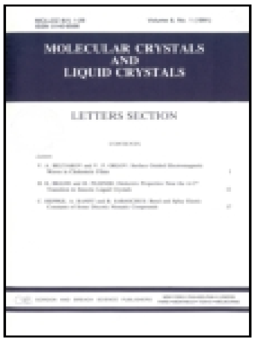

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Mesomorphism Dependence of Chalconyl Derivatives on Position of -CH=CH- Unit

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ABSTRACT

A novel homologous series of heterocyclic chalconyl esters (C_1 to C_{16}) has been synthesized and studied with a view to understanding and establishing the effects of molecular structure on liquid crystal (LC) properties and the degree of liquid crystallinity. The C_1 to C_6 homologues are not liquid crystals, the C_7 homologue is enantiotropic nematic, the C_8 to C_{16} homologues are smectogenic in addition to nematogenic (C_8 & C_{10} monotropic smectic). The texture of nematic phases are threaded or Schlieren and that of the smectic phase are fan-shaped smectic-A or smectic-C type. Transition temperatures and the textures of mesophases were determined by an optical polarizing microscope equipped with a heating stage. Transition curves Cr-I/M, Sm-N/N-Sm, and N-I showing phase behaviors of series in a phase diagram behave in normal manner. Thermal stability for smectic and nematic are 115.66°C and 126.83°C and their mesophase lengths minimum to maximum are 6.3 to 12.9°C and 6.9°C to 17.4°C, respectively. Analytical and spectral data confirms the molecular structures of homologues. LC properties of present novel series are compared with structurally similar other reported series.

KEY WORDS
Liquid crystals; mesomorphism; nematic; smectic

Introduction

The liquid crystal (LC) is an intermediate state of matter between a crystalline solid and an isotropic liquid The LC state has proved useful to mankind and has developed the interest of scientists and technologists all over the world irrespective of their subject of specialization [1]. This study of the LC state through novel chalconyl substances was planned to understand and establish the effects of molecular structure on thermotropic LC properties [2–5] with a view to providing novel substances to explore the thermometric properties and then biological activities by lyotropic properties. Thus, the proposed investigation can be useful in providing novel LC materials to other scientific and technological research communities [6–14]. Many homologous series have been reported to date [15–19]. The novel homologous series consists of two phenyl rings and one heterocyclic ring bonded through –COO–and–CH=CH–CO– central bridges and para substituted -OR terminal end group whose flexibility changes with n-alkoxy group; whose study of thermotropic LC properties are included in present investigation but their lyotropic study is for pharmaceutical and medicinal research. Some thermotropic LC properties of the novel homologous series will be compared, discussed and interpreted in terms of molecular rigidity and flexibility [20–24] of structurally similar isomeric series.

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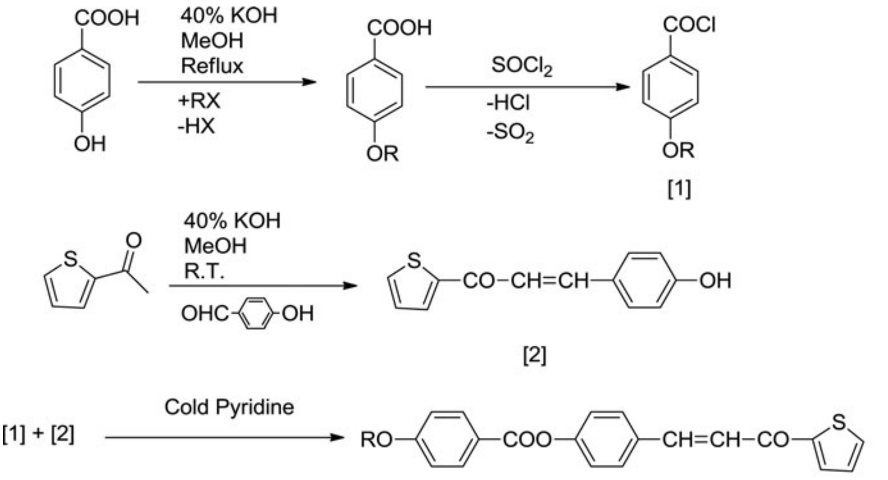

3-[4-(4'-n-alkoxy benzoyloxy) phenyl]-1-(thiophen-2"-yl) prop-2-en-1-one

where, $R=C_nH_{2n+1}$, n=1 to 8, 10, 12, 14 & 16

Scheme 1. Synthetic route to the novel series.

Experimental

Synthesis

4-Hydroxy benzoic acid was alkylated by suitable alkylating agents by the modified method of Dave and Vora [25]. 3-(4-Hydroxy phenyl) 1-(thiophene-2-yl) prop-2-en-1-one was prepared by a well-known method [26]. 4-n-Alkoxy benzoic acids were converted to corresponding acid chlorides using freshly distilled thionyl chloride and condensed with an ice cold solution of the chalcone in pyridine [27]. Final products were individually decomposed in 1:1 hydrochloric acid aqueous solution, filtered, washed, dried and purified until a homologue gave constant transition temperatures.

4-Hydroxy benzoic acid, alkyl halides, KOH, thionyl chloride, methanol, ethanol, pyridine, 4-hydroxy benzaldehyde, 2-acetyl thiophene etc. were used as received except solvents which were dried and purified prior to use. The synthetic route of the novel homologous series is shown in Scheme 1.

Table 1. Elemental analysis for (1) Propyloxy (2) Heptyloxy (3) Decyloxy derivatives.

		Elements%Found			Elements% Calculated		
Sr.No.	Molecular formula	C	H	O	C	H	O
1	$C_{23}H_{20}O_4S$	70.45	5.11	16.28	70.39	5.14	16.31
2	$C_{27}H_{28}O_4S$	72.36	6.25	14.24	72.29	6.29	14.27
3	$C_{30}H_{34}O_4S$	73.41	6.96	13.09	73.44	6.98	13.04

Characterization

Representative members of the novel series were characterized by elemental analysis (Table 1), Infrared spectroscopy, 1HNMR spectroscopy, and mass spectroscopy. Microanalysis was performed on Perkin-Elmer PE 2400 CHN analyzer. IR spectra were recorded on Shimadzu FTIR-8400, ^{1}H NMR spectra were recorded on Bruker spectrometer using $CDCl_3$ as solvent and mass spectra were recorded on Shimadzu GC-MS Model No.QP-2010. The mesogenic behavior of the homologue derivatives were observed through polarizing microscopy. The textures of nematogenic mesophase were determined by miscibility method.

Analytical data

Spectral data

^{1}H NMR in δ ppm for ethyloxy derivative

1.44–1.48 (t, 3H,-CH_3 of -OC_2H_5 group), 4.00–4.15 (m, 2H, Ar-O-$\underline{CH_2}$), 7.18–7.20 & 8.13–8.15 (4H of Phenyl ring with *n*-propoxy group), 7.38–7.42 & 7.84–7.88 (4H of middle Phenyl ring), 7.26–7.29 & 8.13–8.15 (3H of thiophene ring with chloro group) 7.68–7.71 (d, 1H, $-CO-\underline{CH}{=}CH-$), 6.96–6.98 (d, 1H, $-CO-CH{=}\underline{CH}-$). The NMR data are consistent with the molecular structure.

^{1}H NMR in δ ppm for pentyloxy derivative

0.94–0.96 (t, 3H,-CH_3 of $-OC_5H_{11}$ group), 1.34–1.36 (m, 2H, Ar-O-CH_2-CH_2-CH_2-$\underline{CH_2}$), 1.38–1.49 (m, 2H, Ar-O-CH_2-CH_2-$\underline{CH_2}$), 1.77–1.84 (m, 2H, Ar-O-CH_2-$\underline{CH_2}$), 4.00–4.03 (m, 2H, Ar-O-$\underline{CH_2}$), 7.15–7.17 & 8.14–8.16 (4H of Phenyl ring with n-propoxy group), 7.37–7.41 & 7.82–7.87 (4H of middle Phenyl ring), 7.25–7.87 (3H of thiophene ring with chloro group), 7.66–7.69 (d, 1H, $-CO-\underline{CH}{=}CH-$), 6.95–6.97 (d, 1H, $-CO-CH{=}\underline{CH}-$). The NMR data are consistent with the molecular structure.

IR in cm^{-1} for methyloxy derivative

3078 (= C-H Str. of Phenyl nucleus), 2931 & 2846 (CH_2 sym. & asym. stra.), 1720 (Aromatic C = O Str.), 1643 (aromatic C=C str.), 1512 (C-C aromatic str.), 1465 (C–H bend alkanes), 1327 (aromatic& aliphatic C-O str.), 1157 (Ar-O str.), 840 (C-H oop. phenyl ring), 725 & 686 (C-H oop.bending of phenyl ring). The IR data are consistent with the molecular structure.

IR in cm^{-1} for hexyloxy derivative

3070 (=C–H Str. of Phenyl nucleus), 2954 & 2854 (CH_2 sym. & asym. stra.), 1720 (Aromatic C=O Str.), 1643 (aromatic C=C str.), 1512 (C–C aromatic str.), 1465 (C–H bend alkanes), 1327 (aromatic& aliphatic C–O str.), 1165 (Ar-O str.), 848 (C–H oop. phenyl ring), 709 & 694 (C=H oop.bending of phenyl ring). The IR data are consistent with the molecular structure.

Mass of octyloxy derivative

m/z (rel.int%): 462 (M)+, 233, 201, 121, 111, 93, 57, 43

Mass of dodecyloxy derivative

m/z (rel.int%): 518 (M)+, 317, 289, 201, 191, 121, 111, 93, 57, 43

Table 2. Transition temperatures in°C.

		Transition temperatures in°C		
Compound No.	R = n-alkyl chain C_nH_{2n+1}	Sm	N	Isotropic
1	1	—	—	151.8
2	2	—	—	150.0
3	3	—	—	140.4
4	4	—	—	154.3
5	5	—	—	141.8
6	6	—	—	128.5
7	7	—	114.7	121.6
8	8	(94.8)	116.0	125.6
9	10	(104.9)	111.8	129.2
10	12	107.7	114.0	126.5
11	14	106.5	119.4	133.3
12	16	102.4	113.6	124.8

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

Results and discussion

3-(4-Hydroxy phenyl) 1-(thiophene-2-yl) prop-2-en-1-one (m.p-128–130°C; yield- 68.50%) is a nonliquid crystalline component, which on condensing it with dimeric 4-n-alkoxy benzoic acids yielded targeted molecules of chalconyl ester heterocyclic derivatives. Dimerization of *n*-alkoxy benzoic acids disappears on esterification process through the corresponding acid chloride. Transition temperatures (Table 2) were plotted versus the number of carbon atoms present in *n*-alkyl chain bonded to first phenyl ring through oxygen atom. Smooth curves are drawn through like or related points to show the phase behavior of the series in a phase diagram (Fig. 1). Cr-I/M transition curve partly adopts a zigzag path of rising and falling with overall descending tendency. Sm-N, or N-Sm transition curve initially rises from C_8 homologue, then it passed through a maxima at C_{14} homologue and finally descended to C_{16} in usual expected manner. Hence Cr-I/M and Sm-N/N-Sm transition curves behaved in normal

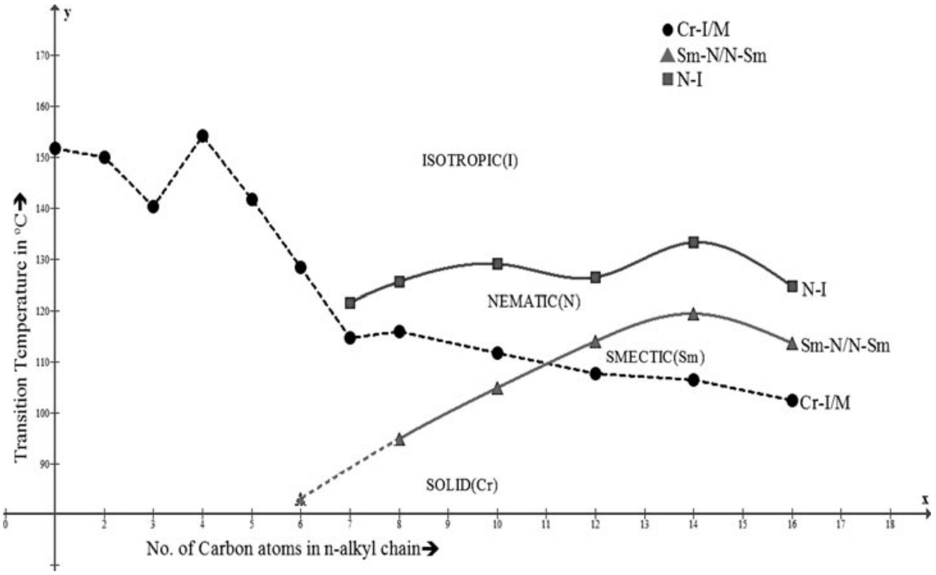

Figure 1. Phase Behavior of Series.

manner. Sm-N Curves behaved in normal manner. Sm-N curve is extrapolated to C_6 homologue to predict its latent transition temperature (L.T.T.) for smectic (83°C). N- I transition curve initially rises from C_7 to C_{14} and then descended, as series is ascended. Thus, N-I transition curve behaved in normal manner except negligible derivation at C_{14}. Odd–even effect in the transition curves of phase diagram is completely absent. The mesomorphic properties and mesophase behaviors from homologue to homologue in the present novel series undergo variations. The smectic and nematic thermal stabilities are 115.66°C and 126.83°C respectively, whose total mesophase length (Sm+N) very between 6.9°C and 26.8°C at the C_7 and C_{14} homologues, respectively. Smectic and nematic mesophases commences from C_8 and C_7 homologues respectively. Hence a novel chalconyl ester homologous series under discussion is party nematogenic and party smectogenic of middle ordered melting type and of low degree of mesomorphism.

Disappearance of dimerization of *n*-alkoxy benzoic acids is attributed to the breaking of hydrogen bonding between acid molecules by the esterification process through their corresponding acid chlorides. The nonmesomorphicity of C_1 to C_6 homologues is attributed to their high crystallization tendency with arises from the low diploe–dipole interaction and the low magnitudes of dispersion forces by the interaction between instantaneous dipole produced by the spontaneous oscillations of electron clouds of the molecules C_1 to C_6 which induces unsuitable magnitudes of anisotropic forces of end to end and lateral attractions as consequence of unfavorable molecular rigidity and flexibility including abrupt breaking of crystal lattices and sharply transform into isotropic state without passing through LC state at Cr-I transition temperature. As series is ascended by progressive addition of methylene unit the molecular length, length to breadth ratio, polarity and polarizibility, permanent dipolemoment across, the long molecular axis, dispersion forces, dipole-dipole and electronic interactions, magnitudes of end to end and lateral attractions etc. alters and create the favorable environment to include mesophase formation by disalignment of molecules at an angle ninety or less than ninety degree and resists the exposed thermal vibration. Hence C_8 to C_{16} homologue molecules arrange themselves in sliding layered arrangement on reversible heating (C_{12} to C_{16}) or irreversibly (C_8 and C_{10}) on coiling due to preoccupied lamellar packing of molecules in their crystal lattices of rigid crystals below N-I transition temperature under floating condition to exhibit smectic mesophase formation. C_7 to C_{16} homologue molecules resist or extend to resist above Cr-Sm or Cr-N transition temperatures; arrange themselves to occupy statistically parallel orientational order of molecules in floating condition to cause formation of nematic phase. However, molecules of C_7 to C_{16} and C_1 to C_6 are randomly oriented in all possible directions with high order of disorder or high entropy ($\Delta S = \Delta H/T$) from and beyond isotropic temperature. The isotropic mass on careful cooling, the C_1 to C_6 homologues solidified to crystalline state, C_7 homologues solidified to crystalline state after acquiring nematogenic phase from and below isotropic temperature, C_8 and C_{10} acquired reversible nematic phase and then irreversibly or monotropically smectic phase and then solidified to crystalline state below isotropic temperature. Homologues C_{12} to C_{16} molecules acquired on coiling solid crystalline state in reversible manner below isotropic temperature i.e. the smectic as well as nematic mesophases persisted exactly at the same transition temperatures at which they were appeared on heating i.e. Sm-N and N-I transition temperatures are enantiotropic and reversible for C_{12} to C_{16} homologues. Absence of odd–even effect in Sm-N and N-I transition curves is due to the late commencement of mesophase formation (C_7 or C_8) for longer *n*-alkyl chain, whose status of *n*-alkyl chain possess uncertainty by coiling bending, flexing or coupling with major axis of the core structure. The changing trend in mesomorphic behaviors from homologue to homologue in the present series is attributed to the sequential addition of

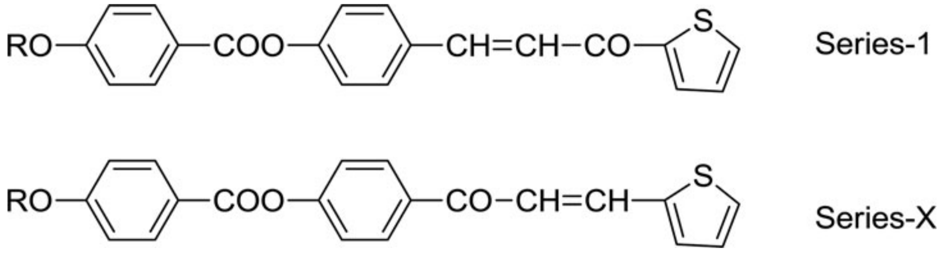

Figure 2. Structurally similar series.

methylene unit or units to the *n*-alkyl chain of the left *n*-alkoxy terminal end group, keeping heterocyclic thiophene ring intact.

The LC behaviors, properties and the degree of mesomorphism which varies from homologue to homologue in the same series and for the same homologue from series to series are compared with an isomerism series-X [28] as under in Figure 2.

Homologous series-1 of present investigation and an isomeric series-X chosen for comparative study are identical with respect to two phenyl rings bonded through same -COO- central bridge heterocyclic thiophene tail end group but they differ from each other with respect to other central bridges viz. –CH=CH–CO and –CO–CH=CH– linking middle phenyl ring and five-membered sulpher containing heterocyclic ring. However series-I and X can be compared for the same homologue from series to series, depending upon the positional difference or displacement of –CO– and/or –CH=CH– of central bridge linking phenyl ring and heterocyclic tail ring. Thus the LC properties and the degree of mesomorphism of present novel series-1 and an isomeric series-X as chosen for comparison can be compared on the basis of differing features of uncommon central bridge other than a common central bridge other than a common central bridge –COO– for the same homologue. Some LC properties of series-1 and X are recorded in Table 3.

From Table 3 it is clear that,

- Present homologues series-1 is smectogenic in addition to nematogenic, whereas series-X under comparative study is only smectogenic.
- Thermal stabilities for smectic of series-1 and X are nearly equivalent because it is higher by 2.09 unit from series-X (115.66°C–113.57°C).
- Nematic thermal stability of series-1 is 126.83°C but nematic mesophase does not stabilize till the lost homologue of series-X.
- Smectic and nematic mesophases commences from C_8 and C_7 homologues of a series-1, where as nematic mesophase it totally absent and smectic mesophase commence late from C_{10} member of a series-X under comparison.

Table 3. Average thermal stabilities in°C.

Series	1 (–CH=CH–CO–)	X (–CO–CH=CH–)
Smectic-Isotropic or	115.66	113.57
Smectic-Nematic	(C_{12}-C_{16})	(C_{10}-C_{16})
Commencement of smectic phase	C_8	C_{10}
Nematic-Isotropic commencement of nematic phase	126.86	—
	(C_7-C_{16})	—
	C_7	
Total Mesophase length range in°C (Sm+N)	6.9 – 26.8	4.1 - 11.1
	C_7 C_{14}	C_{16} C_{10}

- Mesophase length (Sm+N) range of present series-1 is more than a series-X.
- Odd–even effect is absent in transition curves of both series-1 and series-X under comparative study.

Homologous series -1 of present investigation and an isomeric series -X of selected for comparative study resembles each other except positional replacement of –CO– or –CH=CH–. The sp^2 carbon of –CH=CH–bonded to sp^2 carbon of phenyl ring or –CO– group of uncommon central bridge is bonded to heterocyclic ring in the molecules of series-1 whereas –CH=CH– and –CO– groups are conversely bonded to heterocyclic ring and to the phenyl ring respectively. The first alternative and second alternative of the position of –CO– and –CH=CH– are almost equally competent to induce and stabilize smectogenic property of nearly equal magnitudes of thermal stability (115.66°C and 113.57°C) due to the suitable magnitudes of inter molecular lateral attractions whose layers are shielded out from the lattices of rigid crystals under the influence of exposed thermal vibrations. However, the molecules of series-1 due to the –CH=CH – bonded to middle phenyl ring (or –CO– group bonded to hetero cyclic ring) strengthens the end to end attractions of such a suitable magnitudes that layers under floating condition arranges or acquires statistically parallel orientational molecular ordered arrangement subsequent to smectic to induce nematic phase at Sm-N or Cr-N/I-N temperature. The end to end intermolecular attractions of series-X of second alternative are weaker as compared to series-1 of first alternative of chalconyl group which fails to maintain or emerge or stabilize nematic phase formation. Thus, magnitudes of molecular rigidity differs (ΔH) due to replacement of –CO– or –CH=CH– plays the role of mesomorphism. Thus, nematogenic mesophase stabilized beyond smectogenic phase in series-1 but nematogenic mesophase formation does not stabilize beyond smectogenic mesophase in isomeric series-X. The commencement of mesophase early or late depends upon the extent of nonplanarity of a molecule. The bonding of –CH=CH– unit directly at the middle phenyl ring strengthens the molecular rigidity comparatively more in series-1 than, if it (–CH=CH–) is bonded through –CO– group with middle phenyl ring, which reduces the extent of noncoplanarity of molecules and favors layered arrangement of molecules and favorable end to end attraction more efficiently in the preoccupied lamellar molecular packing in crystal lattices of rigid crystal and in sliding layered arrangement under heating condition. Therefore smectic mesophase formation take place earlier from C_8 homologue in series-1 as compared to isomeric series-X, in which smectic mesophase commences late from C_{10} homologue. Moreover reduction in the extent of molecular non co-planarity by bonding –CH=CH– unit directly with middle phenyl ring favors nematic mesophase formation beyond smectic phase; whereas bonding of –CH=CH– unit through –CO– group with middle phenyl ring and the resulted cohesive forces hinders or are unable to induce nematic phase beyond smectic phase under exposed thermal vibrations while floating on the surface as observed in series-X. The exhibition of total mesophase formation for the same homologue from series to series is related with the intermolecular cohesive energy associated with individual molecules for the same homologue from series to series and from homologue to homologue in the same series i.e. thermal resistivity towards exposed thermal vibration of different suitable magnitudes vary from homologue to homologue or in general like or unlike substance to substance. Hence, variation in mesophase length range from minimum to maximum varies from series-1 to isomeric series-X under comparison. The absence of odd- even effect for series-1 and X are attributed to the late commencement of mesophases or due to the absence of mesophase form C_1 to C_6 or C_1 to C_8 homologue molecules in series-1 and X, respectively.

Conclusions

- Presently investigated novel chalconyl ester series-1 is partly smectogenic and partly nematogenic with middle-ordered melting type.
- The group efficiency order derived on the basis of
- (a)Thermal stability (b) early commencement of mesophase (c) mesophase length range for smectic and nematic in isomeric series are as under.
    - (a) Smectic : –CH=CH–CO– > –CO–CH=CH–
      Nematic: –CH=CH–CO– > –CO–CH=CH–
    - (b) Smectic : –CH=CH–CO– > –CO–CH=CH–
      Nematic: –CH=CH–CO– > –CO–CH=CH–
    - (c) Sm+N: –CH=CH–CO– > –CO–CH=CH–
- –CH=CH–CO– central bridge is more efficient to from mesophase or mesogenic phase than an isomeric chalconyl central group –CO–CH=CH–.
- The bonding of –CH=CH– group directly to middle phenyl ring is more efficient to induce mesophase or mesophase of higher phase length and earlier commencement of mesophase as compared to the linking of –CH=CH– group through –CO– with middle phenyl ring.
- Phenomena of mesomorphism are very sensitive and susceptible to molecular structure.
- Chalconyl ester homologues of present investigation are useful for the study of binary systems to prepare articles workable at required temperature. Chaloconyl derivative are being biologically active molecules related to anticancer, antimalarial, antibacterial, anti-dermitological etc. may be studied by the researchers working for pharmaceutical and medicinal fields.

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