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# Synthesis of new mesogenic compounds having the isoflavone core group

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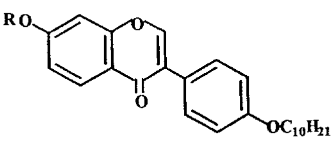
Two new series of calamitic liquid crystals containing the isoflavone moiety within the central core have been prepared and their liquid crystalline properties studied. The compounds exhibit typical nematic, smectic A and C mesophases. The influence of the linking group between a terminal alkyl chain and the central core has been proven to determine the variety of mesomorphism displayed by the compounds.

## 1. Introduction

Tens of thousands of compounds with a classical rod-like structure consisting of a conjugated aromatic central core and two terminal flexible chains have been prepared and their behaviour as liquid crystals studied. The possible structural modifications are almost unlimited and the study and understanding of the relationship between the chemical structure and the liquid crystalline properties of these compounds is one of the main objectives of researchers working of this field. The introduction of heterocycles within the central core of calamitic molecules strongly influences their mesomorphic behaviour due, though not only, to the dipolar moment associated with the heterocycling ring. Many natural products presenting heterocycles in their structures, such as flavone, coumarin and isoflavone derivatives, are involved in biological systems which may be in a liquid crystalline state [1, 2]. Therefore, we have considered interesting the study of some new compounds incorporating the 3-phenyl-4-[H]-4-[one]-benzopyran (isoflavone) moiety in the central core of a rod-like molecule.

Only a few examples describing the synthesis and mesomorphic behaviour of isoflavone derivatives containing only one terminal chain have been reported in the literature [3, 4]. In the present work we describe the synthesis and liquid crystalline properties of two series of isoflavone derived compounds with the classical calamitic structure: two flexible terminal chains and a rigid central core. The structure of the compounds of series I and II are shown in the sketch.

**SKETCH**



**SERIES I:** $R = n\text{-}C_nH_{2n+1}$ ; $n = 3\text{-}10$

**SERIES II:** $R = \text{-}COC_nH_{2n+1}$ ; $n = 3\text{-}9$

## 2. Synthesis

The synthesis of the compounds is outlined in the scheme. Compounds **3** were prepared from resorcinol and 4-hydroxyphenylacetic acid or 4-*n*-decyloxyphenylacetic acid according to the method described [5] for 7-hydroxy-4′-methoxyisoflavone. Freshly distilled $BF_3.Et_2O$ as catalyst and solvent was used for the Friedel Crafts reaction. The intermediates **2** were purified and then were cyclized using methane sulfonylchloride and DMF. Finally, the series of compounds **4**, **5** and **6** were prepared by a conventional alkylation or acylation reactions from compounds **3**.

## 3. Results and discussion

Thermal and mesomorphic properties of compounds in series I and II are gathered in tables 1 and 2, respectively. A graphical representation of the mesomorphic behaviour as a function of the number of

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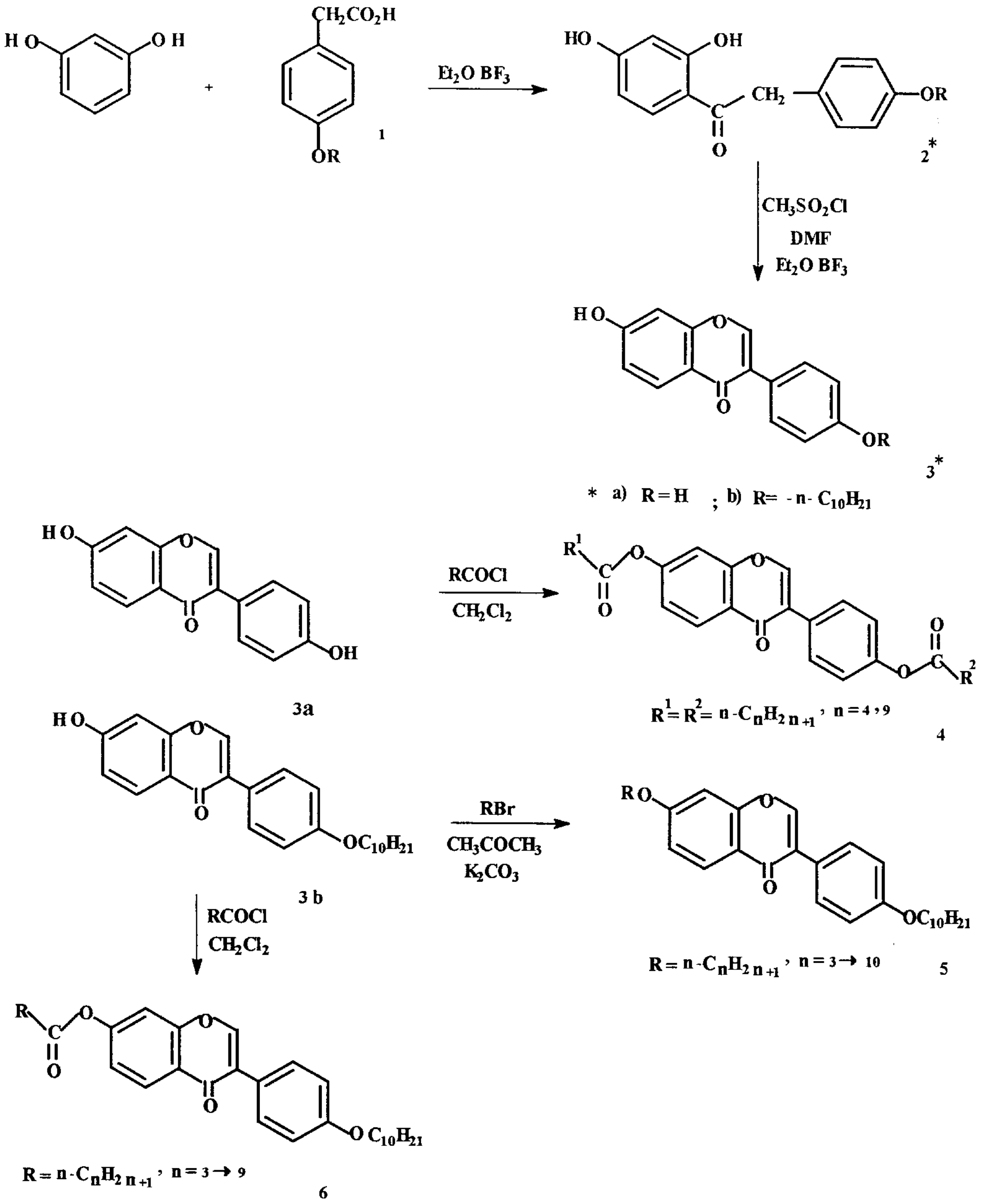


Scheme

carbon atoms in the lateral chain for both series is also presented (figures 1 and 2). The assignment of the mesophase type was made on the basis of the textures observed by polarizing optical microscopy [6]. The nematic textures were marbled or schlieren type and the texture at the clearing point was characteristic of the nematic–isotropic transition. The SmA phase of compound 5 was characterized by the formation of batonnets that colaesce to form a fan-shaped texture. Mechanical stress on such a sample leads to the formation of a homeotropic texture. On cooling the texture during the transition SmA → SmC a schlieren effect was obtained from a homeotropic SmA texture.

Compounds in series I, with two alkyloxy terminal chains, display nematic, smectic A and smectic C mesomorphism. The derivative with $n=3$ shows only a nematic mesophase. This phase is also present for the derivative with $n=4$ and is not observed for members with $n \geqslant 5$. From $n=4$ onwards, all the compounds exhibit smectic A and smectic C mesophases. The former disappears for $n=10$. This mesomorphic behaviour is usually found in the study of homologous series of

Table 1. Transition temperatures and enthalpies for 7-alkyloxy-3-(4-*n*-decyloxyphenyl)-4H-1-benzopyran 4-one (**5**).

| Compound $R = -nC_nH_{2n+1}$ | Transition | Temperature/ °C | ΔH/ kJ mol$^{-1}$ |
|---|---|---|---|
| **5a** ($n = 3$) | $Cr_1$–$Cr_2$ | 80.3 | 1.6 |
| | $Cr_2$–N | 104.3 | 30.2 |
| | N–I | 132.6 | 0.9 |
| **5b** ($n = 4$) | Cr–SmC | 107.0 | 28.0 |
| | SmC–SmA | 113[a] | |
| | SmA–N | 137.9 | |
| | N–I | 143.8 | 1.9 |
| **5c** ($n = 5$) | Cr–SmC | 112.0 | 28.1 |
| | SmC–SmA | 137.0 | |
| | SmA–I | 142.3 | 4.5 |
| **5d** ($n = 6$) | Cr–SmC | 117.2 | 31.6 |
| | SmC–SmA | 125[a] | |
| | SmA–I | 148.5 | 6.3 |
| **5e** ($n = 7$) | Cr–SmC | 120.4 | 35.0 |
| | SmC–SmA | 134.9 | |
| | SmA–I | 146.1 | 7.7 |
| **5f** ($n = 8$) | Cr–SmC | 120.9 | 30.9 |
| | SmC–SmA | 140[a] | |
| | SmA–I | 150 | 7.4 |
| **5g** ($n = 9$) | Cr–SmC | 122.1 | 34.8 |
| | SmC–SmA | 144[a] | |
| | SmA–I | 148.2 | 7.9 |
| **5h** ($n = 10$) | Cr–SmC | 119.6 | 34.9 |
| | SmC–I | 148.1 | 9.7 |

[a] Polarizing optical microscopy data.

Table 2. Transition temperatures and enthalpies for 7-acyloxy-3-(4-*n*-decyloxyphenyl)-4H-1-benzopyran 4-one (**6**).

| Compound $R = -nC_nH_{2n+1}$ | Transition | Temperature/ °C | ΔH/ kJ mol$^{-1}$ |
|---|---|---|---|
| **6a** ($n = 3$) | $Cr_1$–$Cr_2$ | 74.6 | 4.1 |
| | $Cr_2$–SmC | 89.7 | 30.9 |
| | SmC–N | 100.6 | 0.5 |
| | N–I | 140.8 | 1.5 |
| **6b** ($n = 4$) | Cr–SmC | 82.3 | 42.8 |
| | SmC–N | 121.0 | 0.9 |
| | N–I | 137.9 | 1.8 |
| **6c** ($n = 5$) | $Cr_1$–$Cr_2$ | 70.0 | 5.1 |
| | $Cr_2$–SmC | 94.1 | 30.6 |
| | SmC–N | 135.4 | 0.9 |
| | N–I | 143.0 | 3.0 |
| **6d** ($n = 6$) | Cr–SmC | 95.3 | 32.7 |
| | SmC–I | 140.2 | 8.0 |
| **6e** ($n = 7$) | Cr–SmC | 95.4 | 31.2 |
| | SmC–I | 146.3 | 8.6 |
| **6f** ($n = 8$) | Cr–SmC | 98.5 | 34.7 |
| | SmC–I | 146.7 | 9.4 |
| **6g** ($n = 9$) | Cr–SmC | 97.8 | 37.1 |
| | SmC–I | 147.8 | 11.0 |

calamitic liquid crystals. The thermal mesomorphic range is similar for all derivatives (*c*. 30°C) and, with the exception of derivative with $n = 5$, members with short alkyloxy chains have mostly a smectic A behaviour whilst members with long chains display mainly a smectic C phase.

The introduction of a carbonyl group in the linkage between the flexible terminal chain and the central core (series II) gives rise to a change in the mesomorphic behaviour compared to series I. As can be seen in table 2 and figure 2, compounds in series II exhibit nematic and

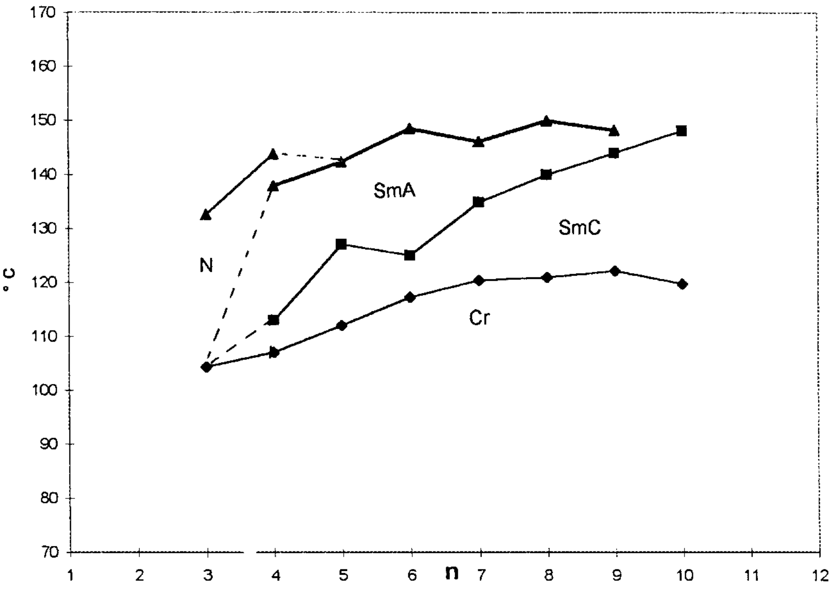


Figure 1. Mesomorphic behaviour as a function of number of carbon atoms in the terminal alkoxy chain for compounds in series I.

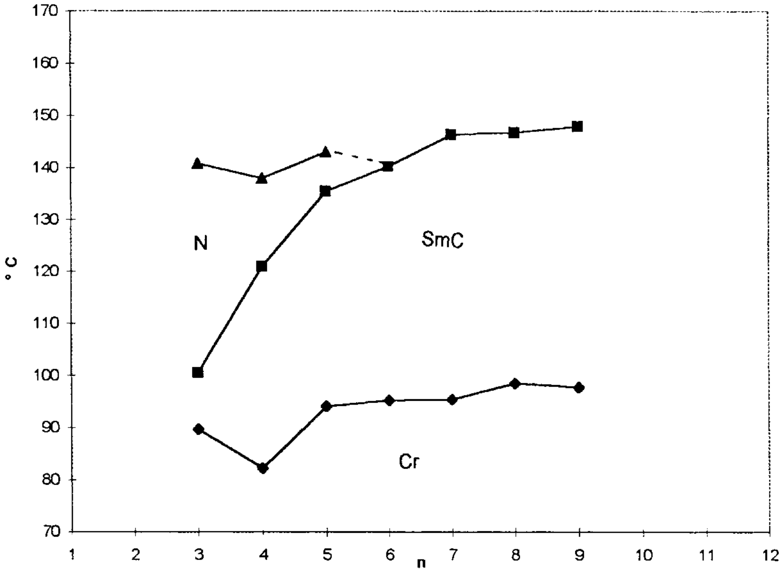


Figure 2. Mesomorphic behaviour as a function of number of carbon atoms in the terminal acyl chain for compounds in series II.

smectic C mesophases. No smectic A mesophases are observed. The melting points of compounds in series II are slightly lower (*c.* 10°C) than those of series I, whilst the clearing points are almost the same. Therefore, the mesomorphic range is also almost constant along the series and slightly larger than for series I. It seems clear that the introduction of a lateral dipolar moment associated with the carbonyl group favours the intermolecular interactions giving rise to a tilted arrangement to the molecules in the smectic layers [7]. This molecular packing is spacially stabilized as it breaks only to give a nematic or isotropic phase without going through an intermediate orthogonal layered arrangement (SmA). The occurrence of a tilted smectic C mesophase in the members of both series opens an interesting possibility for further studies with this type of compound, such as the introduction of a chiral terminal alkyl chain in the calamitic structure in order to obtain chiral mesophases (cholesteric, tilted smectic) which may exhibit interesting electrooptical properties.

## 4. Experimental

The purity of the compounds was evaluated by thin layer chromatography and elemental analysis (see table 3). The structures were confirmed by $^1$H NMR, $^{13}$C NMR (Bruker AC-250P) and FTIR (Nicolet 550)

Table 3. Elemental analysis of 7-alkyloxy-3-(4-*n*-decyloxyphenyl)-4H-1-benzopyran 4-one (series I) and 7-acyloxy-3-(4-*n*-decyloxyphenyl)-4H-1-benzopyran-4-one (series II) compounds.

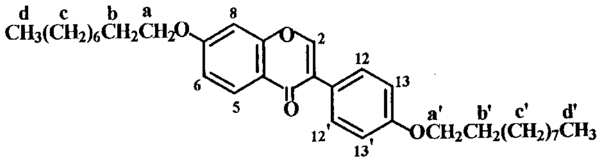
| | | C/% | | H/% | |
|---|---|---|---|---|---|
| | Empirical formula | Calculated | Found | Calculated | Found |
| $R = -OC_nH_{2n+1}$, $n = 3$ | $C_{28}H_{36}O_4$ | 77.06 | 76.84 | 8.26 | 8.45 |
| $n = 4$ | $C_{29}H_{38}O_4$ | 77.33 | 77.11 | 8.44 | 8.89 |
| $n = 5$ | $C_{30}H_{40}O_4$ | 77.59 | 77.15 | 8.62 | 8.87 |
| $n = 6$ | $C_{31}H_{42}O_4$ | 77.82 | 77.94 | 8.79 | 9.05 |
| $n = 7$ | $C_{32}H_{44}O_4$ | 78.05 | 77.94 | 8.91 | 8.49 |
| $n = 8$ | $C_{33}H_{46}O_4$ | 78.26 | 78.07 | 9.09 | 9.01 |
| $n = 9$ | $C_{34}H_{48}O_4$ | 78.46 | 78.44 | 9.23 | 9.23 |
| $n = 10$ | $C_{35}H_{50}O_4$ | 78.65 | 78.42 | 9.36 | 9.16 |
| $R = -OCOC_nH_{2n+1}$, $n = 3$ | $C_{29}H_{36}O_5$ | 75.00 | 74.98 | 7.75 | 7.65 |
| $n = 4$ | $C_{30}H_{38}O_5$ | 75.31 | 75.28 | 7.94 | 7.95 |
| $n = 5$ | $C_{31}H_{40}O_5$ | 75.61 | 75.51 | 8.13 | 8.17 |
| $n = 6$ | $C_{32}H_{42}O_5$ | 75.89 | 75.93 | 8.30 | 8.23 |
| $n = 7$ | $C_{33}H_{44}O_5$ | 76.15 | 75.98 | 8.46 | 8.68 |
| $n = 8$ | $C_{34}H_{46}O_5$ | 76.40 | 76.01 | 8.61 | 8.56 |
| $n = 9$ | $C_{35}H_{48}O_5$ | 76.64 | 76.44 | 8.76 | 8.94 |

spectra. Reported chemical shift values for isoflavone [8, 9], correlation spectra $^{1}H/^{13}C$ and Dept spectra were used to the assign the chemical shifts in $^{13}C$ spectra of the synthesized compounds. Table 4 contains spectroscopy data. The textures of the mesophases were studied with a Leitz Ortholux-Pol polarizing microscope, equipped with a Mettler FP 52 hot stage. Temperatures and enthalpies of transition were determined by differential scanning calorimetry using a Perkin Elmer DSC-7 calorimeter with a heating and cooling rate of 10°C min$^{-1}$. The apparatus was calibrated with indium at 5°C min$^{-1}$ (156.6°C, 28.45 J g$^{-1}$).

### 4.1. *4-n-Decyloxyphenylacetic acid (1)*

To a solution containing 25 g (164.3 mmol) of 4-hydroxyphenylacetic acid in 50 ml of methanol, 18.44 g (328.6 mmol) of potassium hydroxide in 50 ml of methanol was added, cooling with an ice bath during the addition. The ice bath was removed and 34 ml (164.5 mmol) of *n*-decyl bromide were added and the

Table 4. $^{1}H$ and $^{13}C$ NMR spectral data of the compounds **5h** and **6g**, and their IR absorption.



Chemical shifts of $^{1}H$ NMR (CDCl$_3$)

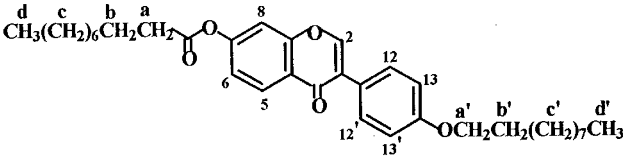
| 2 | 5 | 6 | 8 | 12-12′ | 13-13′ | a | a′ | b+ b′ | c+ c′ | d+ d′ |
|---|---|---|---|---|---|---|---|---|---|---|
| 7.89(s) | 8.19(d)<br>$J_{56}=8.9$ | 6.97(dd)<br>$J_{65}=8.8$<br>$J_{68}=2.4$ | 6.82(d)<br>$J=2.3$ | 7.48(d)<br>$J=8.7$ | 6.95(d)<br>$J=8.8$ | 4.04(t) | 3.98(t) | 1.7–1.9 m | 1.2–1.6 m | 0.88(t) |

Chemical shifts of $^{13}C$ NMR (CDCl$_3$)

| 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12-12′ | 13-13′ | 14 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 151.96 | 124.04 | 175.86 | 127.70 | 114.83 | 163.53 | 100.56 | 157.92 | 118.26 | 124.88 | 130.06 | 114.54 | 159.16 |

Alkyl chains = 68.72, 68.09 (CH$_2$ O); 31.89; 29.54; 29.39; 29.31; 28.96; 26.04; 25.96; 22.66; 14.08.

IR (KBr) cm$^{-1}$ = 1632 (C=O); 1604 (C=C); 1266 (C–O)



Chemical shifts of $^{1}H$ NMR (CDCl$_3$)

| 2 | 5 | 6 | 8 | 12-12′ | 13-13′ | a | a′ | b+ b′ | c+ c′ | d+ d′ |
|---|---|---|---|---|---|---|---|---|---|---|
| 7.96(s) | 8.31(d)<br>$J_{56}=8.7$ | 7.14(dd)<br>$J_{65}=8.7$<br>$J_{68}=2.0$ | 7.28(d)<br>$J_{8.6}=2.0$ | 7.48(d)<br>$J=8.6$ | 6.96(d)<br>$J=8.7$ | 2.60(t) | 3.98(t) | 1.6–1.9 m | 1.2–1.5 m | 0.89(t) |

Chemical shifts of $^{13}C$ NMR (CDCl$_3$)

| 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12-12′ | 13-13′ | 14 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 152.48 | 123.52 | 175.69 | 127.74 | 119.37 | 156.61 | 110.80 | 154.5 | 122.2 | 125.15 | 130.0 | 114.55 | 159.27 |

Alkyl chains = 171.50 (COO); 68.04 (CH$_2$ O); 34.6 (CH$_2$ CO); 31.82; 29.54; 29.37; 29.21; 29.04; 26.01; 24.76; 22.64; 14.07.

IR (KBr) cm$^{-1}$ = 1762.8 (C=O ester); 1631.3 (C=O) mixture was heated at reflux for 18 h. The solvent was removed and the mixture neutralized with 10% aqueous hydrochloric acid. The solid was filtered off, washed thoroughly with water and recrystallized from methanol. M.p. 74–80°C, yield 60%. An analytical sample of **1** was obtained by column chromatography on silica gel using a hexane/ethyl acetate 9 : 1 mixture as eluent; M.p. 80–81°C.

### 4.2. *1-(2,4-Dihydroxyphenyl)-2-(4-hydroxyphenyl)-ethanone (**2a**)*

This was prepared according to the method described for **2b**, using 4-hydroxyphenylacetic acid. The product was purified by column chromatography on silica gel using hexane/ethyl acetate 7 : 3 M.p. 180–185°C, reported m.p. 183–184°C [5].

### 4.3. *1-(2,4-Dihydroxyphenyl)-2-(4-n-decyloxyphenyl)-ethanone (**2b**)*

A mixture containing 5 g (17.12 mmol) of 4-*n*-decyloxyphenyl acetic acid and 1.89 g (17.18 mmol) of 1,3-dihydroxybenzene in 43 ml of freshly distilled $BF_3.Et_2O$, was heated under nitrogen atmosphere at 70–75°C for 4 h, by which time the mixture was solubilized. After cooling, a yellow solid appeared; the mixture was poured into ice–water mixture, filtered and the product was purified by column chromatography on silica gel using hexane/ethyl acetate 9 : 1 as eluent. M.p. 103–105°C, yield 50–65%.

$\delta$H ($CDCl_3$) 12.74 (1H, s, 2-OH), 7.72 (1H, d, $J = 8.2$, 6-H), 7.15 (2H, d, $J = 8.4$, 2′,6′-H), 6.85 (2H, d, $J = 8.5$, 3,5-H), 6.56 (1H, s, 4-OH), 6.35 (1H, d, d, $J_{53} = 2.2$, $J_{56} = 8.0$, 5-H), 6.34 (1H, s, 3-H). $\delta$C ($CDCl_3$) 203.48 (CO), 165.59 (C-4), 162.89 (C-2), 159.52 (C-4′), 134.12 (C-6), 131.20 (C-2′,6′), 127.83 (C-1′), 114.70 (C-3′,5′), 113.47 (C-1), 108.81 (C-5), 103.65 (C-3), 68.19 ($O–CH_2$), 44.17 ($COCH_2$), 31.86, 29.52, 29.34, 29.25, 26.01, 22.62 ($C–(CH_2)_8$), 14.04 ($C–CH_3$). IR (KBr) $cm^{-1}$ 3157.14 (OH), 1625.3 (CO).

### 4.4 *7-Hydroxy-3-(4-hydroxyphenyl)-4H-1-benzopyran-4-one (**3a**)*

This compound was synthesized using the procedure described for **3b**, with **2b** as starting material. The solid was filtered off and recrystallized from methanol. M.p. 340°C, the same as reported [10].

### 4.5. *7-Hydroxy-3-(4-n-decyloxyphenyl)-4H-1-benzopyran-4-one (**3b**)*

In a three necked flask equipped with a magnetic bar stirrer, thermometer, air condenser and nitrogen atmosphere, 5 g (13.0 mmol) of compound **2a** in 33 ml $BF_3.Et_2O$ were placed and 20 ml of dry dimethylformamide were slowly added (exothermic reaction). The mixture was heated to 50°C and 3.5 ml (44.9 mmol) of methane sulfonyl chloride in 2 ml of DMF were slowly added. The mixture was kept at 75–80°C for 1.5 h, cooled to room temperature and poured into ice–water mixture. The product was filtered off and purified by column chromatography on silica gel using hexane/ethyl acetate 9 : 1 as eluent. M.p. 165–167°C, yield 45–60%.

$\delta$H ($CDCl_3$) 8.96 (1H, s, 7-OH), 8.00 (1H, d, $J = 8.8$, 5-H), 7.81 (1H, s, 2-H), 6.82 (1H, dd, $J_{65} = 8.7$, $J_{68} = 2.1$, 6-H), 7.34 (1H, d, $J = 8.7$, 12-H), 6.82 (1H, d, $J = 8.7$, 13-H), 6.73 (1H, d, $J = 2.1$, 8-H), 3.84 (2H, t, $O–CH_2$), 1.66 (2H, m, $O–CH_2–\underline{C}H_2$), 1.1–1.4 (14H, m, $(–CH_2)_7$), 0.78 (3H, t, $–CH_3$). $\delta$C ($CDCl_3$) 176.99 (C-4), 162.25 (C-7), 158.28 (C-9), 152.76 (C-2), 130.26 (C-12). 127.87 (C-5), 124.94 (C-11), 123.85 (C-3), 117.66 (C-10), 115.68 (C-6), 102.92 (C-8). Alkyl chain 68.27 ($O–CH_2$), 31.90, 29.57, 29.41, 29.30, 26.07, 22.64, 14.02. IR (KBr) $cm^{-1}$ 3236 (OH), 1625 (C=O).

### 4.6. *7-Acyloxy-3-(4-acyloxyphenyl)-4H-1-benzopyran-4-one (**4**)*

To a mixture of 0.3 g (1.18 mmol) of 7,4′-dihydroxy-isoflavone (**2a**), 24 ml of dichloromethane and 0.2 ml of triethylamine, 3.4 mmol of the acid chloride was added. The mixture was stirred for 6 h at room temperature, then the solvent was removed and 25 ml of diethyl ether were added to the residue; triethylamonium chloride was filtered off and the product was purified by chromatography using hexane/ethyl acetate 9 : 1 as eluent. It was finally recrystallized from ethanol. Yield 45–50%.

Compound **4** ($n = 5$), elemental analysis: found C 71.35, H 6.28; calculated for $C_{25}H_{26}O_6$ C 71.09, H 6.16%. DSC and microscope data: Cr–N = 103.62°C ($\Delta H$ = 21.42 kJ $mol^{-1}$); N–I = 148.92°C ($\Delta H$ = 0.71 kJ $mol^{-1}$). Second cooling: I–N = 147.91°C; N–SmC = 96.74°C; SmC–Sm = 80.21°C; Sm–Cr = 75.59°C.

Compound **4** ($n = 9$), $\delta$H ($CDCl_3$) 8.21 (1H, d, $J = 8.7$, 5-H), 7.89 (1H, s, 2-H), 7.48 (1H, d, $J = 8.5$, 12-H), 7.20 (1H, d, $J = 1.9$, 8-H), 7.06 (1H, dd, $J_{65} = 8.6$ $J_{6,8} = 1.9$, 6-H), 7.06 (1H, d, $J = 8.6$, 13-H), 2.6–2.8 (4H, m, $COCH_2$), 2.4–2.6 (4H, m, $COCH_2\underline{C}H_2$), 1.1–1.4 (24H, m, $(–CH_2)_6$), 0.8 (6H, t, $–CH_3$). $\delta$C ($CDCl_3$) 175.24 (C-4), 156.53 (C-7), 154.60 (C-9), 153.04 (C-2), 150.74 (C-14). 128.96 (C-11), 127.65 (C-5), 124.61 (C-3), 122.08 (C-10), 121.58 (C-13), 119.49 (C-6), 110.78 (C-8). Acyl chains 172.05 and 171.24 (C=O), 34.28, 31.75, 29.30, 29.14, 28.99, 24.83, 24.68, 22.56, 13.99. IR (KBr) $cm^{-1}$ 1745.6 (COO), 1647.0 (C=O). Elemental analysis: found C 74.65, H 8.50; calculated for $C_{35}H_{46}O_6$ C 74.73, H 8.18%. DSC and microscope data: Cr–SmC = 91.08°C ($\Delta H$ = 27.34 kJ $mol^{-1}$); SmC–I = 158.02°C ($\Delta H$ = 10.12 kJ $mol^{-1}$)

### 4.7. *7-Alkyloxy-3-(4-n-decyloxyphenyl)-4H-1-benzopyran-4-one (**5**)*

To a mixture containing 0.4 g (1 mmol) of 7-hydroxy-4′-*n*-decyloxyisoflavone (**3b**) and 0.152 g (1.1 mmol) of potassium carbonate in 15 ml of dry acetone, 1.1 mmol of alkyl bromide was added and the mixture was heated under reflux for 6 h. The solvent was removed and the crude product purified by chromatography using hexane/ethyl acetate 9.5:0.5 as eluent. It was recrystallized from ethanol, yield 50%.

### 4.8. *7-Acyloxy-3-(4-n-decyloxyphenyl)-4H-1-benzopyran-4-one (**6**)*

These compounds were synthesized using the procedure described for compounds **4**. The crude products were twice recrystallized from ethanol, yield 55%.

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## References

[1] Adlercreutz, H., Fotsis, T., Bannwart, C., Makela, T., Wähälä, K., Hase, T. A., and Brunow, G., 1986, *J. Steroid Biochem.*, **25**, 791.

[2] Hirose, T., Tsuya, K., Nishigaki, T., Idaka, E., and Yano, S., 1989, *Liq. Cryst.*, **4**, 653.

[3] Chudgar, N. K., Bosco, R., and Shah, S. N., 1991, *Liq. Cryst.*, **10**, 141.

[4] Chudgar, N. K., Parekh, M. K., Madhavrao, S. S., and Sharma H. C., 1995, *Liq. Cryst.*, **19**, 807.

[5] Wähälä, K., and Hase, T. A., 1991, *J. chem. Soc. Perkin Trans. I*, 3005.

[6] Richter, D., 1980, *Textures of Liquid Crystals* (Leipzig: VEB Deutcher Verlag Für Grundstoffindustrie).

[7] Barberá, J., Marcos, M., and Serrano, J. L., 1987, *Mol. Cryst. liq. Cryst.*, **149**, 225.

[8] Pelter, A., Ward, R. S., and Bass, R. J., 1978, *J. chem. Soc. Perkin Trans. I*, 666.

[9] Jha, H. C., Zilliken, F., and Breitmaier, E., 1980, *Can. J. Chem.*, **58**, 1211.

[10] Adlercreutz, H., Musey, P. I., Fotsis, T., Bannward, C., Wähälä, K., Mäkalä, T., Brunow, G., Hase, T., 1986, *Clin. Chim. Acta*, **158**, 147.