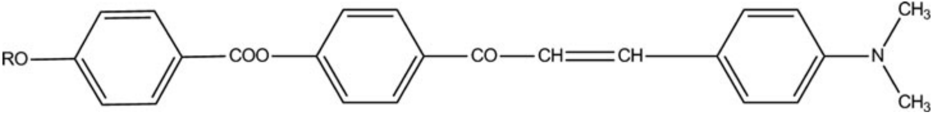


Figure 1. (A) Smectic C phase of C_8 at 62°C. (B) Smectic-C phase of C_{18} at 44°C. (C) Nematic phase of C10 at 61°C. (D) Nematic phase of C_{14} at 65°C.

Table 2. Texture by miscibility method.

Homologue	Texture image
C_4	Threaded nematic phase
C_6	Threaded nematic phase
C_{12}	Schlieren Nematic phase
C_{18}	Smectic C phase

Table 3. Transition Temperatures in °C for Series-1.



Homologous Series: α-4-[4$'$-n-alkoxy benzoyloxy]-β-4$'''$-N,N-dimethyl Ethylene.

		Transition Temperatures in(°C)		
Compound No	n-alkyl chain C_nH_{2n+1}	Smectic	Nematic	Isotropic
1	C_1	—	—	125.0
2	C_2	—	—	118.0
3	C_3	—	—	115.0
4	C_4	—	64.0	81.0
5	C_5	—	89.0	95.0
6	C_6	—	92.0	105.0
7	C_7	65.0	77.0	88.0
8	C_8	62.0	74.0	92.0
9	C_{10}	47.0	61.0	79.0
10	C_{12}	54.0	70.0	82.0
11	C_{14}	52.0	65.0	78.0
12	C_{16}	46.0	61.0	81.0
13	C_{18}	44.0	58.0	75.0

Analytical data

IR Spectra in cm^{-1} for Chalcone, Butyloxy, Octyloxy Derivatives:

Chalcone: 3016 (-C-H- str in CH_3), 1743 (-C=O) stretch, 1512 (-C=C-) str, 1365 and 1219 (-C-O str), 979 (-C=CH), 802 1,4-disubstituted aromatic ring.

Butyloxy: 636 Polymethylene (-CH_2-)n of $-OC_4H_9$, 839(–C-H- def. di-substituted-Para), 759 Polymethylene (-CH_2-) of $-OC_4H_9$, 952 (–C-H- def. hydrocarbon), 1028 and 1068(-C-O-) Str, 1197 and 1228 (-C-O str in –(CH_2)n chain, 1427 and 1427 (-C-H- def. in CH_2)1514 (-C=C-)str, 1604 and 1666 (-C=O group), 1726 (-COO- ester group), 2850 and 2970 (-C-H str in CH_3).

Octyloxy: 640 Polymethylene (-CH_2-)n of $-OC_8H_{17}$, 725 Polymethylene (-CH_2-)n of C_8H_{17} 817(–C-H- def. di-substituted-Para), 987 (–C-H- def. hydrocarbon), 1068 (-C-O-) Str, 1344 and 1271, 1186 (-C-O) str in –(CH_2)n chain, 1344 and 1435 (-C-H- def. in CH_2),1531(-C=C-)str, 1622 (-C=O group), 1732 (-COO- ester group), 2850 and 2927(-C-H str in CH_3).

^{1}H NMR spectra in $CDCl_3$ in δ ppm for Hexyloxy, Decyloxy Derivative:

Hexyloxy: 0.86(t, -CH_3 of $-C_6H_{13}$), 1.25-1.45(m, n-poly methylene groups of-C_6H_{13}), 1.45(m, n-poly methylene groups of $-C_6H_{13}$),4.0 (s,-OCH_2-CH_2-of -C_6H_{13}),4.01 (s,-OCH_2-CH_2-of C_7H_{15}),7.2-7.3(s,$CDCl_3$), 6.9-7.1(s,-CO-CH=CH),7.5-7.7(s, p-substituted phenyl ring).8.1 (s, p-disubstituted phenyl ring).

Decyloxy:0.90(t, -CH_3 of $-C_{12}H_{25}$), 1.25-1.33(m, of $-C_{10}H_{21}$), 1.7-1.9(m, n-poly methylene groups of- $OC_{10}H_{21}$),3.9(s,-OCH_2-CH_2-of -$OC_{10}H_{21}$),3.05 (s, -$(CH_3)_2$), 3.9-4.0(t, of -OCH_2-CH_2-of $OC_{12}H_2$), 6.9-7.1(s,-CO-CH=CH),7.5-7.7(s, p-substituted phenyl ring).7.2-7.3(s, $CDCl_3$), 8.1(s, p-substituted phenyl ring).

Texture by POM image.

Results and discussion

α-4-Hydroxy benzoyl β-4$'$-N,N,dimethyl phenyl ethylene (m.p. 68°C, yield 74%) on linking with dimeric 4-n-alkoxy benzoic acid yielded mesomorphic chalconyl homologue derivatives with transition and melting temperatures respectively lower than corresponding n-alkoxy acids. Novel homologues of a series are smectogenic plus nematogenic (C_7 to C_{18}) and only nematogenic (C_4 to C_6). Rest of the homologues are nonmesomorphic. Transition temperatures are determined on POM are plotted against the number of carbon atoms present in n-alkyl chain of the left n-alkoxy (-OR) group and on linking like or related points, phase transition curves (Cr-M/I, Sm-N, and N-I) are obtained showing phase behaviors of series as shown in phase diagram in Figure 2. Cr-M/I transition curve adopt a zigzag path of rising and falling with overall descending tendency. Sm-N transition curve initially falls and then passes through maxima at the dodecyloxy (C_{12}) homologue and finally descends upto the last homologue (C_{18}). N-I transition curve initially rises and then descends as series ascended. Sm-N and N-I transition curves exhibited odd-even effect upto decyloxy homologue (C_{10}). Sm-N transition curve is extraploted [27–29] to C_5 and C_6 homologue to predict and determine probable monotropic transition temperatures and to intensity odd-even effect of Sm-N transition curve. Thus, Cr-M/I, Sm-N and N-I transition curves behaved in normal manner, curves for odd and even members are merging into each other at the decyloxy or homologues and further prolonged as a single curve upto the last homologue (C_{18}) bearing longer n-alkyl chain. Odd-membered N-I transition curve occupied lower position than even-membered transition curve up to C_{10} and in case of Sm-N transition curve reverse is the order of occupation of odd-even curves. Mesomorphic properties of present series varies from homologue

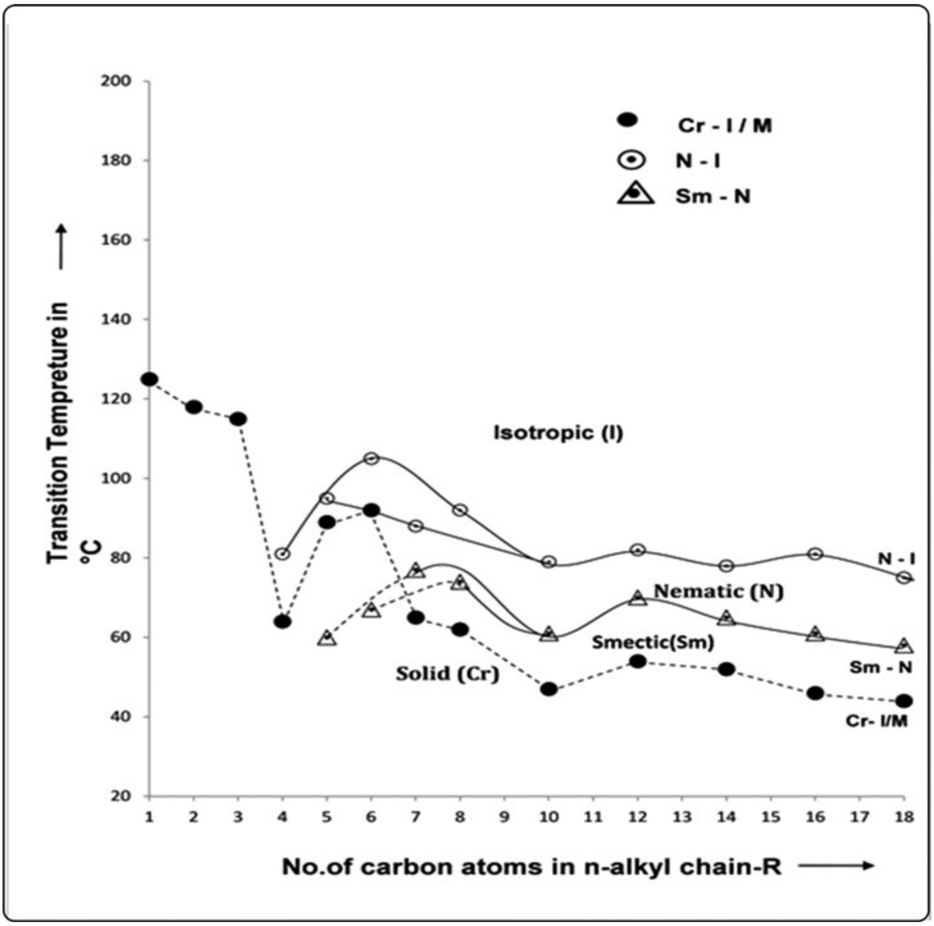


Figure 2. Phase diagram of Series-1.

to homologue keeping right $-N(CH_3)_2$ tail end group unchanged throughout the series. Analytical and spectral data confirmed the molecular structure.

Homologous Series : α-4-[4$'$-n-alkoxy benzoyloxy]-β-4$'$-N,N-dimethyl Ethylene

The lowering of transition temperatures of homologues belonging to novel chalconyl series is attributed to the breaking of hydrogen bonding between two molecules of corresponding 4-n-alkoxybenzoic acid through esterification process. The odd–even effect and the alternation of transition temperatures of homologues is due to the odd and even number of methylene units present in the n-alkyl chain of left n-alkoxy group. The exhibition of smectogenic character from and beyond C_7 homologue, indicates formation of lamellar packing of molecules in the crystal lattices of rigid crystals which maintains sliding-layered molecular arrangement in floating condition under the influence of exposed thermal vibrations, which causes the formation of smectogenic mesophase in enantiotropic manner. The exhibition of nematic property from C_4 to C_{18} homologue is attributed to the suitable magnitudes of anisotropic end to end intermolecular attractions as a consequence of favorable molecular rigidity and flexibility caused by appropriate permanent dipolemoment across the long molecular axis., dispersion forces, dipole–dipole interactions, shape, size, magnitudes of polarity and polarizability etc., which maintains floating of the homologue in stastically parallel orientational order to adopt nematogenic character, under exposed thermal vibrations; either directly or via smectic phase in enantiotropic manner. The late (from C_7) commencement of smectogenic character is due to the favorable extent of noncoplanarity by gradual increase of n-alkyl chain(-R), which commences to build up stable lamellar packing of molecules in their preoccupied rigid crystal lattices to facillate the formation and stabilization of smectic property, under thermometric environment. The absence of nematic and smectic property in C_1, C_2 and C_3 is attributed to their high crystallizing tendency which arises from low intermolecular dispersion forces and low dipole-dipole interactions which causes unsuitable magnitudes of intermolecular anisotropic forces of attractions, resulting into abrupt breaking of crystal lattices which sharply converted to isotropic state from solid crystalline state. Thus, a sample under microscopic observation fails to exhibit mesomorphic. The disappearance of odd-even effect from and beyond C_{10} homologue of longer n-alkyl chain is attributed to the uncertainty in the status of n-alkyl chain which may coil or bend or flex or couple to lye with the major axis of the core structure of a molecule. The changing magnitudes of mesogenic properties from homologue to homologue in the same series is due to the changing number of methylene unit or units which causes variations in the gradual variations in molecular rigidity and flexibility based on induced changing molecular polarity and polarizability. Molecules of every homologue randomly orient in all possible directions with high order of disorder or entropy ($\Delta S = \Delta H/T$) from and beyond isotropic temperature. But, on cooling the same from and below isotropic temperature, the mesophase is persisted to appear reversibly at the same temperature at which mesophase was disappeared. Thus, presently investigated novel series is predominantly nematogenic and partly smectogenic with low thermal stability and low degree of smectic and nematic mesophase formation with its low ordered melting type. Some mesomorphic properties of presently investigated novel series are compared with a structurally similar known series as under in Figure 3.

Homologous series-1 of present investigation and series X and Y chosen for comparative study are similar with respect to molecular rigidity contributed by three phenyl rings and two central bridges –COO- and –CO-CH=CH- for all the homologues of the same series as well as for the same homologue from series to series. Left n-alkoxy terminally situated end group –OR contributing to the total molecular flexibility is identical for the same homologue from series to series, but differs from homologue to homologue in the same series. Right handed tail group differs from series to series ($-N(CH_3)_2$, $-NO_2$, -Cl) but remain unchanged for the same series. Therefore the combined effect of molecular rigidity and flexibility operating the phenomena of mesomorphism differ from homologue to homologue in the same series and from

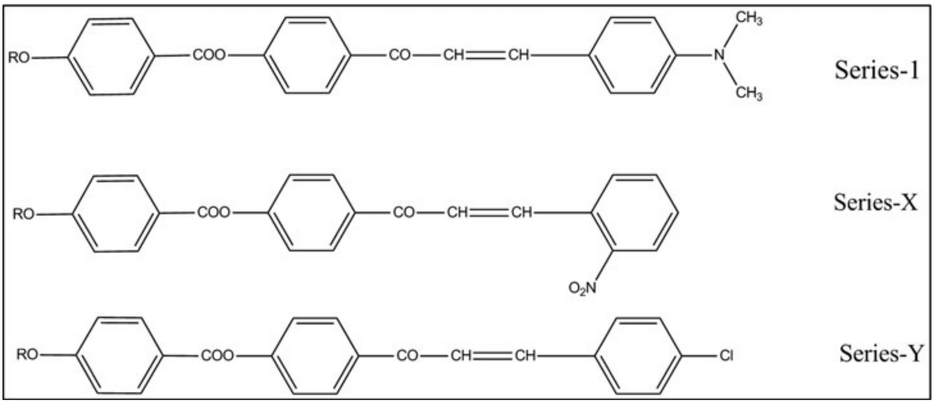
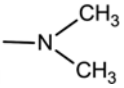


Figure 3. Structurally similar series.

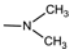
Table 4. Relative thermal stabilities in °C

Series	1 	X Ortho -NO_2	Y Para -Cl
Smectic-nematic Or Smectic-isotropic Commencement of Smectic phase	59.6 (C_7-C_{18}) C_7	138.0 (C_3-C_{14}) C_3	–
Nematic- Isotropic Commencement of Nematic phase	76.6 (C_4-C_{18}) C_4	163.7 (C_3-C_{16}) C_3	149.9 (C_5-C_{14}) C_5
Total upper and lower mesophase length range in °C. Ci to C_p	06.0 to 35.0 C_5 C_{16}	16.0 to 49.0 C_{16} C_6	14.0 to 25.0 C_7 C_{12} C_8

series to series for the same homologue which differs by more or less extent. Therefore the mesomorphic properties like degree of mesomorphism, thermal stability, commencement of mesophase or mesophases, group efficiency order, mesomorphic behaviors of transition curve in a phase diagram etc. differ by more or less extent depending upon the varying magnitudes of molecular rigidity and/or flexibility from one molecular structure to other molecular structure. Following Table 4 represent some mesomorphic properties of series-1, X [30] and Y [31] in comparative manner.

Careful observation of above Table 4 indicates that,

- Presently investigated series-1 and a structurally similar series-X are smectogenic in addition to nematogenic, whereas, series-Y is only nematogenic with absence of smectogenic character.
- Smectic property commences late from C_7 homologue of a series-1 whereas, it commences earlier from C_3 homologue by series-X and does not commence till the last member of a series-Y.
- Nematic property commences earliest from C_3 homologue in series-X and it commences from C_4 and C_5 homologue respectively in case of series 1 and Y.
- Smectic thermal stability of present novel series 1 is lower than a series-X, whereas it does not stabilize for single homologue in series-Y.
- Nematic thermal stability of present novel series-1 is the lowest among the series under comparison, whereas it is the highest for a series-X and it is of intermittent valued 149.9 of series-Y.
- Total upper and lower mesophase length is in increasing order from series-Y to series-1 to series-X and it and it is in decreasing order from series-X to series-Y to series-1 respectively.
- Odd-even effect is exhibited by the transition curve or curves of respective series.

The exhibition of smectogenic and /or nematogenic property by a homologous series is attributed to the suitable magnitudes of anisotropic forces of intermolecular end to end and lateral attractions by favorable molecular rigidity and flexibility which facilitate to float homologue molecules on the surface by a molecular arrangement, either as sliding layered type ordered (C_7 to C_{18}) or / and stastically parallel orientational order (C_4 to C_{18}) under exposed thermal vibrations for different range of temperatures, depending upon suitable magnitudes of thermal resistivity. N,N dimethyl para substituted and ortho substituted –NO_2 tail group of series-1 and –X broadens the molecular width while, mono atomic –Cl tail group in para position comparatively reduces intermolecular distance and two molecules of series-Y comes more closer as compared to series-1 and X, which strengthens the end to end attractions, and induces only nematic phase in series-Y with absence of smectogenic character. However, ortho substituted $–NO_2$ group and para substituted $–N(CH_3)_2$ group increase more molecular polarizability due to widening of molecular structure then –Cl. Thus, increased intermolecular attractions due to polarizability of series 1 and X including effect due to molecular polarity, induces smectogenic property in addition to nematogenic property. Broadening of a molecules of series-1 due to terminally substituted  group lowers the transition temperature to considerable extent as compared to the lowering due to ortho substituted $–NO_2$ group, because $–NO_2$ group is more polar and polarizable as compared to $–N(CH_3)_2$ group which strengthens intermolecular attractions of series-X through end to end attractions and induced polarizability by differing magnitudes of molecular broadening. –Cl tail group being monoatomic situated at para position is inducing intermolecular end to end attractions only but intermolecular attraction due to lower polarizability factor is comparatively less favorable. Thus, possibility of lamellar molecular packing in their (series-Y) crystal lattices is eliminated excluding smectogenic character and exhibits only nematogenic character of higher thermal stability than the nematic thermal stability of present novel series-1. Thus, smectic and nematic thermal stability of series-1 are lower than the series-X including only nematic thermal stability of series-Y, which is more than a series-1, excluding its destabilization of smectic mesophase. Total mesophaselength range suggest the resistivity towards exposed thermal vibrations to maintain smectic and/or nematic mesophase as related to thermal stability or the value of thermodynamical quantity enthalpy (ΔH). Early or late commencement of smectic or / and nematic mesophase depends upon the extent of molecular noncoplanarity. The molecules of series-X are more noncoplanar than other series under comparison. Hence, mesophases commences relatively earlier, whereas, the molecules of series-Y and 1 being more coplanar (para substituted) which facilitate late commencement of smectic and / or nematic phase as compared to series-X. Thus, changing magnitudes of mesomorphic properties for the same homologue from series to series depend upon flexibility of tail end group and from homologue to homologue in the same series depends upon the flexibility of left n-alkoxy group.

Conclusion

- Homologous series of present investigation is predominantly nematogenic and partly smectogenic of low thermal stability and of low melting type.
- Group efficiency order derived on the bases (i) thermal stability (ii) early commencement of mesophase and (iii) upper and lower mesophaselength range for smectic and nematic are as under in case of chalconyl ester series.

(i) Smectic
Ortho $–NO_2 > -N(CH_3)_2 > -Cl$
Nematic
Ortho $–NO_2 > -Cl > -N(CH_3)_2$
(ii) Smectic
Ortho $–NO_2 > -N(CH_3)_2 > -Cl$
Nematic
Ortho $–NO_2 > -N(CH_3)_2 > -Cl$
(iii) Total upper mesophase length

Ortho $-NO_2 > -N(CH3)_2 > -Cl$

- Mesomorphism is very sensitive and susceptible to a molecular structure.
- Novel chalconyl derivatives being biologically active molecules are antibacterial, antimalarial, antifungal, anticancer, which may be studied further in lyotropic condition by pharma and medicinal chemists.
- Novel thermotropic LCs may be useful for LC devices to be operated at lower or desired temperature through study of binary system.
- Present investigation supports and raises the credibility to the conclusions drawn earlier.

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