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New FLC Compounds with High Optical Anisotropy and the Compositions Based Upon Them

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New FLC Compounds with High Optical Anisotropy and the Compositions Based Upon Them

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Continuing our interest in the ferroelectric liquid crystalline materials for display applications we have synthesized new chiral aryl esters of 4-alkyl-3-methylbiphenyl-4′-carboxylic acids. Using different percentage of these compounds and aryl esters of 4-alkyl-3-chlorobiphenyl-4′-carboxylic acids we have designed the FLC mixtures with values of spontaneous polarization from 30 to 170 nC/cm^2, tilt angle from 20° to 40°, helical pitch (>0.2 μm) and the constant value of the optical anisotropy (∼0.35).

Keywords: ferroelectric liquid crystalline materials; optical anisotropy; tilt angle

INTRODUCTION

We have shown that chiral aryl esters of 4-alkyl-3-chlorobiphenyl-4′-carboxylic acids and optically active chlorine lateral substituted terphenyls form smectic C phase at low temperature and over wide temperature ranges [1–4]. In continuation of these investigations and in an attempt to obtain new promising components of the ferroelectric liquid crystalline materials for display applications we have synthesized new chiral aryl esters of 4-alkyl-3-methylbiphenyl-4′-carboxylic acids (**Ia-d**), investigated their mesomorphic properties and physical and electrooptical properties of the FLC compositions based upon them.

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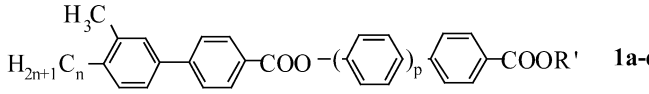
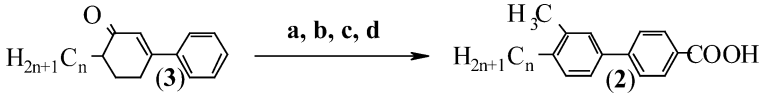


1a-d

n = 5, 10 ; p = 0 or 1; R' = $CH_2CH^*(CH_3)C_2H_5$, $C^*H(CH_3)C_6H_{13}$

RESULTS AND DISCUSSION

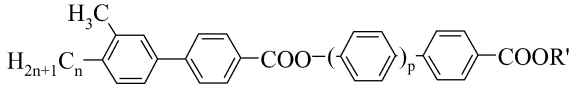
The esters (**1a-d**) (see Table 1) were synthesized by the interaction of 4-alkyl-3-methylbiphenyl-4′ -carboxylic acid (**2**) with the corresponding chiral 4-substituted phenols in the presence of dicyclohexyl-carbodiimide (DCC) and 4-dimethylaminopyridine as catalyst. For the preparation of acids (**2**) we used the synthetic pathway (see the scheme below) based on the transformation of 3-biphenyl-6-alkylcyclohex-2-enones (**3**), which are formed in high yield (75–80%) by Michael condensation of appropriate Mannich salt with ethyl 2-alkylacetoacetic ester [4,5].



a.CH_3MgJ; ***b***.J_2, *isoPrOH*; ***c***.CH_3COCl, $AlCl_3$; ***d***.*NaOBr*

As can be seen from the Table, the compounds (**1a,c,d**) are strongly smectogenic compounds forming smectic A and smectic C phases at low temperatures. It should be noted that the ester (**1b**) does not form mesophases.

TABLE 1 Transition Temperatures of Esters (**1**)



				Transition temperatures/°C								
N	n	p	R′	Cr		Sm C		Sm A		N		I
a	5	0	$CH_2CH(CH_3)C_2H_5$	•	27	•	66	•	80	•	88	•
b	10	0	$CH(CH_3)C_6H_{13}$	•		–		–		–	34	•
c	10	1	$CH(CH_3)C_6H_{13}$	•	44	•	107	•		–	140	•
d	10	1	$CH_2CH(CH_3)C_2H_5$	•	68	•	129	•	195	•	200	•

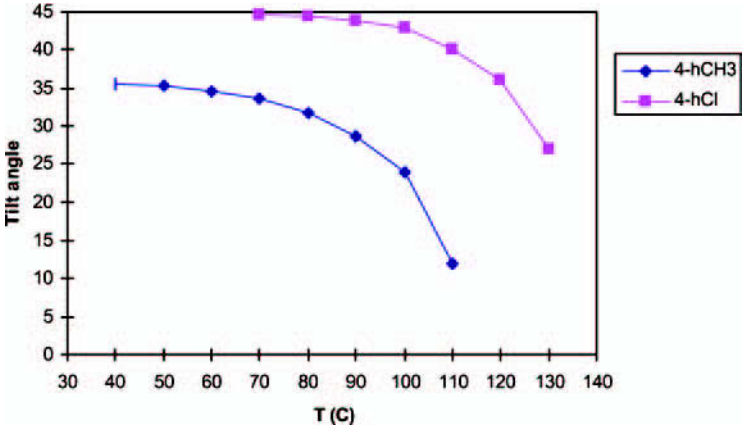


FIGURE 1 Temperature dependence of the tilt angle of (**1a**) the analogous chloroderivative.

Investigations of the electro-optical properties of compounds (**1a-d**) have shown their main advantages in comparison with the analogous esters of 4-alkyl-3-chlorobiphenyl-4′ -carboxylic acids [1,2]. The spontaneous polarization and tilt angle of these compounds are not so high and vary from 80 to140 nC/cm^2 and from 20° to 36° dependent upon the chemical structures of the compounds (see Figs. 1 and 2). However, it was found that methyl lateral substituted esters (**1a-d**) allow the temperature transition from crystal to smectic C phase to be essentially decreased, and also to increase the spontaneous polarization of the FLC mixtures without significant changes in other parameters.

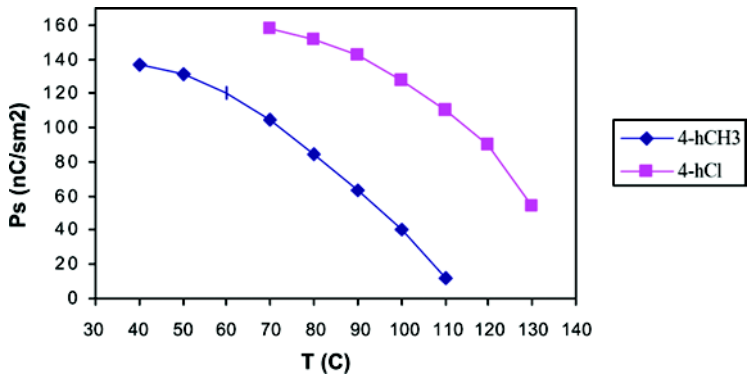


FIGURE 2 Temperature dependence of the spontaneous polarization of (**1a**) and the analogous chloroderivative.

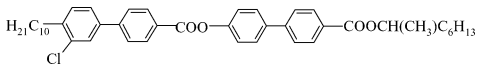
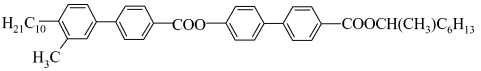
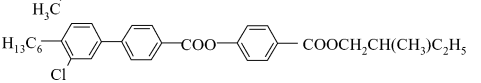
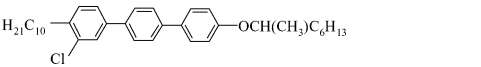
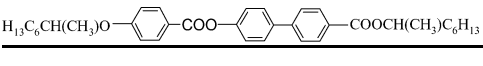
Taking this into account and also that four ring esters (**1b-d**) are characterized by high birefringence, we have developed high optical anisotropy FLC mixtures based upon them and the corresponding chloroderivatives We reported before [1], that the chiral aryl esters of 4-alkyl-3-chlorobiphenyl-4′ -carboxylic acids form smectic C phase in the temperature range 70–140°C, have a tilt angle 44° and are promising components of FLC mixtures.

Detailed investigations of the electro-optical properties of prepared FLC mixtures and the data presented in Tables 2 and 3, Figs. 3 and 4 show, that using different percentage of the components, the FLC mixtures with a wide temperature range of Sm C phase $<20--+83$°C, value of spontaneous polarization from 30 to 170 nC/cm^2, tilt angle from 20° to 40°, helical pitch ($>0.2\,\mu$m) and the constant value of the optical anisotropy (~ 0.35) can be prepared.

TABLE 2 Physical Parameters of the FLC-Mixtures with High Optical Anisotropy

Mixture	SmC* temperature range (°C)	Operating voltage (V/micrometer)	Spontaneous polarization (nC/cm^2)	Tilt angle	Δn
LBHS-9	$20<--+69$	10	173	35,8	0.245
LBHS-12	$20<--+85$	10	55	31,5	0.335
LBHS-13	$20<--+72$	10	147	29,2	0.335
LBHS-17	$\sim 20--+73$	10	75	36,9	0.335
LBHS-19	$20<--+83$	10	82	30,2	0.34

TABLE 3 The Influence of the Chemical Structures of Compounds on the Main Parameters of FLC-Mixtures

Compound	Δn	Tilt angle	P_s	$T_{Cr\text{-}SmC}$	$T_{SmC\text{-}SmA}$
	↑	↑	=	=	↑
	↑	=	↑	↓	↑
	=	↑	=	↓	=
	=	↓	↓	↓	=
	=	↑	↑	↓	=

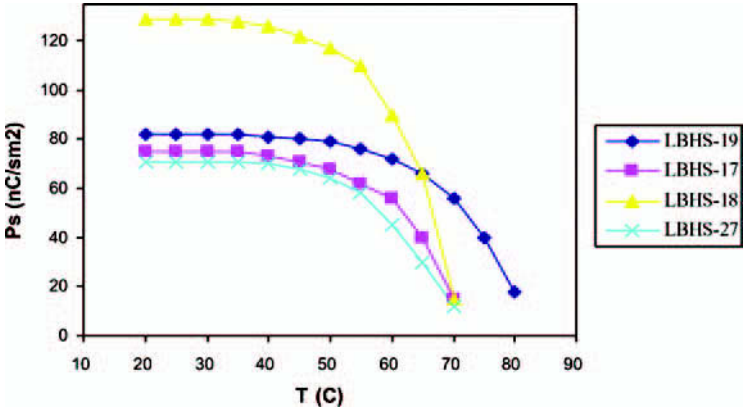


FIGURE 3 Temperature dependence of the spontaneous polarization of the FLC mixtures.

It should be noted that we have prepared the mixtures with a wide temperature range of the SmC*-phase, low melting point and high switching angle using only two compounds.

These results reveal that the effect on the parameters of the mixtures strongly depends on the chemical structures and content of their components and that the chiral aryl esters of methyl or chlorobiphenylcarboxylic acids or analogous of them compounds are the favorable for preparation of FLC mixtures with a wide temperature range of Sm C phase and high optical anisotropy.

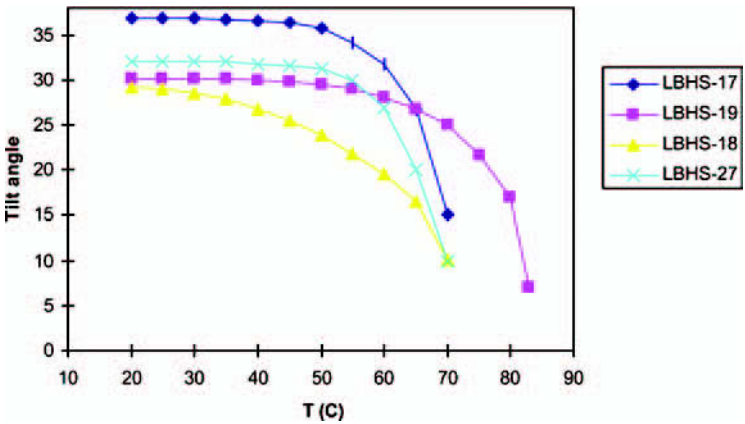


FIGURE 4 Temperature dependence of the tilt angle of the FLC mixtures.

We are going to synthesize other structurally similar LC compounds to assess their ability to generate the Sm C phase at low temperature and in a wide temperature range. We hope that the new results of our investigations may lead to better understanding of the nature of the ferroelectric liquid crystals and their use for display applications.

EXPERIMENTAL

The structures of the prepared compounds were confirmed by ^{1}H-NMR and mass spectroscopy. Phase transition temperatures were measured using a Linkam heating stage having a polarizing PZO microscope and also using a Setaram DSC 92.

Electro-optic studies were performed in the glass cells supplied with ITO electrodes (with the receptivity 150 Ohm/cm^2) and SiO_2 insulating layers 170 nm thick. Aligning layers (nylon 6, 130 nm) were spinned and unidirectionally rubbed. The thickness of the cells was about 1.9 μm and measured in each case interferometrically. During electro-optic measurements the temperature of the cells was controlled with the accuracy 0.3°C and the gradients across the sample did not exceed 1°.

The intermediates, 4-alkyl-3-methylbiphenyl-4′ -carboxylic acid (**2**), 3-biphenyl-6-alkylcyclohex-2-enones (**3**) and the final esters (**1a-d**) were prepared according to published methods [1–6].

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