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Prospects of the Development of New Approaches of the Preparation of Anisotropic Compounds and Materials

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New synthetic approaches for the preparation of various anisotropic and liquid-crystalline compounds with different combinations of cyclic, bridge, terminal fragments and lateral substituents are proposed and realized. Both chiral and nonchiral nematic, smectic mesomorphic and anisotropic compounds were prepared by the transformations of the corresponding 3,6-disubstituted cyclohex-2-enones, 2,5-disubstituted cyclohexanones, 5-substituted cyclohexan-1,3-diones, 1,2-disubstituted cyclopropanoles and 3,5-disubstituted 2-isoxazolines, substituted cyclohex-2-enonyl-2-isoxazolines.

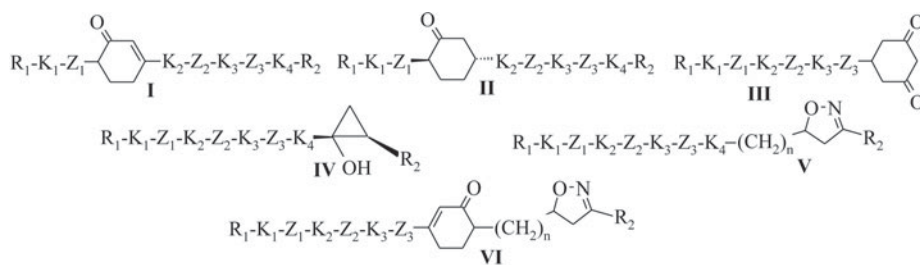
Keywords anisotropic compounds; liquid crystals; intermediates for anisotropic compounds and liquid crystals; synthesis; properties

1. Introduction

The synthetic routes for the preparation of liquid-crystalline compounds are usually multistage and need low temperatures or an inert atmosphere, palladium-catalyzed coupling of arylboronic acids or Grignard coupling of 4-substituted cyclohexanones or 4-substituted halogenarenes [1–11]. Additionally, the methods for the preparation of Grignard reagents, arylboronic acids from 4-bromo-1-(*trans*-4-alkylcyclohexyl)benzenes or 4-bromo-4-substituted biphenyls and 4-(4-substituted phenyl)cyclohexanones and 4-(*trans*-4-substituted cyclohexyl)cyclohexanones from the corresponding 4-substituted benzylmethylketones, 4-substituted benzylcyanides, 1,4-cyclohexanedione, or from the adducts of the condensation of 2-chloro(ethoxy, formyloxy, trimethylsilyloxy)-1,3-butadienes with 4-substituted styrenes, or from the products of Birch reduction of benzenes are usually complicated and are characterized by a low yield of the products [12–15].

In this contribution the research efforts on the intermediates, synthetic routes of the preparation of various anisotropic and liquid-crystalline compounds having different practical applications are reviewed. The results are summarized here, emphasizing structure-

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$R_{1,2}$ = alkyl or alkoxy fragments, F, Cl, CN, CF_3 , OCF_3 or chiral fragment. K_{1-4} = benzene, cyclohexane, or cyclohexene rings; n = 0-5; Z_{1-3} = single bond or CH_2CH_2 , or other bridge fragments

Figure 1. The intermediates (I–VI) for the preparation of liquid-crystalline and anisotropic compounds.

property relations and the properties of the compounds compared with structurally related homologous series.

It has been reported that the mesomorphic derivatives of benzene, cyclohexane, biphenyl, or terphenyl, which are useful components of LC mixtures for display applications, can be easily prepared in a high yield from the corresponding 3,6-disubstituted cyclohex-2-enones [17–22]. Continuing our interest in the intermediates for the preparation of liquid-crystalline and anisotropic compounds, we summarize and show here how the advanced both nonchiral and chiral nematic, smectic mesomorphic and other anisotropic compounds can be synthesized by the transformations of the available 3,6-disubstituted cyclohex-2-enones (I), 2,5-disubstituted cyclohexanones (II), 5-substituted cyclohexan-1,3-diones (III), 1,2-disubstituted cyclopropanols (IV) and 3,5-disubstituted 2-isoxazolines, substituted cyclohex-2-enonyl-2-isoxazolines (VI) (Figure 1).

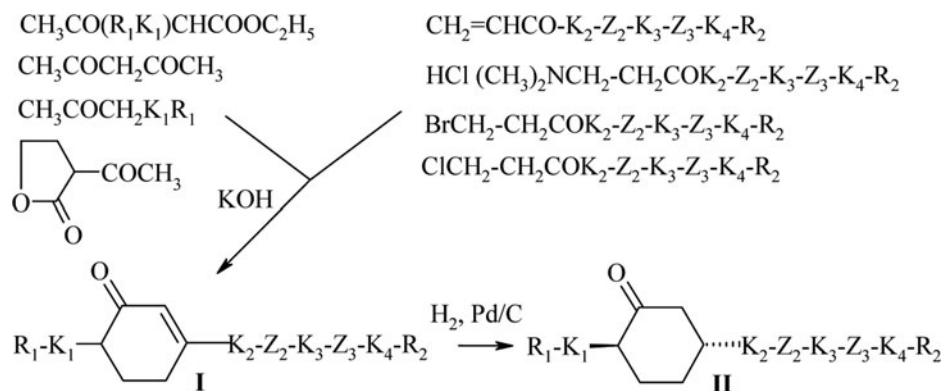
2. Results and Discussion

2.1. Synthesis of the intermediates

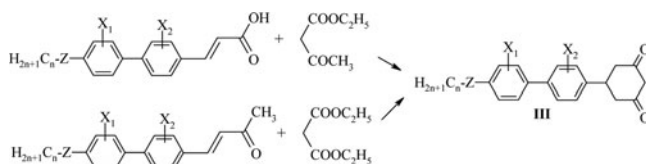
The 3,6-disubstituted cyclohex-2-enones (I) with different combinations of cyclic, bridge, terminal fragments and lateral substituents can be prepared in a high yield and in a “one pot procedure” by the condensation of the corresponding Mannich salts or 2-chloro(bromo)ethylaryl(alkyl)ketones with 2-substituted acetoacetic esters, 4-substituted methylbenzylketones or other β -dicarbonylic compounds in the presence of potassium hydroxide in a boiling dioxane [23]. The following catalytic hydrogenation of the cyclohex-2-enones (I) in the presence of 10% palladium on charcoal and potassium hydroxide in isopropanol or THF-isopropanol mixture leads to the appropriate *trans*-2,5-disubstituted cyclohexanones (II) (Scheme 1).

The 5-substituted cyclohexan-1,3-diones (III) are formed by the condensation of substituted cinnamic acids or benzylideneacetones with esters of acetoacetic or malonic acids [24] (Scheme 2).

The reaction of carboxylic esters with ethylmagnesium bromide or higher homologues, or with ethylmagnesium bromide and excess of alkenes in the presence of titanium tetraisopropoxide of Scheme 3 leads to the corresponding 1,2-disubstituted cyclopropanols (IV) [25, 26].



Scheme 1. Preparation of the 3,6-disubstituted cyclohex-2-enones (I) and 2,5-disubstituted cyclohexanones (II).

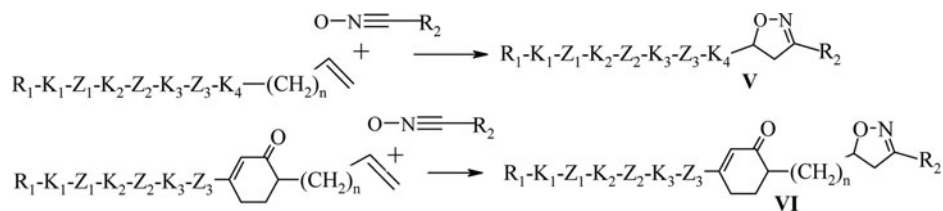


Scheme 2. Preparation of the 5-substituted cyclohexan-1,3-diones (III).



Scheme 3. Preparation of the 1,2-disubstituted cyclopropanols (IV).

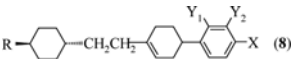
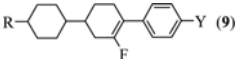
The 1,3-dipolar cycloaddition of nitrile oxides to 1-alkenes or 4-substituted styrenes in the presence of N-chlorosuccinimide and triethylamine has proved to be an useful strategy for the preparation of 3,5-disubstituted 2-isoxazolines (V) and substituted cyclohex-2-enonyl-2-isoxazolines (VI) [27, 28] (Scheme 4).



Scheme 4. Preparation of the 3,5-disubstituted 2-isoxazolines (V) and substituted cyclohex-2-enonyl-2-isoxazolines (VI).

It was found [29] that 3,6-disubstituted cyclohex-2-enones (I), 2,5-disubstituted cyclohexanones (II), 5-substituted cyclohexan-1,3-diones (III), 1,2-disubstituted cyclopropanols (IV) and 3,5-disubstituted 2-isoxazolines (V) are really promising

Table 1. Transition temperatures [$^{\circ}\text{C}$] of cyclohexenes (**8**), fluorocyclohexenes (**9**)

							
R	Y ₁	Y ₂	X	T, $^{\circ}\text{C}$	R	Y	T, $^{\circ}\text{C}$
C ₃ H ₇	H	H	F	Cr 27 N 78 Iso	C ₃ H ₇	F	Cr 33 N 122 Iso
C ₄ H ₉	H	F	F	Cr <0 N 59 Iso	C ₃ H ₇	OCH ₃	Cr 56 N 188 Iso
C ₄ H ₉	F	H	OCH ₃	Cr <0 N 90 Iso	C ₅ H ₁₁	F	Cr 34 N 129 Iso
C ₅ H ₁₁	H	H	F	Cr 25 N 72 Iso	C ₅ H ₁₁	OCH ₃	Cr 54 N 188 Iso

intermediates for the preparation of various anisotropic and liquid-crystalline compounds with different combinations of cyclic, bridge, terminal fragments and lateral substituents, which are in turn useful components for liquid crystalline mixtures for display and other practical applications. Both chiral and nonchiral nematic and smectic liquid-crystalline or anisotropic compounds can be easily prepared by the transformations of cyclic fragments and functional groups located in the molecules. It should be noted that different reaction possibilities for the functional groups and cyclohexenone, cyclohexanone, cyclohexan-1,3-diones, isoxazoline or cyclopropanol fragments allow transformations to be achieved selectively and give the opportunity of preparing mesomorphic and anisotropic compounds with novel combinations of the structural fragments of molecules.

2.2. Synthesis of liquid-crystalline and anisotropic compounds

From lots of the 3,6-disubstituted cyclohex-2-enones (**I**), *trans*-2,5-disubstituted cyclohexanones (**II**) and their derivatives, which have been synthesized, we have to describe the following:

(A). Substituted cyclohexenes, fluorocyclohexenes and fluorocyclohexanes (**1-7**), which are characterized low optical anisotropy and viscosity, and temperature formation of the nematic phase (Scheme 5, Table 1) [30–33].

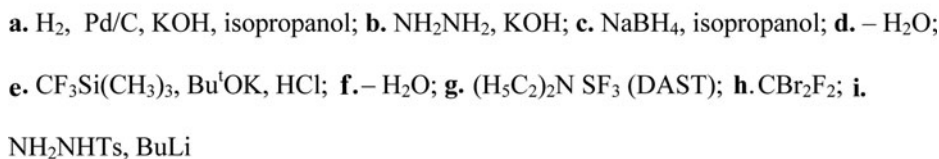
(B). Lateral substituted terphenyls, quaterphenyls and pentaphenyls (**10-17**), which are easily formed in a one-stage procedure from the corresponding 3,6-disubstituted cyclohex-2-enones (**1,2**) (Scheme 6) [34, 35].

These compounds are useful in LC mixtures because they have low melting points and wide temperature ranges of their nematic and smectic phases, excellent solubility in mesogenic media, and a high optical anisotropy (Figure 2).

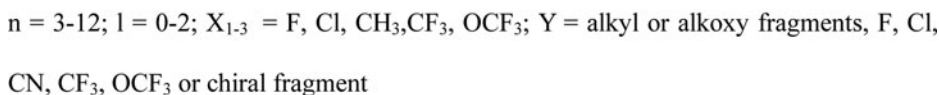
(C). Liquid-crystalline compounds (**26, 28-30**) with positive dielectric anisotropy ($+\Delta\epsilon$) similar to presented on Scheme 7 [36].

It has been found that these compounds (Table 2) and similar to them compounds can be successfully used for the preparation of LC mixtures with low threshold and saturation voltages of the twist-effect, steep voltage-contract characteristics, and fast response times.

(D). Liquid-crystalline compounds with negative dielectric ($-\Delta\epsilon$) anisotropy, which can be prepared from the corresponding 3,6-disubstituted cyclohex-2-enones (**I**) and 2,5-disubstituted cyclohexanones (**II**) in the aromatization of cyclohexenone or modifications of cyclohexanone fragments (Scheme 8) [31, 37].



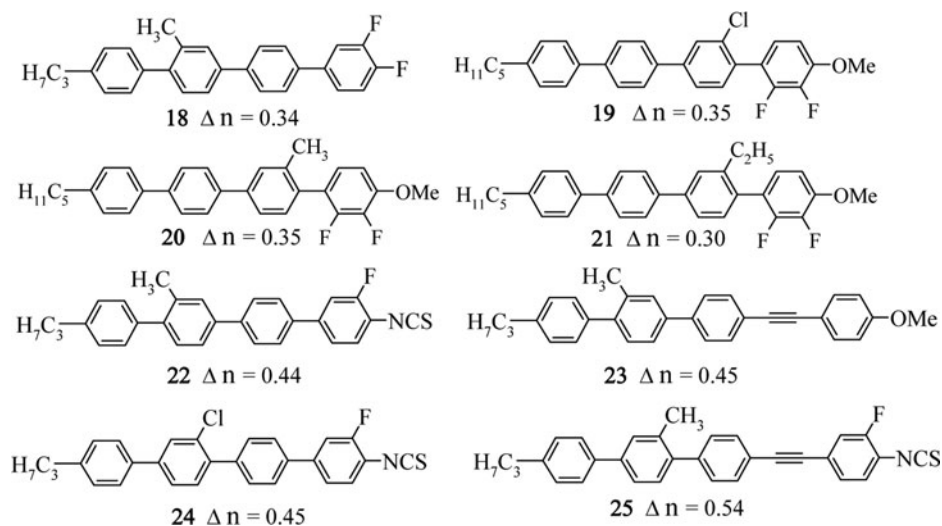
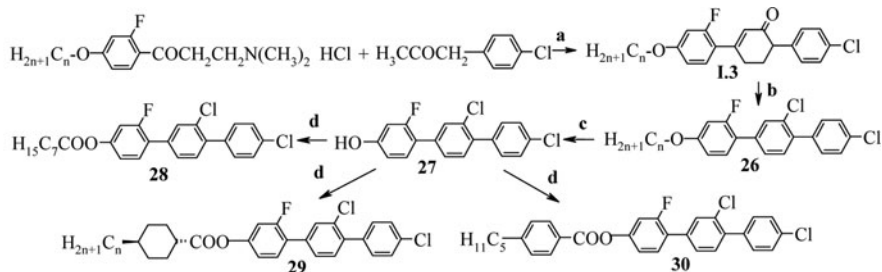
Scheme 5. Preparation of the substituted cyclohexenes, fluorocyclohexanes (**1-7**)



Scheme 6. Preparation of the terphenyls, quaterphenyls and pentaphenyls (**11-17**).

Investigations of the electro-optic and dynamic parameters of LC mixtures containing compounds (**37-44**) presented in Table 3 have shown that they are really promising components of the materials for VA displays.

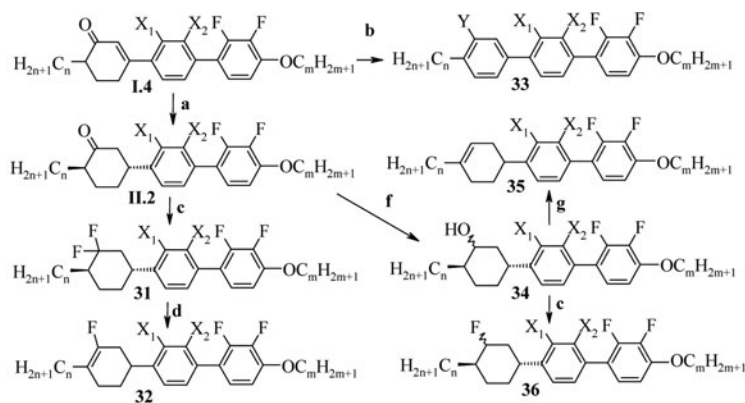
These **(37-44)** and similar to them polyfluoroderivatives allow the threshold voltage of the liquid-crystalline mixtures to decrease. Using the polyfluoroderivatives we have developed the LC compositions with low the threshold voltage, low values of switch-on

**Figure 2.** Derivatives of terphenyls and quaterphenyl (18-25).

$n = 5-12$; **a.** KOH, dioxane; **b.** PCl_5 ; **c.** HI, CH_3COOH ; **d.** RCOCl , Py

Scheme 7. Preparation of LC compounds (26, 28-30)**Table 2.** Transition temperatures [$^{\circ}\text{C}$] of LC compounds (26, 28-30) with positive dielectric anisotropy

R	T, $^{\circ}\text{C}$	R	K	T, $^{\circ}\text{C}$
C_8H_{17}	Cr 52 N 72 Iso	C_5H_{11}		Cr 133 N 233 Iso
C_{12}H_9	Cr 34 N 67 Iso	C_5H_{11}		Cr 79 N 269 Iso
$\text{C}_7\text{H}_{15}\text{CO}$	Cr 59 N 75 Iso	C_7H_{15}		Cr 63 N 240 Iso



$n = 3-7$; $m = 1-3$; $X_{1,2} = H, F$; $Y = F, Cl, CF_3$; **a.** H_2 , Pd/C ; **b.** PCl_5 $[CF_3Si(CH_3)_3]$ and etc.]; **c.** $(H_5C_2)_2N SF_3$; **d.** KOH ; **f.** $NaBH_4$; **g.** $-H_2O$;

Scheme 8. Preparation of LC compounds (**31-36**)

Table 3. Transition temperatures [$^{\circ}C$] of LC compounds (**37-44**) with negative dielectric anisotropy

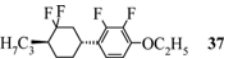
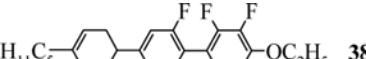
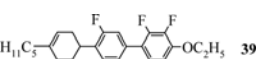
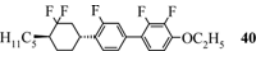
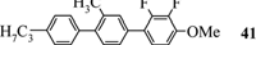
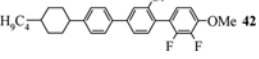
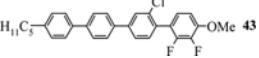
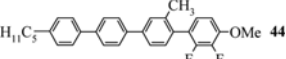
Compound	T, $^{\circ}C$	Extrapolated data		
		Δn	$\Delta \epsilon$	γ
 37	Cr 93,8 Iso	0,0997	-7.1	105
 38	Cr 46,2 N 70,3 Iso	0,1492	-6.03	263
 39	Cr 66,7 SmB 73,5 N 88,3 Iso	0,1692	-6.9	298
 40	Cr 100,3 N 128,5 I	0,1501	-7.35	353
 41	Cr (41,4) N 86,6 I	0,2097	-6,07	277
 42	Cr 106,7 N 229,9 I	0,2382	-5.9	—
 43	Cr 113,7 N 232,4 I	0,3457	-5.8	—
 44	Cr 146,7 N 346,9 I	0,3417	-5.82	—

Table 4. Phase transition temperatures [$^{\circ}\text{C}$] of the chiral quaterphenyls (**45**)

n	X	R	T, $^{\circ}\text{C}$
6	Cl	$\text{CH}(\text{CH}_3)\text{C}_5\text{H}_{11}$	Cr 50.2 SmC 98.8 Sm A 165.2 Iso
6	Cl	$\text{CH}(\text{CH}_3)\text{C}_6\text{H}_{13}$	Cr 44.7 SmC 85.4 Sm A 158.6 Iso
6	Cl	$\text{CH}(\text{CH}_3)\text{COOC}_4\text{H}_9$	Cr 43.2 SmC 100.1 Sm A 149.7 Iso
7	Cl	$\text{CH}(\text{CH}_3)\text{C}_6\text{H}_{13}$	Cr 42.2 SmC 85.1 Sm A 157.7 Iso
8	Cl	$\text{CH}(\text{CH}_3)\text{C}_4\text{H}_9$	Cr 52.3 SmC 172.3 Iso
8	Cl	$\text{CH}(\text{CH}_3)\text{C}_5\text{H}_{11}$	Cr 37.9 SmC 117.2 Sm A 151.3 Iso
8	CH_3	$\text{CH}(\text{CH}_3)\text{C}_5\text{H}_{11}$	Cr 40.1 SmC 125.3 Sm A 160.1 Iso
8	Cl	$\text{CH}(\text{CH}_3)\text{C}_6\text{H}_{13}$	Cr 28.7 SmC 122.2 Sm A 182.3 Iso
8	CH_3	$\text{CH}(\text{CH}_3)\text{C}_6\text{H}_{13}$	Cr 32.1 SmC 99.6 Sm A 140.7 Iso
8	Cl	$\text{CH}(\text{CH}_3)\text{COOC}_4\text{H}_9$	Cr 45.6 SmC 142.0 Sm A 147.3 Iso
8	Cl	$\text{CH}(\text{CH}_3)\text{CH}_2\text{OC}_3\text{H}_7$	Cr 38.9 SmC 115.3 Sm A 155.7 N 160.0 Iso
10	Cl	$\text{CH}(\text{CH}_3)\text{C}_6\text{H}_{13}$	Cr 39.3 SmC 129.2 Sm A 146.1 Iso
10	CH_3	$\text{CH}(\text{CH}_3)\text{C}_6\text{H}_{13}$	Cr 47.6 SmC 118.8 Sm A 145.3 Iso

and switch-off times low and a wide temperature range of the nematic phase (-30°C – $+105^{\circ}\text{C}$) for VA displays.

(E). Optically active lateral substituted quaterphenyls (**45**) and pentaphenyls (Table 4) and the ferroelectric liquid crystal (FLC) compositions based upon them [38, 39].

The FLC compositions containing the new quaterphenyl derivatives (**45**) are characterized by a wide temperature range of the SmC^* phase, a low operating voltage and a very good quality of orientation in the cells (thermal and the mechanical stable ‘shock-free’) (Table 5).

Our investigations have shown that the mechanical shock [40, 41] does not cause irreversible alignment destruction of FLC cells. We did not find any changes in alignment conditions after dozens of tests. All the cells kept the same alignment quality and were defect-free.

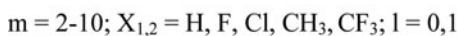
These results reveal that the effect on the parameters of the FLC mixtures strongly depends on the chemical structures of their components and that the substituted chiral

Table 5. Physical parameters^a of the FLC mixtures.

Mixture	SmC^* range ($^{\circ}$)	P_s (nC/cm ²)	Θ ($^{\circ}$)	t_{on} (ms)	T_{of} (ms)	CR^*	CR^{**}
LS-2	<0 – $+66.4$	13	23.8	0.37	0.49	140:1	140:1
LS-6	<0 – $+66.2$	15	24.7	0.33	0.44	170:1	170:1
LS-9	<20 – $+94.1$	12	23.1	0.47	0.51	160:1	150:1
LS-15	<20 – $+64.0$	9	21.5	0.42	0.41	155:1	155:1
LS-18	<20 – $+65.2$	13	23.6	0.53	0.57	140:1	140:1

Notes: P_s , spontaneous polarizations; Θ , tilt angle; CR^* , contrast ratio before deformation, CR^{**} , contrast ratio after deformation; ^ameasured at 25°C .

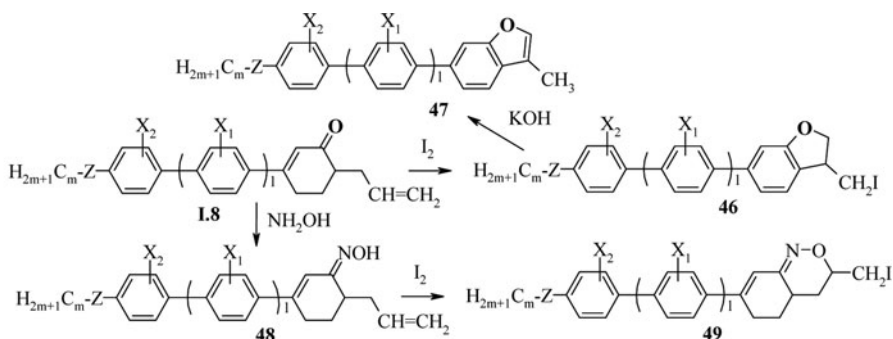
(F). 3-Acetyl-6-arylcylohex-2-enones (**L5**) and other substituted cyclohex-2-enones (**L6-L10**) containing additional functional groups (Scheme 9) [42].



Scheme 9. Preparation of functional substituted cyclohex-2-enones (**I.5-I.10**)

It should be noted that 4-aryl-2-hydroxyacetophenones (**50**) can be used for the preparation of various anisotropic metal complexes and dyes (**56-60**) (Scheme 12) [43].

1,2-Disubstituted cyclopropanols (**IV**) are transformed into saturated (**71**) or unsaturated (**72**) ketones or corresponding allyl halogenides (**73**) (Scheme 14) [26]. This allows in addition to various mesomorphic compounds, which can be synthesized by the modifications of these substances preparing anisotropic polymers or surface modified polymers as the result of the bond formation between polymer activated centers and the functional groups of the unsaturated ketones.

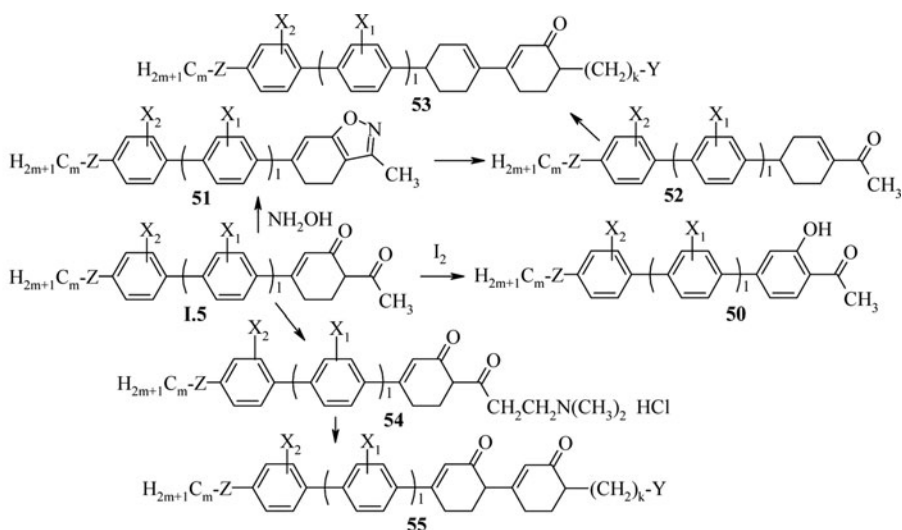


$m = 2-10$; $X_{1,2} = \text{H, F, Cl, CH}_3, \text{CF}_3$; $l = 0, 1$

Scheme 10. Preparation of substituted methyl benzofuranes (**47**) and 6-halomethyl-5,6-dihydro-4H-1,2-oxazines (**49**) or different derivatives (**51-55**) of 3-acetyl-6-aryl cyclohex-2-enones (**1.5**) (Scheme 11).

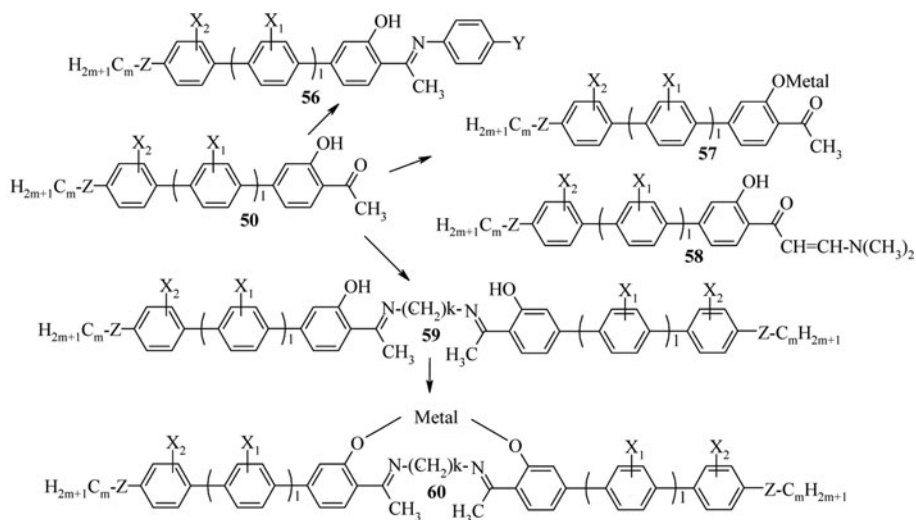
3,5-Disubstituted 2-isoxazolines (**IV**) can serve as building blocks in the synthesis of anisotropic compounds with radically different combination of structural fragments in the terminal position of the molecules, such as polycyclic 1,3-amino alcohols (**75**), β -hydroxy ketones (**76**), 1,3-diols (**77**), β -diketones (**79**), α, β -unsaturated ketones (**78**) (Scheme 15) [28].

Our investigations have shown [29] that 1,3-amino alcohols (**75**) may be transformed under the influence of mineral acids into corresponding anisotropic water soluble salts; β -hydroxy ketones (**76**), 1,3-diols (**77**) – into appropriate chlorine or fluorine derivatives,



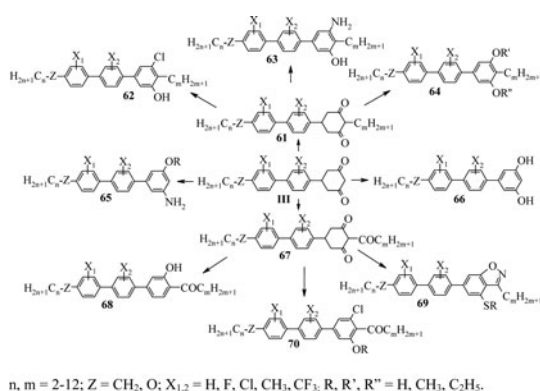
$m = 2-10$; $X_{1,2} = \text{H, F, Cl, CH}_3, \text{CF}_3$; $l = 0, 1$; $k = 1-7$; $Y = \text{H, F, CF}_3$.

Scheme 11. Transformations of 3-acetyl-6-aryl cyclohex-2-enones (**1.5**)



$m = 2-10$; $X_{1,2} = H, F, Cl, CH_3, CF_3$; $l = 0, 1$; $k = 1-5$; $Y = H, \text{alkyl, alkoxy, } F, CF_3$.

Scheme 12. Preparation of anisotropic metal complexes and dyes (**56-60**).

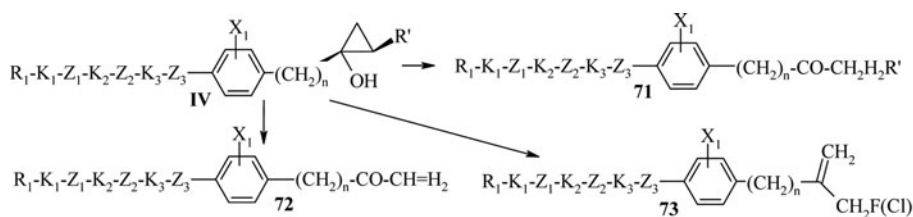


Scheme 13. Transformations of 3,6-disubstituted cyclohex-2-enones (**III**)

which are formed by the interaction with the hydrochloric or hydrofluoric acids, or diethylaminosulphur trifluoride (DAST).

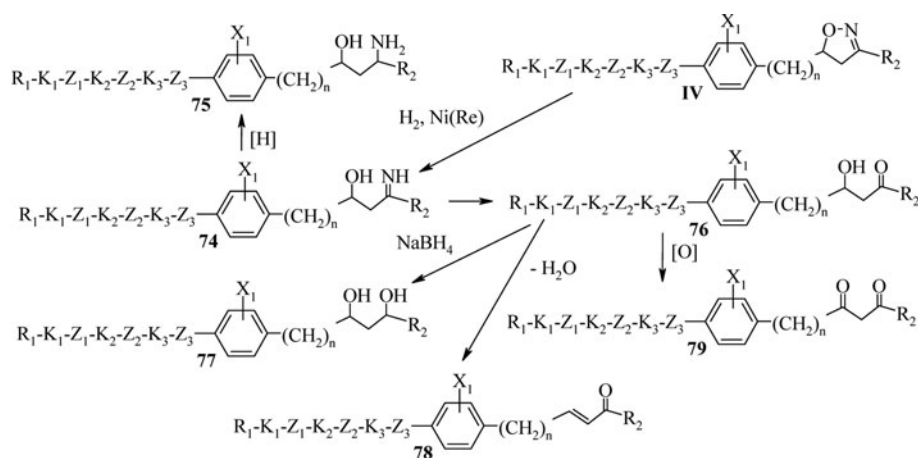
Diketones (**79**) may also be used for the preparation of appropriate anisotropic metal complexes.

It should be additionally noted that diversity of the variants of the modification of 3,5-disubstituted 2-isoxazolines (**IV**), substituted cyclohex-2-enonyl-2-isoxazolines (**VI**) and also 1,2-disubstituted cyclopropanols (**IV**), 5-substituted cyclohexan-1,3-diones (**III**), 2,5-disubstituted cyclohexanones (**II**), 3,6-disubstituted cyclohex-2-enones (**I**) and their derivatives allows to generate aromatic and hetero cycles or introduce different functional groups in lateral or terminal positions accordingly to the presented transformations [29, 36, 44].



$R_{1,2}$ = alkyl or alkoxy fragments, F, Cl, CN, CF_3 , OCF_3 or chiral fragment; K_{1-3} = benzene, cyclohexane, or cyclohexene rings; Z_{1-3} = single bond or CH_2CH_2 , or other bridge fragments; $n = 0 - 5$; $X_1 = H, F, Cl, CH_3, CF_3$;

Scheme 14. Transformations of 1,2-disubstituted cyclopropanols (IV)



$R_{1,2}$ = alkyl or alkoxy fragments, F, Cl, CN, CF_3 , OCF_3 or chiral fragment; K_{1-3} = benzene, cyclohexane, or cyclohexene rings; Z_{1-3} = single bond or CH_2CH_2 , or other bridge fragments; $n = 0 - 5$; $X_1 = H, F, Cl, CH_3, CF_3$;

Scheme 15. Transformations of 3,5-disubstituted 2-isoxazolines (V)

3. Experimental

The structures of the prepared compounds are consistent with the analytical data including H^1 NMR and mass spectra. Phase transition temperatures were measured using a Linkam heating stage in conjugation with a polarising PZO microscope and also using a Setaram DSC 141.

3,6-Disubstituted cyclohex-2-enones (I), 2,5-disubstituted cyclohexanones (II), 5-substituted cyclohexan-1,3-diones (III), 1,2-disubstituted cyclopropanols (IV) and 3,5-disubstituted 2-isoxazolines, substituted cyclohex-2-enonyl-2-isoxazolines (VI) and the

products of their transformations were prepared according to published methods [17–23, 27–44].

4. Conclusion and Perspectives

Thus, the presented results have shown that 3,6-disubstituted cyclohex-2-enones (**I**), 2,5-disubstituted cyclohexanones (**II**), 5-substituted cyclohexan-1,3-diones (**III**), 1,2-disubstituted cyclopropanols (**IV**) and 3,5-disubstituted 2-isoxazolines, substituted cyclohex-2-enonyl-2-isoxazolines (**VI**) are very promising intermediates for the preparation of different types of liquid-crystalline and anisotropic materials. Lots of various chiral and non-chiral mesomorphic and anisotropic compounds with different combinations of cyclic, bridge, terminal fragments, functional groups, and lateral substituents can be prepared from these intermediates using different chemical reagents and reaction conditions.

The diversity of the variants of the modification 3,6-disubstituted cyclohex-2-enones (**I**), 2,5-disubstituted cyclohexanones (**II**), 5-substituted cyclohexan-1,3-diones (**III**), 1,2-disubstituted cyclopropanols (**IV**) and 3,5-disubstituted 2-isoxazolines, substituted cyclohex-2-enonyl-2-isoxazolines (**VI**) under the influence of different chemical reagents and reaction conditions allow the transformations to be achieved selectively and give the opportunity of preparing mesomorphic and anisotropic compounds with novel combinations of the structural fragments of molecules.

It should be noted that the intermediates (**I–VI**) in addition to liquid crystalline compounds for display application may be also transformed under the influence of water, mineral acids, amines or other chemical reagents into corresponding anisotropic α -diols, or halogenhydrines, or amino alcohols, or water soluble salts, or metal complexes. The substrates may be also used for the preparation of anisotropic surface modified polymers as the result of the bond formation between polymer activated centers and the double bond of the unsaturated ketones.

The reviewed results demonstrate that the approaches, intermediates, and synthetic routes can be successfully used for the preparation of original mesomorphic and anisotropic compounds and the creation of new generation anisotropic materials with a very wide spectrum of practical application.

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