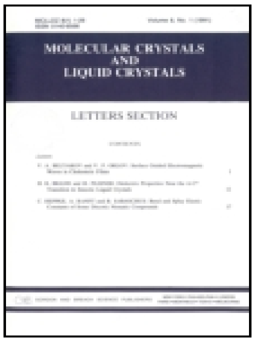

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Mesomorphism Dependence on Positional Substitution of Same Polar Group on Same Phenyl Ring in Isomeric Homologous Series

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Novel homologous series 4-(4′-n-alkoxy benzoyloxy) napthyl azo 2″ – nitro benzenes, consisted of eleven members of a series. All the eleven members (methoxy to tetradecyloxy) homologs are only enantiotropically nematogenic without exhibition of any smectogenic character. Transition temperatures and the textures are determined by an optical polarizing microscopy equipped with a heating stage. Textures of a nematic phase are threaded or schlieren. Analytical and spectral data supported the molecular structure of homologs. Transition curves viz., solid-nematic and nematic-isotropic showing phase behavior of the mesophase in a phase diagram behave in normal manner. Odd-even effect and alternation of transition temperatures are exhibited by N-I transition curve. N-I thermal stability is 129.7°C and Nematic mesophase vary from 12 to 64°C. Thus, novel series is entirely nematogenic and middle ordered melting type. Mesomorphic behavior of a novel series are compared with the structurally similar homologous series.

Keywords Anisotropic; liquid crystals; monotropy; nematic; smectic

Introduction

Since the birth of an unique liquid crystal state [1, 2] scientist and technologist have showed their keen interest in synthesizing novel liquid crystalline (LC) materials and exploited them in number of fields of applications all over the world including India. In India, Bangalore, Pune, Bomaby, Baroda-M.S.Uni., Punjab, and Assam were contributors to LC research. USA, UK, China, Pakistan, Korea, Japan, Russia, Germany, etc. were the pioneer contributors to LC study [3]. Saurashtra University-Rajkot-Gujarat has developed interest in LC study since last 30 years in combination with faculty of technology and engineering, The M.S. Uni. of Baroda-Vadodara. Dave et al. [4, 5] studied homologous series of novel Schiff's bases, carboxy ester, and their binary systems, Lohar et al. studied binary systems and LC ester homologous series of two phenyl rings [6, 7]. Vora et al. [8–10] studied cholesteric LC and conventional and unconventional LC, N.H. Shah and Jadav studied azoester [11] compounds, Doshi et al. studied ester azoester, chalcones, Schiff's bases [12–14] in homologous series and binary systems, Prajapati et al. studied dimers of novel

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varieties [15]. The chemists have synthesized novel LC compounds, evaluated their LC properties and characterized respective molecular structure of novel LC material. Thus, dependence of mesomorphism and the degree of mesomorphism on the molecular structure are mainly studied by Gray et al. [16–18]. Physicists and biologist studied physical properties and biological activity of LC compounds respectively. Technocrats and technologists applied their knowledge in the scientific pharmaceutical, medicinal and other allied fields of applications [19–21]. Present investigation is planned to synthesize novel azoester homologous series consisting of napthyl unit linking a phenyl ring through –COO- and other phenyl ring through –N=N- central bridges to which –OR/*n*-alkoxy terminal end group and at the other end ortho substituted –NO_2 polar group with respect to –N=N- group to be synthesized and characterized by analytical data. Then mesomorphic characteristics will be evaluated and compared with structurally isomeric series with –NO_2 group substituted at different positions of same phenyl ring. Thus study is aimed to understand and establish the effect of molecular structure on mesomorphism as a consequence of molecular rigidity and flexibility [22–24].

Synthesis

4-hydroxy benzoic acid was alkylated by suitable alkylating agents (R-X) to convert it into corresponding *n*-alkoxy benzoic acids by the modified method of Dave and Vora [25]. Azodye 4-hydroxy napthyl 2′-nitro benzene was prepared by usual established method (M.P is 134.0°C & Yield is 73.0%) [26]. 4-*n*-alkoxy benzoic acids and Azodye were individually condensed in ice cooled pyridine to get a series of final azoester products [27]. Final products were decomposed, filtered, washed, dried and purified till constant transition temperature, obtained.

The chemicals 4-hydroxy benzoic acid, alkyl halides [R-X], methanol, Ethanol, KOH, thionyl chloride pyridine, α – napthol, ortho nitro aniline, HCl, $NaNO_2$, etc. required for synthesis were used as received excepts solvents which were dried and distilled prior to use. The synthetic route to the series is mentioned in Scheme 1.

Characterization

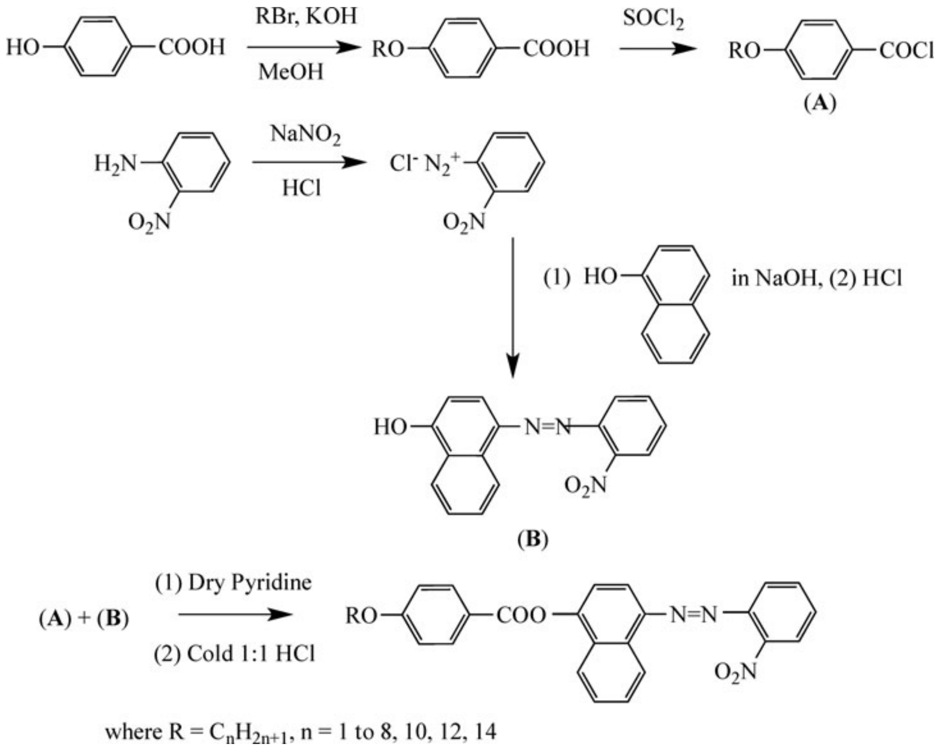
Some selected members of a novel series were characterized by elemental analysis, polarizing microscopy, mass spectra, Infra red spectra, ^{1}H-NMR spectra, Texture determination by miscibility method. Thermodynamic quantity enthalpy (δH) and entropy (δS) are qualitatively discussed instead of DSC scan. Microanalysis of the compound was performed on Perkin Elmer PE 2400 CHN analyzer, IR spectra were performed on Perkin Elmer spectrum, and ^{1}H-NMR spectra were performed on Bruker spectrometer using $CDCl^3$ as solvent (Table 1).

Analytical Data

IR Spectra in Cm^{-1}: for Ethoxy and Propyloxy derivatives:

Ethoxy: 2920, 2849, 1457 (alkyl group), 1733, 1684, 1201 (ester group), 1559, 1349 (Ar-NO_2), 1167 (C-N Str. due to –N=N-), 741(o-sub. benzene), and 849 (p-sub benzene).

Propyloxy: 2919, 2879, 1489 (alkyl group), 1733, 1683, 1207 (ester group), 1559, 1349 (Ar-NO_2), 1168 (C-N Str. due to –N=N-), 748 (o-sub. benzene), and 849 (p-sub benzene).



Scheme 1. Synthetic route of the series.

^{1}H- NMR Spectra in $CDCl_3$ in δ ppm: for Octyloxy and Dodecyl derivatives

Octyloxy: 0.89 (t, 3H, $-CH_3$), 1.27(m, 20H, $-CH_2$), 3.99 (t, 2H,$-OCH_2$), and 6.94-8.90 (m, 14H, Ar-H).

Dodecyl: 0.88(t, 3H, $-CH_3$), 1.27(m, 20H, $-CH_2$), 3.99 (t, 2H,$-OCH_2$), and 6.94-8.90 (m, 14H, Ar-H).

Mass spectra (molecular weight) Butyloxy homolog:
Calculated-469
Experimental-469

Textures by miscibility method
Nematic:
Pentyloxy homolog – Threaded texture
Tetradecyloxy homolog – Schlieren texture
Heptyloxy homolog – Threaded texture

Results and Discussion

Novel homologous series 4-(4′-n-alkoxy benzoyloxy) napthyl azo-2″-nitrobenzenes consisted of eleven members. All the members are enantiotropically nematogenic without exhibition of any smectogenic property. Dimerization of 4-*n*-alkoxy benzoic acids are disappeared on esterification process. Transition and melting points of homologs (Table 1, Table 2) as determined by an optical polarizing microscopy, equipped with a heating stage were plotted versus the number of carbon atoms present in *n*-alkyl chain of left *n*-alkoxy

Table 1. Elemental analysis for methoxy, butoxy, hexyloxy, and decyloxy derivatives

		Elements % found (calculated %)		
Sr. No.	Molecular formula	C	H	N
1	$C_{24}H_{17}N_3O_5$	69.88(69.90)	3.39(3.91)	9.56(9.80)
2	$C_{27}H_{23}N_3O_5$	68.88(69.08)	4.84(4.90)	8.89(8.95)
3	$C_{29}H_{27}N_3O_5$	70.11(70.02)	5.29(5.43)	8.35(8.45)
4	$C_{33}H_{35}N_3O_5$	71.44(71.60)	6.24(6.32)	7.36(7.59)

terminal end group of the molecules of each homolog. The like or related points were linked to draw the solid-nematic and nematic-isotropic transition curves to form a phase diagram (Fig. 1) showing the phase behavior of a novel series.

Solid-nematic transition curve initially rises from methoxy to hexyloxy homolog and then descended up to decyloxy homolog and again ascended to the last tetradecyloxy homolog derivative with overall descending tendency and adopting partly zigzag path of propagation. Nematic-Isotropic transition curve initially rises (odd members) and then descended from second member to the last member of a series with exhibition of odd-even effect. Thus, both of the transition curves behaved in normal established manner. N-I transition curves for odd and even members merge into each other at the nonyloxy homolog and then propagated into a single curve. The mesomorphic (only nematic) phase length vary between minimum of 12°C at the pentyloxy homolog and maximum of 64°C at the decyloxy homolog. Thermal stability for nematic mesophase is 129.7°C. Isotropic temperatures are ranging from 113 to 161°C. Mesogenic behaviors vary from homolog to homolog in the presently investigated azoester series.

Azodye 4-hydroxy napthyl azo 2′-nitro benzene is a nonliquid crystal (NLC) component, which on condensation with some nonliquid crystal or liquid crystal *n*-alkoxy

Table 2. Transition temperatures in °C for 4-(4’-*n*-alkoxy benzoyloxy)-napthyl azo-2”-nitro benzene

		Transition temperatures in °C		
Compound no.	*n*-alkyl group $C_n\ H_{2n+1}$ (*n*)	Sm	N	Isotropic
1	1	—	85.0	113.0
2	2	—	104.0	161.0
3	3	—	109.0	134.0
4	4	—	114.0	140.0
5	5	—	118.0	130.0
6	6	—	119.0	132.0
7	7	—	112.0	128.0
8	8	—	96.0	129.0
9	10	—	61.0	125.0
10	12	—	67.0	120.0
11	14	—	70.0	115.0

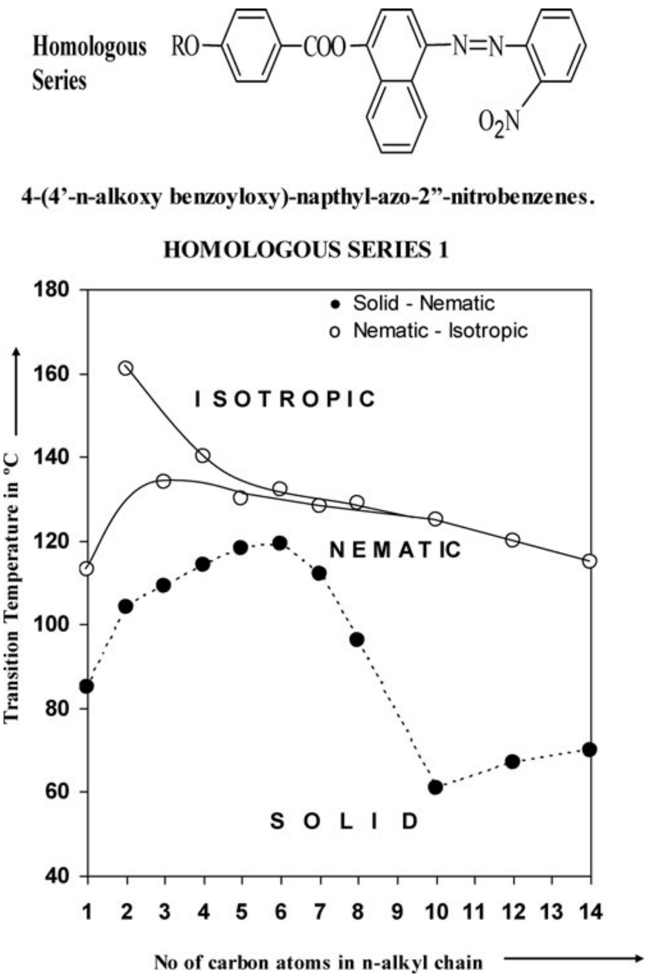


Figure 1. Phase behavior of series.

benzoic acids yielded nematogenic liquid crystal homologs of considerable range of liquid crystallinity, through their corresponding acid chlorides. Linking of two components through –COO- and –N=N- central bridges increases molecular length and breadth. Hence, molecular polarity and polarizability alter to suitable magnitudes of anisotropic forces of intermolecular attractions, which facilitate to induce nematogenic mesophase formation as a consequence of favorable molecular rigidity and flexibility. The disappearance of dimerization of 4-*n*-alkoxy benzoic acids is attributed to the breaking of hydrogen bonding between two molecules of 4-*n*-alkoxy benzoic acids by esterification process. Exhibition of odd-even effect and alternation of transition temperatures are attributed to the even and odd numbered carbon atoms present in *n*-alkyl chain of left n-alkoxy terminal end group. The exhibition of nematogenic mesophase formation by the all members of a novel series is due to the disalignment of the molecules of all homologs at an angle less than ninety degree with the surface of floating which, maintains the floating of the molecules of each homolog with statistically parallel orientational order for definite range of temperature under a hot stage polarizing microscope, corresponding to suitable magnitudes of anisotropic forces of intermolecular adhesion, as a consequence of molecular rigidity and flexibility or internal energy stored by a homolog concerned. Thus, the molecules of all the homologs of a present series are capable enough to resist exposed thermal vibrations for definite range of temperatures, depending upon their thermal withstanding capacity, related to thermal stability or molecular rigidity/flexibility. Absence of smectogenic character in a series indicates absence of lamellar packing of molecules in their crystal lattices due to the inadequate quantum of energy associated to build up two- or three-dimensional net work, which can float on the surface as sliding layers under the influence of exposed thermal vibrations. Thus, facilitating of lamellar packing of molecules is hindered by unsuitable magnitudes of anisotropic forces of intermolecular attractions, which causes disappearance of smectic mesophase formation till the last tetradecyloxy homolog. From and above the isotropic temperatures of each homolog of a series, the mesophase ceases to appear during microscopic observation, because the molecules are randomly oriented in all possible directions with high order of disorder or high entropy($\delta S = \delta H/T$). However, on cooling the same, the nematogenic mesophase formation reappears from and below isotropic temperature of a homolog concerned, till a sample of a substance undergoes solidification. Disappearance of odd-even effect from and beyond nonyloxy homolog is attributed to the coiling, bending, flexing, or coupling of *n*-alkyl chain with the major axis of a core structure. The mesogenic behaviors from homolog to homolog in the presently investigated novel azoester series are due to the sequential addition of methylene unit or units which, causes gradual variations in molecular rigidity and/or flexibility. Thus, the present series is entirely nematogenic whose range of liquid crystallinity is considerably good and of middle ordered melting type. Some liquid crystal behaviors of presently investigated homologous series 1 are compared with the structurally similar or ISOMERIC known homologous series X [28] and Y [29] as under in Fig. 2.

Homologous series 1, X and Y are isomeric to each other. All the three series under comparison are perfectly identical with respect to their rigid core consisted of two phenyl rings, one napthyl ring bonded through -COO- and –N=N- central bridges. The flexible part of all the comparative series are -O*R* and –NO_2 ($R = C_nH_{2n+1}$). Flexible group-O*R* varies with number of carbon atoms present in *n*-alkyl chain (n = 1,2,3,. . .) which contributes

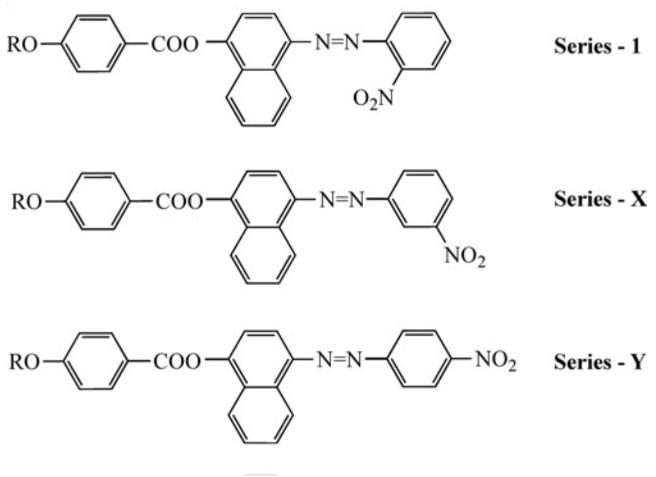


Figure 2. Structurally similar homologous series.

to the total molecular flexibility varying from homolog to homolog in the same series but, its contribution to flexibility remains identical for the same homolog from series to series. The flexibility contribution by polar –NO_2 group remains identical for the same series but it varies from series to series for the same homolog due to changing positional substitutional variation(ortho, meta, para to –N=N-) of –NO_2 polar group.

Thus, total molecular flexibility plus molecular rigidity constituting suitable magnitudes of anisotropic forces of intermolecular end to end attractions depending upon intermolecular closeness, molecular polarity, polarizability, and positional substitution of –NO_2 etc., vary from homolog to homolog in the same series as well as from series to series for the same homolog. Hence, a phenomenon of mesomorphism and the degree of mesomorphism being susceptible and sensitive to molecular structure or molecular rigidity-flexibility will differ with differing positions of –NO_2 at ortho, meta, or para position only in presently investigated novel series 1 and the series X and Y chosen for comparison respectively. Following Table 3 represents some mesogenic characteristics for the series 1, X and Y as under.

Table 3 indicates that the homologous series 1, X and Y under comparison are:

- Only nematogenic without exhibition of any smectogenic character.
- Nematogenic mesophase commences from very first member of each series but smectogenic mesophase does not commence till the last member of any of the series under comparison.
- Thermal stability for nematic, gradually increases by 6.48°C from ortho nitro to meta nitro, i.e., from series 1 to X, but, it, drastically increases 57.22°C in case of para substituted –NO_2 isomeric series Y.
- The degree of mesomorphism for all the three homologous series 1, X and Y under comparison are varied as maximum in case of series Y and minimum in case of series X whereas it is of intermittent valued for series 1 between the values of series X and Y.

An exhibition of mesomorphism by any molecule requires the suitable magnitudes of anisotropic forces of an intermolecular attraction as a consequence of favorable molecular rigidity and flexibility, as well as appropriate intermolecular closeness. Homologous series 1, X and Y possess one of the fused phenyl ring of napthyl unit as common lateral substitution. Homologous series 1 and X possess –NO_2 group occupying its position of substitution at ortho and meta places to –N=N- central bridge and also acts as lateral substitution. However –NO_2 group occupied at para position in series Y does not act as lateral substitution but acts as a terminal end group. Therefore, fused phenyl ring of napthyl unit is being commonly considered as lateral substitution to all the series under comparison which contributes to molecular polarizability factor in equivalent manner.

Table 3. Average thermal stabilities in °C

Series →	1	X	Y
Smectic-nematic or isotropic			
Commencement of smectic mesophase	—	—	—
Nematic-isotropic	129.7	136.18	193.4
commencement of nematic phase	$(C_1$-$C_{14})$ C_1	$(C_1$-$C_{14})$ C_1	$(C_1$-$C_{14})$ C_1

However, ortho- and meta-substituted –NO_2 group in series 1 and X adds some contribution to the molecular polarizability factor. But at the same –NO_2 substituted at para position in series Y relatively increases molecular length and decreases intermolecular distance. Hence, increase of intermolecular distance (or breadth) in case of series 1 and X can reduce the intermolecular attractions on one hand and the intermolecular attractions enhanced on other hand by increasing molecular polarizability. Thus, two opposing effects are operated at a same time for a same reason. The net magnitudes of intermolecular attractions will depend upon the predominancy of any one of the forces out of two opposing forces operating at a time for the same reason. Hence, the effect to the intermolecular attractions due to lateral substitution to the molecules under comparison is almost the equivalent irrespective of ortho or meta substituted -NO_2 molecules, but it largely affect to the molecules of para substituted -NO_2 of series Y, because, para substituted -NO_2 increases molecular length, length to breadth ratio and end to end intermolecular attractions comparatively more than ortho and meta substituted -NO_2 of series 1 and X. Thus increased intermolecular distance by lateral substitution predominates in by decreasing intermolecular attractions of series 1 and X and while it predominates in by increasing end to end attractions of series Y. Hence the thermal stability for nematic is the highest and drastically raised to 193.4 for series Y due to decreased intermolecular distance, but the same are lower 136.18 or 129.7 for series X and 1 due to combined effect of increased intermolecular distance and poor polarizability factor. Thus, nematic mesophase is thermally facilitated to stabilize relatively more in case of series Y than the series 1 and X under comparison. The commencement of nematogenic mesophase formation from very first member of each series 1, X and Y is obvious due to favorable molecular rigidity contributed by napthyl unit and flexibility contribution by highly polar –NO_2 and –OR groups. The extent of molecular noncoplanarity in case of all the series under comparison hinders the formation of lamellar packing of molecules in their crystal lattices, which does not allow to induce smectogenic character till the last homolog member of each series under comparative study.

Conclusions

- The group efficiency order for nematic derived on the basis of (i) thermal stability and (ii) the early commencement of nematic mesophase are as under.

  (i) Nematic:

$$\text{para} - NO_2 > \text{meta} - NO_2 > \text{ortho} - NO_2$$

  (ii) Nematic:

$$\text{ortho} - NO_2 = \text{meta} - NO_2 = \text{para} - NO_2$$

- A phenomenon of mesomorphism and the degree of mesomorphism in isomeric homologous series, bearing, same flexible polar groups, same central bridges and the same number of aromatic phenyl rings, depend upon the position (ortho, meta, para) of substitution of same polar group on the same phenyl ring.
- Transition temperature alternates with overall descending order.
- Homologous series with napthyl unit are generally nematogenic.
- Presently investigated novel homologous series is entirely nematogenic with relatively middle ordered melting type and of considerable rich degree of mesomorphism.

- A phenomenon of mesomorphism and the degree of mesomorphism are very sensitive and susceptible to the favorable magnitudes of molecular rigidity and/or flexibility or molecular structure.

Hence present investigation supported early views about mesomorphism and raised the credibility to the conclusions drawn earlier [18].

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