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Unsymmetrical banana-shaped liquid crystalline compounds derived from 2,7-dihydroxynaphthalene

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Abstract: The synthesis and characterization of new asymmetric bent-core compounds derived from 2,7-dihydroxynaphthalene with various connecting groups between the aromatic rings and alkyloxy terminal substituents at the end of the long arm are presented. Some 1,4-disubstituted phenylene rings with an azo or ester linkage between them have been used as calamitic pro-mesogen units. The synthetic strategies to obtain the final esteric derivatives involved the esterification of 7-(benzyloxy)naphthalen-2-ol with 4-((4-(alkyloxy)phenyl)-azo)benzoyl chlorides or with 4-((4-(alkyloxy)benzoyl)oxy)benzoic acids in the presence of dicyclohexylcarbodiimide (DCC) and 4-(dimethylamino)pyridine (DMAP). The mesomorphic properties were assigned by optical polarizing microscopy and differential scanning calorimetry. All the compounds showed mesomorphic properties of the enantiotropic or monotropic type, the liquid crystalline behavior depending on the linking group between the phenylene rings. Thermogravimetric studies evidenced that all compounds were stable in the range of the existence of mesophases.

Keywords: liquid crystals; asymmetric bent-core; 2,7-naphthalenediol.

INTRODUCTION

Although classical liquid crystals are widely used in a variety of applications, especially in displays, significant research has been performed on non-conventional, bent-core liquid crystals in order to establish structure–property relationships. The first mention of the existence of mesogenic properties exhibited by non-linear compounds was reported in 1929 by Vorländer for isophthalic acid derivatives, which showed nematic phases.¹ The research on bent-core liquid crystals (BCLC) was taken and further extended by Matsunaga and co-workers.^{2–5} The field of bent-core liquid crystals has greatly advanced since

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1996, when Niori showed that because of limitation of the rotational freedom in the mesophase, bent-shaped compounds are able to organize into a compact arrangement and exhibit spontaneous polarization, which leads to ferroelectric properties.⁶

The bent-shaped molecular structure is provided for by proper selection of a central core with an adequate bent angle and by covalent connection of two mesogenic units to this. The positions used for a bent connection are at 1,3-, 2,6-, 2,7- or 1,3'- for benzene, pyridine, naphthalene or biphenyl, respectively. Such structures induce a bent angle of about 120°. While the mesophases exhibited by such compounds are similar to those displayed by classical calamitic liquid crystals, some characteristic B phases are usually present in such structures. Thus, while compounds based on 1,2-disubstituted benzene or 2,3-disubstituted naphthalene as the central core provide an angle of about 60° and display only conventional nematic and smectic phases,^{2,4} 2,7-disubstituted naphthalenes lead to derivatives that exhibit typical banana phases.⁷⁻¹⁰

The substitution of the central core could be symmetrical or non-symmetrical. Compared to symmetrical structures, the non-symmetrical ones induce a lowering of the transition temperatures. Most of the banana-shaped compounds contain five aromatic rings connected through various linking groups. Increasing of the number of phenyl rings enhances the core rigidity and tends to increase the transition temperatures, whereas terminal alkyl tails induce flexibility (which lowers the phase transition temperatures) and permits the formation of B phases.

The presence of a 2,7-disubstituted naphthalene unit as the central core in banana-shaped compounds was first reported in 1999 by Shen *et al.*¹¹ and Pelzl *et al.*¹² While the compounds reported by Shen *et al.* did not show mesomorphic properties, those reported by Pelzl *et al.* showed a B₁ phase. Further studies on such compounds focused on the introduction of azomethine linking groups, which exhibited smectic¹³⁻¹⁵ or nematic and B phases.¹⁶⁻¹⁸

In this paper, the synthesis, structural characterization and mesomorphic properties of two new classes of liquid crystalline compounds based on a non-symmetrical 2,7-disubstituted naphthalene core and containing esteric or azo linking groups in the pro-mesogenic moiety are reported.

EXPERIMENTAL

Materials, instruments and methods

Benzyl chloride, 2,7-dihydroxynaphthalene (Aldrich), potassium iodide, potassium carbonate (Fluka), tetrabutylammonium hydrogensulfate (TBAHS) (Aldrich), 1,3-dicyclohexylcarbodiimide (DCC) (Fluka), 4-(dimethylamino)pyridine (DMAP) (Fluka) and silica gel-60 (Merck) were used as received. Acetone and dichloromethane were refluxed over phosphorus pentoxide (Merck) and distilled before use. Other solvents and chemicals were used without further purification. 7-(Benzyloxy)naphthalen-2-ol (**1**),¹⁹ 4-((4-(alkyloxy)benzoyloxy)benzoic

acids (**3a–e**),²⁰ 4-((4-(alkyloxy)phenyl)azo)benzoic acids²¹ and 4-((4-alkyl)oxyphenyl)azo-benzoyl chlorides (**2a–e**)²² were synthesized according to literature procedures.

Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Avance DRX 400 MHz spectrometer. Chemical shifts are reported in ppm relative to tetramethylsilane (TMS) as internal standard. The mass spectra were recorded on a quadrupole-time of flight mass spectrometer equipped with an electrospray ion source (Agilent 6520 Accurate Mass Q-ToF LC/MS). FT-IR spectra were recorded on a Nicolet Magna 550 FT-IR spectrometer (NaCl crystal window).

The transition temperatures were determined using a Linkam heating stage connected with a Linksys 32 temperature control unit in conjunction with an Axioscop 40 Zeiss polarizing optical microscope (POM) and Qimaging/Retiga-1000R camera for image capture. The transitions were confirmed by DSC analysis (Mettler Toledo DSC1). Heating and cooling cycles were run at rates of 10 °C min⁻¹ under a nitrogen atmosphere. The samples were measured in closed lid aluminum pans. Mesophase type was assigned by visual comparison (under the microscope) with known phase standards.²³

All the thermal analysis were run under the same conditions, on 2.8 – 4.3 mg samples, on a Mettler-Toledo TGA SDTA851 derivatograph under a dynamic N₂ atmosphere, with a flow rate of 20 ml min⁻¹ and a heating rate of 10 °C min⁻¹ from 25 to 900 °C. In order to obtain comparable data, constant operational parameters were employed for all samples.

The melting points were recorded using a melting point meter Krüss Optotronic KSPI-N and are uncorrected.

General method for the preparation of 7-(benzyloxy)naphthalen-2-yl 4-((4-(alkyloxy)phenyl)diazenyl)benzoates (4a–e)

Compounds **4a–e** were prepared by adapting a literature method.²⁴ A mixture of 7-(benzyloxy)naphthalen-2-ol (0.20 g, 0.799 mmol), 4-((4-(alkyl)oxyphenyl)azo)benzoyl chloride (0.877 mmol), potassium carbonate (0.14 g, 1.00 mmol), tetrabutylammonium hydrogensulfate (0.006 g, 0.017 mmol) in dichloromethane (150 mL) and water (40 mL) was vigorously stirred for 24 h at room temperature. The organic layer was separated, washed several times with distilled water, dried over anhydrous magnesium sulfate and concentrated on rotaevaporator. The compounds were separated by column chromatography on silica gel using a mixture of dichloromethane:hexane 20:1 as eluent. Orange products were obtained.

The following compounds were synthesized: 7-(benzyloxy)naphthalen-2-yl 4-((4-(hexyloxy)phenyl)diazenyl)benzoate (**4a**) using 4-((4-(hexyloxy)phenyl)azo)benzoyl chloride 0.302 g (0.877 mmol); 7-(benzyloxy)naphthalen-2-yl-4-((4-(heptyloxy)phenyl)diazenyl)benzoate (**4b**) using 4-((4-(heptyloxy)phenyl)azo)benzoyl chloride 0.315 g (0.879 mmol); 7-(benzyloxy)naphthalen-2-yl 4-((4-(octyloxy)phenyl)diazenyl)benzoate (**4c**) using 4-((4-(octyloxy)phenyl)azo)benzoyl chloride 0.327 g (0.878 mmol); 7-(benzyloxy)naphthalen-2-yl 4-((4-(nonyloxy)phenyl)diazenyl)benzoate (**4d**) using 4-((4-(nonyloxy)phenyl)azo)benzoyl chloride 0.339 g (0.877 mmol); 7-(benzyloxy)naphthalen-2-yl 4-((4-(decyloxy)phenyl)diazenyl)benzoate (**4e**) using 4-((4-(decyloxy)phenyl)azo) benzoyl chloride, 0.352 g (0.879 mmol).

The physical and spectral data for **4a–e** are given in the Supplementary material to this paper.

General method for the preparation of 7-(benzyloxy)naphthalen-2-yl 4-((4-(alkyloxy)benzoyl)oxy)benzoates (5a–e)

Compounds were prepared by adapting literature data.²⁵ A mixture of 1 equivalent of 7-(benzyloxy)naphthalen-2-ol (0.20 g, 0.799 mmol), 1.1 equivalents of 4-((4-(alkyloxy)ben-

zoyl)oxy)benzoic acid and 0.2 equivalents of DMAP (0.019, g 0.155 mmol), dissolved in dry dichloromethane (50 mL), was stirred for 15–20 min at room temperature, cooled to 0 °C on an ice bath and then 1.2 equivalents of DCC (0.197 g, 0.954 mmol), dissolved in dry dichloromethane (50 mL), were added dropwise. After 30 min, the ice bath was removed and the reaction mixture was stirred for 48 h at room temperature after which the precipitated *N,N'*-dicyclohexylurea (DCU) was filtered off. The solvent was evaporated under vacuum and the solid residue was chromatographed on silica gel using a 20:1 mixture of dichloromethane:ethyl acetate as the eluent. White products were obtained.

The following compounds were synthesized: 7-(benzyloxy)naphthalen-2-yl 4-((4-(hexyloxy)benzoyl)oxy)benzoate (**5a**) using 4-((4-(hexyloxy)benzoyl)oxy)benzoic acid (0.300 g, 0.876 mmol); 7-(benzyloxy)naphthalen-2-yl 4-((4-(heptyloxy)benzoyl)oxy)benzoate (**5b**) using 4-((4-(heptyloxy)benzoyl)oxy)benzoic acid 0.313 g (0.878 mmol); 7-(benzyloxy)naphthalen-2-yl 4-((4-(octyloxy)benzoyl)oxy)benzoate (**5c**) using 4-((4-(octyloxy)benzoyl)oxy)benzoic acid 0.325 g (0.877 mmol); 7-(benzyloxy)naphthalen-2-yl 4-((4-(nonyloxy)benzoyl)oxy)benzoate (**5d**) using 4-((4-(nonyloxy)benzoyl)oxy)benzoic acid 0.339 g (0.877 mmol); 7-(benzyloxy)naphthalen-2-yl 4-((4-(decyloxy)benzoyl)oxy)benzoate (**5e**) 4-((4-(decyloxy)benzoyl)oxy)benzoic acid 0.350 g (0.878 mmol).

The physical and spectral data for **5a–e** are given in the Supplementary material to this paper.

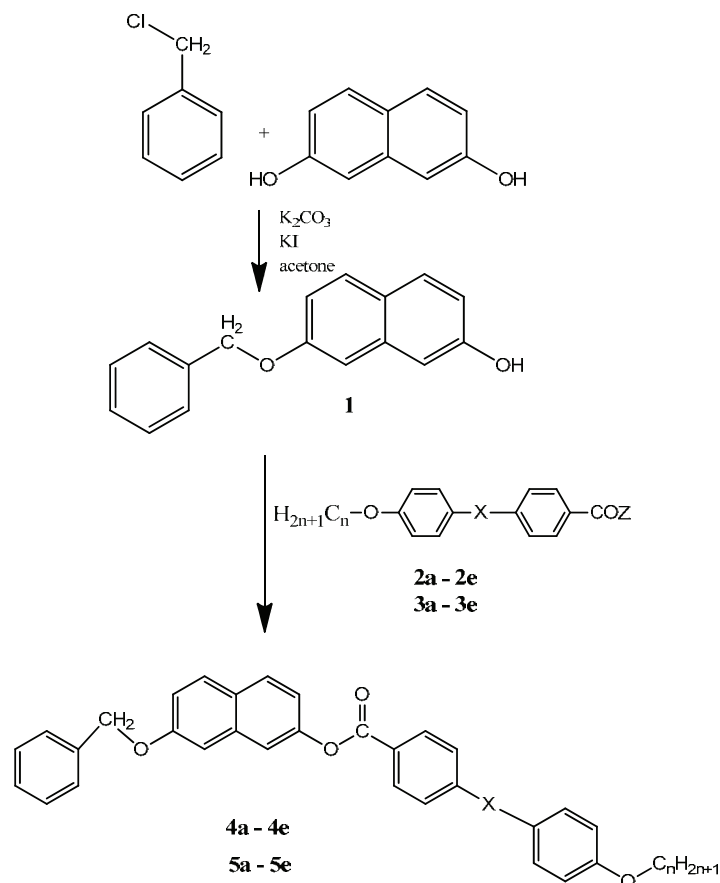
RESULTS AND DISCUSSION

The synthetic approach used to prepare the intermediate and final compounds is outlined in Scheme 1. The short arm compound (**1**) was prepared by alkylation of 2,7-dihydroxynaphthalene with benzyl chloride.¹⁹ Depending of the chemical structure, the longer arm was coupled to the remaining hydroxyl group of the central mono-alkylated naphthalene core by two methods, which gave two series of final compounds. The first series used 4-((4-(alkyloxy)phenyl)azo)benzoyl chlorides **2a–e** in the presence of potassium carbonate as base and TBAHS as phase transfer catalyst to give the target compounds **4a–e**. The second one coupled 4-((4-(alkyloxy)phenyl)azo)benzoic acids **3a–e** using DCC and DMAP to achieve the final compounds **5a–e**.

All the obtained compounds were purified by column chromatography using dichloromethane:hexane 20:1 (for series **4a–e**) or dichloromethane:ethyl acetate 20:1 (for series **5a–e**) as eluents. The yields were similar for both series (between 61–79 %). The structure and purity of the obtained final compounds were checked and confirmed by ¹H-NMR, ¹³C-NMR, FT-IR and mass spectrometry (data given in the Supplementary material to this paper).

Thermogravimetric studies performed for both the **4a–e** and **5a–e** series evidenced very good thermal stability for all the investigated compounds, the T_{onset} values (temperatures at which the degradation processes begin) being at least 155 °C higher than the isotropization temperatures (the T_{onset} values are given in Tables I and II).

The phase transition temperatures and phase transition enthalpy changes were determined by differential scanning calorimetry (Tables I and II).



Scheme 1. Synthesis of bent-core liquid crystalline compounds; **4a-e**, X = -N=N-, Z = -Cl, $n = 6-10$, aq. K_2CO_3 , TBAHS, CH_2Cl_2 , 24 h, RT; **5a-e**, X = -OCO-, Z = -OH, $n = 6-10$, DCC, DMAP, CH_2Cl_2 , 48 h, RT.

The first compound of the series **4**, compound **4a**, exhibited monotropic behavior, visible under polarized light between 126–95 °C. The transition isotropic liquid–liquid crystal was a very slow process and difficult to identify on the DSC curve; nevertheless, it was identified from POM observations at about 126 °C (similar behaviors were evidenced in the case of the other compounds of series **4** and **5**, Tables I and II). The microscopic analyses highlighted an interesting unidentified B type texture at 110 °C during the first cooling (Fig. 1a), which did not appear on the second cooling curve when only a classical B type texture was evidenced (Fig. 1b). The mesophase was stable in a temperature domain of 31 °C; the crystallization process started at 95 °C and the process was completed at 91 °C after a crystalline/crystalline transition.

TABLE I. Transition temperatures and associated enthalpy values of compounds **4a–e**; Cr, crystalline; LC, liquid crystal; I, isotropic; *MI*, mesophase interval; t_{onset} , the initial temperature at which the degradation processes begin

Cmpd.	$t / ^\circ\text{C} [\Delta H / \text{J g}^{-1}]$									$t_{\text{onset}} / ^\circ\text{C}$
	Heating				Cooling					
	Cr ₁	Cr/LC	LC/I	<i>MI</i> / °C	I/LC	LC/Cr ₃	Cr ₃ /Cr ₂	Cr ₂ /Cr ₁	<i>MI</i> / °C	
4a	–	–	155	–	126 ^a	95	91	–	31	308
			[–87.60]			[38.07]	[1.55]			
4b	–	–	143	–	118	100	–	–	18	315
			[–77.24]		[0.51]	[79.23]				
4c	107	120	150	30	129 ^a	118	82	–	11	316
	[–3.37]	[–14.06]	[–26.94]			[0.32]	[37.52]			
4d	–	110	137	27	128 ^a	92	87	83	36	314
		[–6.15]	[–43.12]			[34.32]	[84.54]	[78.30]		
4e	–	113	148	35	123 ^a	89	85	–	34	319
		[–30.04]	[–72.59]			[36.63]	[0.34]			

^aData obtained from POM

TABLE II. Transition temperatures (°C) and associated enthalpy values (J g^{–1}) of compounds **5a–e**

Cmpd.	$t / ^\circ\text{C} [\Delta H / \text{J g}^{-1}]$									$t_{\text{onset}} / ^\circ\text{C}$
	Heating				Cooling					
	Cr ₁	Cr/LC	LC/I	<i>MI</i> / °C	I/LC	LC/Cr ₃	Cr ₃ /Cr ₂	Cr ₂ /Cr ₁	<i>MI</i> / °C	
5a	–	122	144	22	109	84	–	–	25	336
		[–27.81]	[–63.03]		[0.44]	[62.79]				
5b	109	116	129	13	104	75	–	–	29	324
	[–6.74]	[–11.02]	[–37.22]		[0.48]	[56.08]				
5c	–	127 ^a	148	21	107	83	–	–	24	331
			[–83.93]		[0.56]	[70.12]				
5d	–	–	146	–	108	87	80	–	28	334
			[–84.80]		[0.73]	[0.66]	[66.91]			
5e	–	–	136	–	108	90	68	59	49	318
			[–76.63]		[0.58]	[0.72]	[5.62]	[28.16]		

^aData obtained from POM

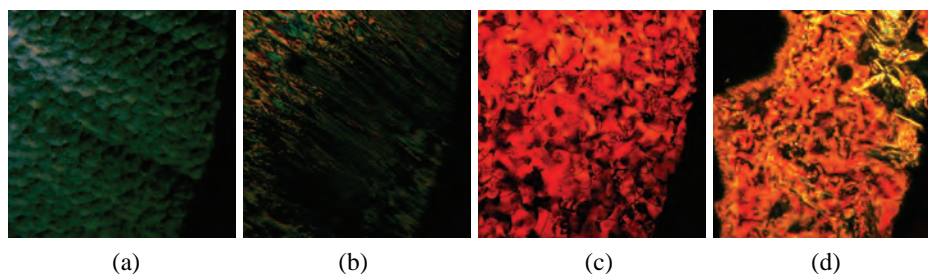


Fig. 1. Microphotographs of the mesophase textures: a) compound **4a**, first cooling, 110 °C; b) compound **4a**, second cooling, 126 °C; c) compound **4b**, first cooling, 107 °C; d) compound **4c**, first heating, 142 °C.

The increasing of the number of carbon atoms in the alkyloxy tail from 6 (compound **4a**) to 7 (compound **4b**) lowered the isotropization temperature and the domain of the mesophase stability. The mesophase changed into a nematic type with characteristic Schlieren texture (Fig. 1c). The mesomorphic properties switched significantly for compound **4c**, which exhibited an enantiotropic liquid crystalline behavior. A nematic phase was stable for 30 °C on heating (Fig. 1d). However, the stability of the mesophase on cooling decreased when compared with the first two compounds.

The enantiotropic behavior was maintained for the next two compounds of the series, **4d** and **4e**, and the stability of mesophases on heating increased gradually, compound **4e** showing the broadest domain of about 35 °C. On cooling, compound **4d** showed liquid crystalline properties in a 36 °C temperature domain, plus a rich polymorphism during the crystallization, evidenced on the DSC curves (Fig. 2). The microscopic analysis for these compounds evidenced mesophases of nematic and B types (Fig. 3a and b).

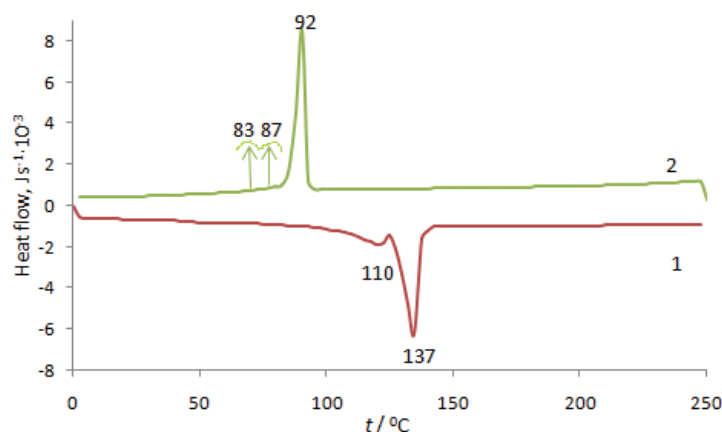


Fig. 2. DSC curves for compound **4d**: 1 – first heating, 2 – first cooling.

The first compound of the second series, **5a**, exhibited an enantiotropic behavior, with characteristic Schlieren nematic textures both on the heating and cooling cycles (Fig. 3c). The mesophase appeared late under the visual field of the microscope, the sample being extremely sensitive to any touch when it crystallized. A similar behavior was observed for compound **5b**, the isotropization point of which was lower than that of **5a** (Table II). On heating, the mesophase was of B type, while on cooling the ordering began with Schlieren nematic textures that changed into a smectic one at 87 °C (Fig. 3d).

The same behavior of the mesophase was evidenced for compound **5c** on heating. On heating, a transition crystalline to a B type mesophase was identified from POM observations at about 127 °C (Fig. 4a). On cooling, the mesophase

was of nematic type with Schlieren texture that turned into a ribbon type one (Fig. 4b) near the crystallization temperature.

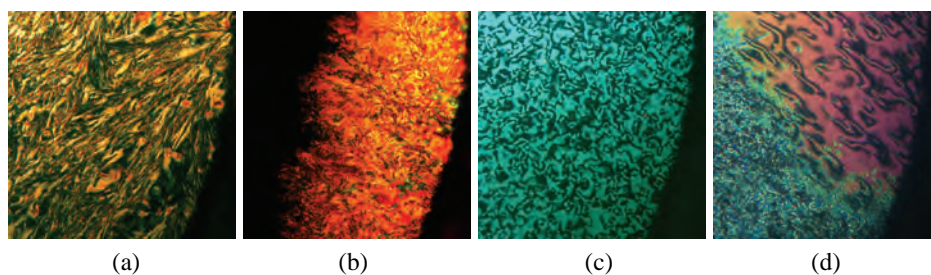


Fig. 3. Microphotographs of the mesophase textures: a) compound **4d**, fourth cooling, 126 °C; b) compound **4e**, second cooling, 122 °C; c) compound **5a**, first cooling, 96 °C; d) compound **5b**, second cooling, 85 °C.

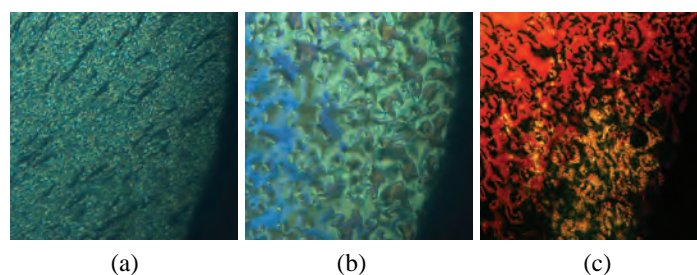


Fig. 4. Microphotographs of the textures: a) compound **5c**, second heating, 133 °C; b) compound **5c**, second cooling, 95 °C; c) compound **5d**, first cooling, 91 °C.

The last two compounds of the series, **5d** and **5e**, changed the mesomorphic behavior and showed liquid crystalline properties only on cooling (Fig. 4c), with several crystalline to crystalline transitions evidenced on the DSC curves (Fig. 5).

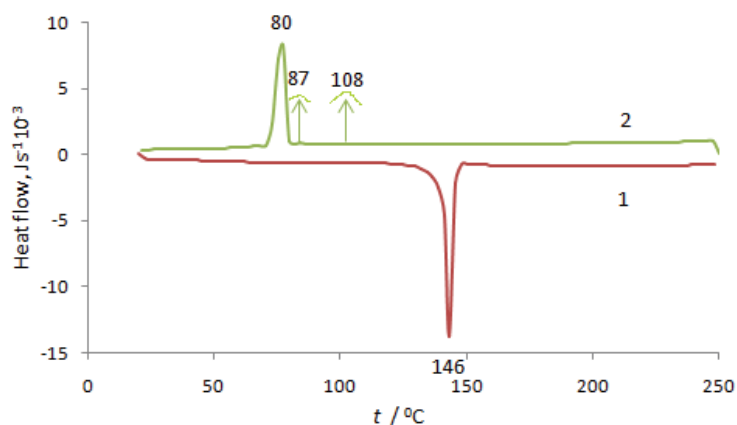


Fig. 5. DSC curves for compound **5d**: 1 – first heating, 2 – first cooling.

With only one exception (compound **4d**), there were significant differences between the isotropization temperatures and the temperatures at which the compounds entered into mesophase on cooling (between 21 and 41 °C, 9 °C for compound **4d**, Fig. 6).

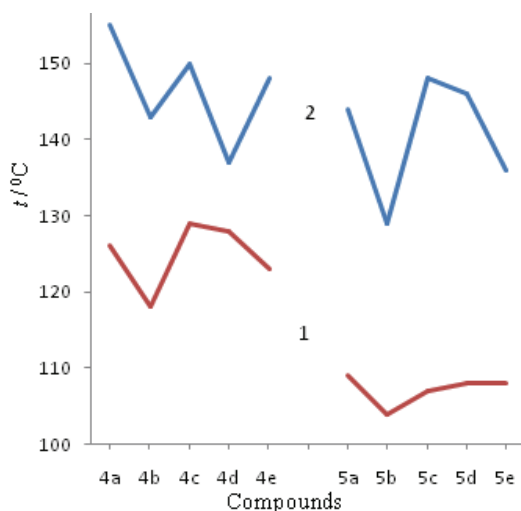


Fig 6. Comparison between the isotropization temperatures and LC temperatures (temperatures at which the compounds are entering into mesophase on cooling): 1 – LC temperatures; 2 – isotropization temperatures.

For series **4a–e**, an even/odd effect related to the number of carbon atoms in the tail may be evidenced (Fig. 6). In the same time, the differences between the isotropization temperatures and the LC temperatures on cooling for series **5a–e** were 14–22 °C higher than those for series **4a–e**.

CONCLUSIONS

Two new series of asymmetrical bent-core liquid crystalline compounds based on a 2,7-dihydroxynaphthalene core were synthesized and characterized. All compounds contained a benzyloxy non-mesogenic shorter flexible arm and a pro-mesogenic arm containing two benzenic rings connected by azo or ester linking groups. All the investigated compounds exhibited liquid crystalline properties, mainly of the enantiotropic type, as evidenced by optical polarizing microscopy and differential scanning calorimetry. Despite the absence of a long flexible tail in the shorter arm, the isotropization temperatures were relatively low (between 129 and 155 °C). There were no significant differences related to the transition temperatures and the stabilities of the mesophases as a function of the nature of the linking groups between the aromatic rings. The investigated compounds showed nematic and B type mesophases.

Thermogravimetric studies evidenced that all compounds were stable in the range of the existence of mesophases, the degradation processes beginning at temperatures more than 155 °C higher than the isotropization temperatures.

SUPPLEMENTARY MATERIAL

Physical and spectral data for the prepared compounds are available electronically from <http://www.shd.org.rs/JSCS/>, or from the corresponding author on request.

ИЗВОД

АСИМЕТРИЧНА ЈЕДИЊЕЊА ТЕЧНИХ КРИСТАЛА У ОБЛИКУ БАНАНЕ ИЗВЕДЕНА ИЗ 2,7-ДИХИДРОКСИНАФТАЛЕНА

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У овом раду представљена је синтеза и карактеризација нових асиметричних једињења савијеног језгра изведених из 2,7-дихидрокси нафталена са различитим везивним групама између ароматичних прстенова и алкилокси терминалних супституената на крају дуге руке. Као каламитичне промезогене јединице коришћени су неки од 1,4-дисупституисаних фениленских прстенова повезаних преко азо или естарских веза. Стратегија синтезе крајњих естарских производа садржавала је естерификацију 7-(бензилокси)нафтален-2-ола употребом 4-((4-алкилокси)фенил)азобензоил-хлорида или 4-((4-(алкилокси)бензоил)окси)бензоое киселине у присуству дициклохексилкарбодимида (DCCI) и 4-(диметиламино)пиридина (DMAР). Мезоморфна својства одређене су оптичком поларизационом микроскопијом и диференцијалном скенирајућом калориметријом. Сва једињења показују мезоморфне особине енантиотропног и монотропног типа, понашање течних кристала зависи од група које повезују фениленске прстенове. Термогравиметријска истраживања су показала да су сва једињења стабилна у опсегу постојања мезофаза.

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