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Synthesis and mesomorphic properties of three-benzene-ring-containing banana-shaped liquid crystals

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A homologous series of three-benzene-ring-containing banana-shaped compounds, N,N-bis(4-alkylcarbonyloxybenzylidene)benzene-1,3-diamine, were synthesised by esterifying aliphatic acids with the three-benzene-ring bent core 1,3-phenylene-bis(4-hydroxybenzylideneamine). As the number of carbon atoms in the aliphatic acids increased from 1 to 12, 12 novel banana-shaped compounds resulted with increasing lengths in their terminal tails. The mesomorphic properties of this homologous series of three-benzene-ring-containing banana-shaped compounds were characterised by means of polarised optical microscopy, differential scanning calorimetry, X-ray diffraction and electro-optical studies. Our results have demonstrated that this series of banana-shaped compounds can form mesophases, although each of their bent cores contains only three benzene rings.

Keywords: banana-shaped liquid crystal; three-benzene-ring; bent core; mesophase; phase transition temperature

1. Introduction

Liquid crystals (LCs) have been vigorously investigated for about half a century and numerous important applications have been developed for them in modern technologies. For example, LC display panels and LC-based organic light emitters are widely used in the information display industry. Being a new member in the LC family, banana-shaped LCs have attracted intense attention because of their electro-optically switchable ferro- and antiferroelectricity although the individual molecules are achiral [1–5]. The relationship between the molecular structures and their mesomorphic properties is one of the most intensively studied topics for banana-shaped LCs. To study the influence of structural variations on mesomorphic properties and transition temperatures, different parts of the banana-shaped molecules have been varied and more than 1000 banana-shaped mesogens have been synthesised in the past 14 years [6–10]. For instance, banana-shaped compounds with different central cores, with different linkages in the wings of the molecules, and with varying lengths of the terminal alkyl tails have been extensively studied. A brief review of the literature reveals that most of the synthesised banana-shaped LCs contain five benzene rings in their bent cores [1, 2, 6, 11, 12], and the remaining banana-shaped LCs are composed of either four benzene rings [13], six benzene rings [6, 14, 15] or seven benzene rings [6, 16, 17] in their bent cores. One question now emerges: could three-benzene-ring-containing banana-shaped compounds form mesophases? In order to answer this question, Tang *et al.* synthesised a three-benzene-ring-containing banana-shaped compound

m-bis(4-*p*-octoxysyrenyl)benzene, but their results showed that this compound did not exhibit any mesophases [18]. Recently, we synthesised another kind of three-benzene-ring-containing banana-shaped compound, 1,3-phenylene-bis(4-methoxybenzylideneamine). Our results demonstrated that it was a crystal with its melting point at about 100°C [19].

To answer the question of whether three-benzene-ring-containing banana-shaped compounds can form mesophases, a systematic investigation may be helpful. In this paper, we report the synthesis and characterisation of a series of three-benzene-ring-containing banana-shaped compounds N,N-bis(4-alkylcarbonyloxybenzylidene)benzene-1,3-diamine. Their bent cores are fixed to have three benzene rings, while their terminal tails are systematically varied in length, simply by increasing the number of carbon atoms in the tails. As the number of carbon atoms in the terminal tails increases from 1 to 12, 12 novel banana-shaped compounds are obtained. Their mesomorphic properties are characterised by means of polarised optical microscopy, differential scanning calorimetry (DSC), X-ray diffraction and electro-optical investigations. Our results have demonstrated that this series of three-benzene-ring-containing banana-shaped compounds can form mesophases as the four-, five-, six- and seven-benzene-ring containing banana-shaped compounds do.

2. Experimental details

The homologous series of three-benzene-ring-containing banana-shaped compounds N,N-bis(4-

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alkylcarbonyloxybenzylidene)benzene-1,3-diamine were synthesised in our laboratory. For brevity, these banana-shaped compounds were denoted as C_n , where n was the number of carbon atoms in each of the two identical terminal chains. Analytical reagents such as *m*-phenylenediamine, the 12 kinds of aliphatic acids (i.e. formic acid, acetic acid, propionic acid, butyric acid, . . . , dodecanoic acid), 4-hydroxybenzaldehyde, 1,3-dicyclohexylcarbodiimide (DCC) and dimethylaminopyridine (DMAP) were purchased either from Alfa Aesar (Tianjin, China) or from Sinopharm Chemical Reagents Co (Shanghai, China). These reagents were used as received.

The synthesis of the homologous series of banana-shaped compounds was completed in two steps. Here we take the banana-shaped compound C_9 as an example to illustrate the details of its synthesis. The other banana-shaped compounds (C_7 – C_8 and C_{10} – C_{12}) were synthesised using a similar method. In the first step, the intermediate 1,3-phenylene-bis(4-hydroxybenzylideneamine) was synthesised. In a round-bottomed flask (250 ml), *m*-phenylenediamine (0.01 mol) and 4-hydroxybenzaldehyde (0.02 mol) were dissolved in the solvent of absolute ethyl alcohol (50 ml), and then a catalytic amount of acetic acid was added. The mixture was stirred at room temperature for 24 h. After purification and crystallisation of the mixture, the intermediate 1,3-phenylene-bis(4-hydroxybenzylideneamine) was obtained. In the second step, nonanoic acid was esterified with the intermediate to synthesise the banana-shaped compound C_9 . The intermediate 1,3-phenylene-bis(4-hydroxybenzylideneamine) (0.01 mol) and nonanoic acid (0.02 mol) were added into the solvent of dichloromethane (50 ml). In the presence of catalysts DCC and DMAP, the esterification was completed at room temperature after having been stirred for 24 h. The precipitates were filtrated and crystallised from absolute ethyl alcohol. After drying in an oven for about 40 h, the target compound C_9 was obtained in the form of white powder. Both the ^1H NMR and the ^{13}C NMR spectra were obtained with a Bruker DRX500 spectrometer. Tetramethylsilane was employed as an internal marker in the NMR characterisations. Element analysis was carried out using a Vario EL III (Germany). For C_9 , its yield was about 31%.

The phase transitions of the synthesised compounds were characterised with a differential scanning calorimeter (Nanjing Dazhan, China). As fast heating and cooling rates could introduce significant temperature differences between the temperature sensor and the sample, we used slow heating rates and cooling rates (as slow as $0.5^\circ\text{C min}^{-1}$) in our DSC characterisations. Standard aluminium pans were used as containers in order to improve the accuracy of our DSC

measurements. For structural characterisation, X-ray diffraction studies were performed on a commercially available X-ray diffractometer (Tongda Instruments, China). The X-ray wavelength of 1.5405 \AA was used. For the optical and electro-optical studies, the synthesised compounds C_7 – C_{12} were placed into LC cells using the capillary method. The gap of the cells was measured to be about $10 \mu\text{m}$. Glass plates were coated with transparent and electrically conductive indium tin oxide layers. An alternating voltage with triangular waveform was applied to the cells. The textures of the banana-shaped compounds C_7 – C_{12} were recorded with a polarised optical microscope (Alpha Technologies, China). A hot stage was used to regulate the temperature of the banana-shaped compounds.

3. Results and discussion

Figure 1 depicts the synthetic scheme and the molecular structures of the homologous series of the three-benzene-ring-containing banana-shaped compounds C_7 – C_{12} . As shown in Figure 1, this series of banana-shaped compounds share the following characteristics: (1) they have the same bent core in which the number of benzene rings is only three; (2) via a Schiff base each benzene ring in the two branched wings is linked to the central benzene ring at its 1 or 3 positions; (3) through an ester linkage each of the aliphatic tails in a banana-shaped molecule is bonded to the 4 position of the terminal benzene ring in the branched wings; and (4) the length of the terminal tails gets longer as the number of carbon atoms in the terminal tails increases from 1 to 12. Our survey of the literature has demonstrated that this series of banana-shaped compounds are novel in the aspects of their molecular structures because they contain only three benzene rings [6–14].

The DSC thermograms are shown in Figure 2 for the banana-shaped compounds C_7 – C_{12} when they underwent the second cyclic heating scans (left panel) and the second cyclic cooling scans (right panel). As shown in Figure 2, each member in the series of homologues exhibits two endothermic peaks in its heating scan and two exothermic peaks in its cooling scan. For example, the two endothermic peaks in the DSC thermogram for C_2 (in the left panel of Figure 2) indicate that this banana-shaped compound experienced its solid→mesomorphic phase transition at 217°C and then underwent its mesomorphic→isotropic phase transition at 234°C . The two exothermic peaks in the DSC thermogram for C_2 (in the right panel of Figure 2) demonstrate that this compound experienced its isotropic→mesomorphic phase transitions at 230°C and then underwent its mesomorphic→solid phase transition at 188°C . Consequently, our results in

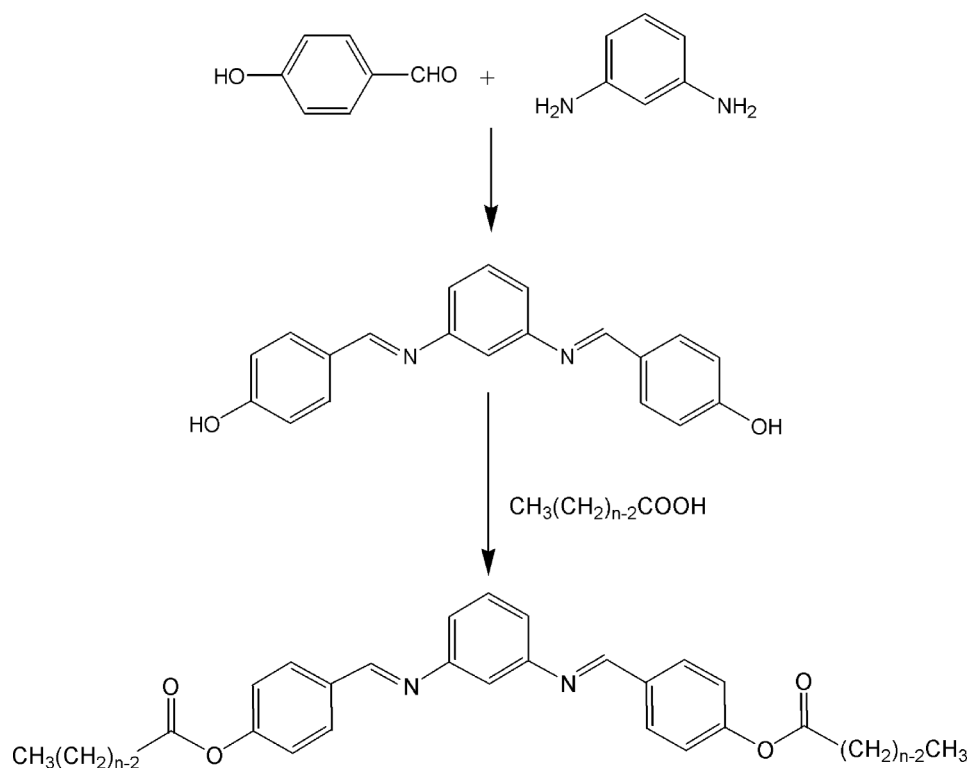


Figure 1. Synthetic scheme and molecular structures of the three-benzene-ring-containing banana-shaped compounds N,N-bis(4-alkylcarboxybenzylidene)benzene-1,3-diamine.

Figure 2 indicate that this series of three-benzene-ring-containing banana-shaped compounds can form mesophases.

To provide a quantitative description of the phase transition temperatures, we list the phase transition temperatures and enthalpy (kJ mol^{-1} in brackets) in Table 1 for this series of three-benzene-ring-containing banana-shaped compounds. For LCs, phase transition temperatures and enthalpies are preferentially taken from the exothermic DSC thermograms on cooling scans. Here, both the temperatures and the enthalpies in Table 1 were derived from the *exothermic* DSC thermograms in the second cooling scans (right panel of Figure 2). For example, the compound C_7 has two endothermic peaks at about 213°C and 236°C in the heating scan, whereas C_7 exhibits two exothermic peaks at about 193°C and 230°C in the cooling scan. Consequently, only the data 193°C and 230°C are listed in Table 1 to represent the phase transition temperatures for C_7 . As shown in Table 1, the melting points and clearing points have no clear dependence on the number of carbon atoms in the terminal tails of the banana-shaped compounds. As documented in the literature, the electro-optical properties and transition temperatures of some rod-like LCs exhibit odd–even dependence on the length of the alkyl chain, although

the odd–even effect is not universal for all LCs. In our case, it is important for us to know whether the transition temperatures of our banana-shaped compounds show the odd–even effect because the structure–property relationship has been a focus in this field. Our experimental results, shown in Table 1, demonstrate that neither the melting points nor the clearing points exhibited an odd–even effect as the number of carbon atoms in the terminal tails increases from 1 to 12. As for why the banana-shaped LCs do not exhibit odd–even effects, this is one of the many questions to be answered in the future, because the physical reasons for the odd–even effects are not completely understood, even for the well-studied rod-like LCs.

As shown in Table 1, the clearing enthalpy for compounds with $n = 9, 10$ and 12 are quite high, while that for $n = 11$ is very high. In order to understand the high enthalpies, we re-characterised the banana-shaped compounds with DSC (model DSC-7, Perkin Elmer, USA). Our re-examination has shown that the high enthalpies are reproducible. Therefore, we believe that the differences in the molecular structures of C_9 – C_{12} are responsible for their high enthalpies.

When subjected to heating, the banana-shaped homologues exhibit quite similar optical properties.

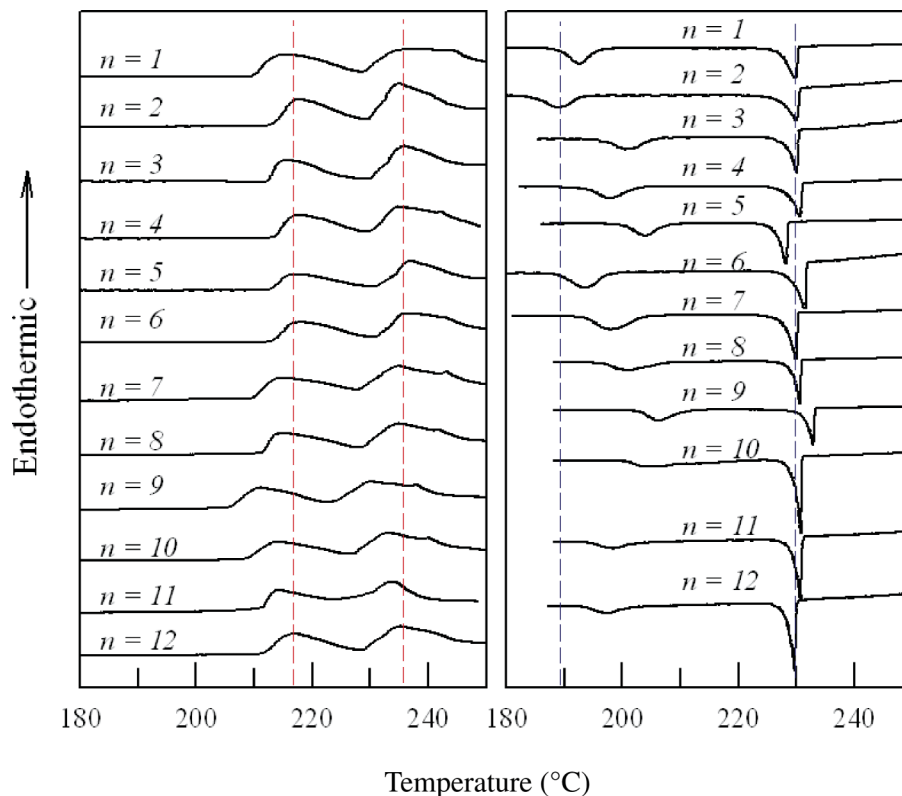


Figure 2. Differential scanning calorimetry thermograms of the three-benzene-ring-containing banana-shaped compounds N,N -bis(4-alkylcarbonyloxybenzylidene)benzene-1,3-diamine. All the data were taken in the second cyclic heating scans (left panel) and the second cyclic cooling scans (right panel).

The typical LC textures observed by polarised optical microscopy in heating scans are shown in Figure 3 for the banana-shaped compounds C_2 , C_8 and C_{10} . The textures in Figure 3 were taken at about 10°C below their mesophase \rightarrow isotropic phase transition temperatures. For example, the compound C_2 has two endothermic peaks in the DSC curve at about 217°C

and 234°C . That is, the image of the texture of C_2 was taken at 224°C in the heating scan. Similarly, the images of textures of C_8 and C_{10} were taken at 224°C and 223°C , respectively, because the mesophase \rightarrow isotropic transition temperatures of C_8 and C_{10} were recorded to be 234°C and 233°C in their heating scans. As shown by the panels in Figure 3, all three

Table 1. Transition temperatures ($^\circ\text{C}$) and enthalpy (kJ mol^{-1} in brackets) of the three-benzene-ring banana-shaped compounds as a function of the number of carbon atoms in the terminal tails. The data were derived from the cooling thermograms shown in the right panel of Figure 2.

n	K		Mesophase phases	I	
1	•	193 (12.2)	•	230 (16.8)	•
2	•	188 (14.6)	•	230 (25.9)	•
3	•	201 (10.8)	•	230 (23.2)	•
4	•	198 (12.7)	•	231 (22.9)	•
5	•	204 (11.5)	•	228 (14.6)	•
6	•	193 (14.7)	•	231 (22.5)	•
7	•	197 (18.0)	•	230 (22.5)	•
8	•	200 (11.8)	•	231 (25.2)	•
9	•	206 (20.0)	•	233 (31.8)	•
10	•	204 (22.1)	•	231 (28.1)	•
11	•	198 (11.8)	•	231 (41.6)	•
12	•	197 (19.4)	•	229 (32.1)	•

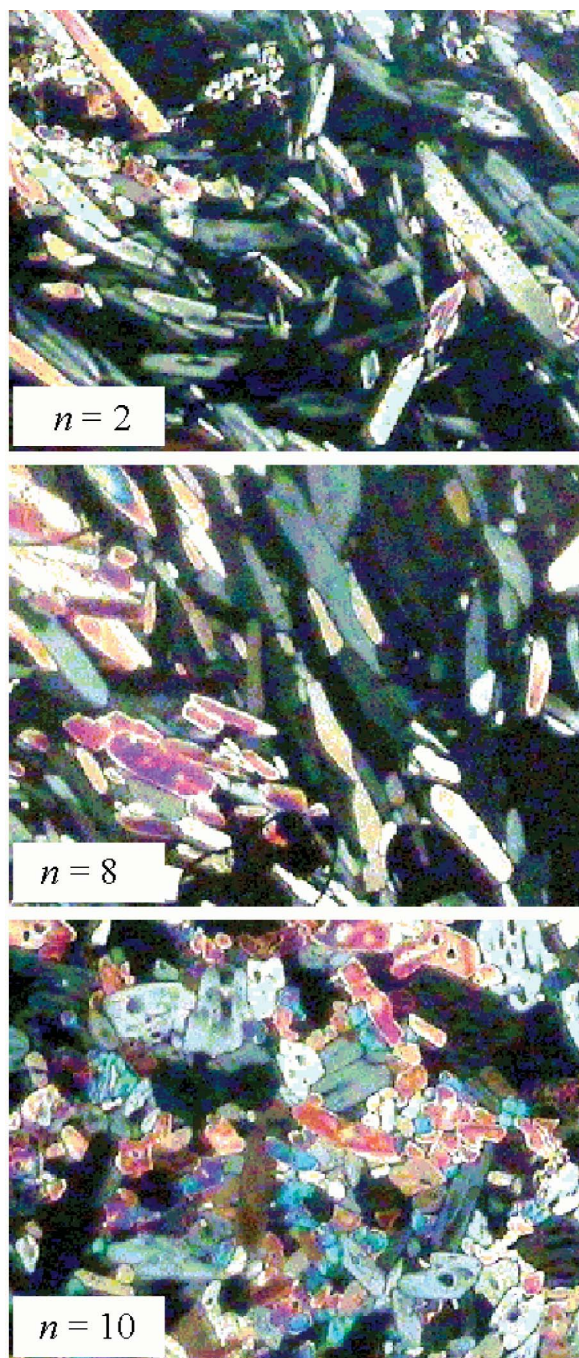


Figure 3. Typical mosaic textures observed by polarised optical microscopy in the heating scans for the three-benzene-ring-containing banana-shaped compounds C_2 , C_8 and C_{10} . The textures were recorded at about 10°C below their mesomorphic \rightarrow isotropic phase transition temperatures, i.e. the textures of C_2 , C_8 and C_{10} were recorded at about 224°C , 223°C and 224°C , respectively (colour version online).

banana-shaped compounds C_2 , C_8 and C_{10} exhibit quite similar mosaic textures, although the number of carbon atoms in their terminal tails varies from 2,

8 to 10. After characterising all this homologous series of the banana-shaped compounds, we found that all of them exhibited similar mosaic textures no matter whether n was even or odd.

On cooling the isotropic liquids of the banana-shaped compounds C_1 – C_{12} , the mesophases grow out from their isotropic phases as dendritic nuclei which coalesce to mosaic textures. Figure 4 shows the typical LC textures observed by polarised optical microscopy in cooling scans for the homologous series of the banana-shaped compounds C_1 , C_4 , C_8 and C_{12} . The textures were recorded at about 5°C below their isotropic \rightarrow mesomorphic phase transition temperatures. That is, the textures of C_1 , C_4 , C_8 and C_{12} were recorded at about 225°C , 226°C , 226°C and 224°C , respectively. As shown in Figure 4, the four panels show that the banana-shaped LCs exhibit leaf-like textures for C_1 , C_4 , C_8 and C_{12} , respectively. After characterising this homologous series of the banana-shaped compounds, we found that they all exhibited similar leaf-like textures no matter whether n was even or odd. A comparison of the textures in Figure 4 with those in Figure 3 reveals that the textures recorded in the cooling scans are different from those recorded in the heating scans.

On the basis of our experimental data, we conclude that this series of banana-shaped compounds can form mesophases. When compared with the two kinds of the banana-shaped crystals in the literature [18, 19], it is clear that the molecular structures of the banana-shaped compounds play a decisive role in the formation of mesophases. If evaluated solely on the basis of the recorded textures, the mesophases formed by this series of banana-shaped LCs look like the B_1 phase. Further characterisation with the technique of X-ray diffraction is required in order to have a comprehensive understanding of the features of the mesophases formed by the banana-shaped compounds.

X-ray diffraction is often employed to determine if mesophases are indeed of the B type for banana-shaped compounds. We performed X-ray diffraction analysis for all 12 homologues. The plot of the intensity versus diffraction angle 2θ is shown in Figure 5 for the banana-shaped compound C_6 when it was in its mesophase at 220°C . The X-ray diffraction curves were recorded upon cooling C_6 from its isotropic phase to its mesophase. Under the influences of an electric field, the samples were cycled several times through the clearing points in order to achieve monodomains for the X-ray diffraction studies, although it was difficult to obtain a monodomain of large size. When the temperature was about 220°C , the X-ray pattern shows two reflections in the low-angle region. In the higher-angle region a broad diffuse scattering indicates the liquid-like order of the

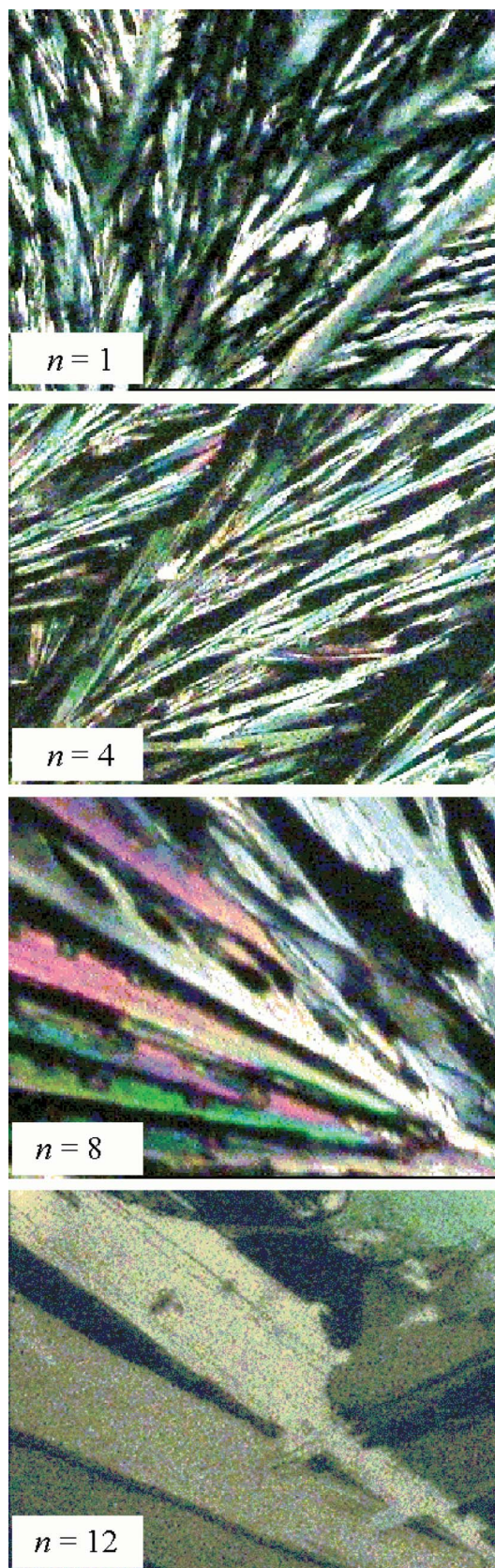


Figure 4. Typical leaf-like textures observed by polarised optical microscopy in the cooling scans for the three-benzene-ring-containing banana-shaped compounds C_1 , C_4 , C_8 and C_{12} . The textures were recorded at about 5°C below their isotropic \rightarrow mesomorphic phase transition temperatures, i.e. the textures of C_1 , C_4 , C_8 and C_{12} were recorded at about 225°C , 226°C , 226°C and 224°C , respectively (colour version online).

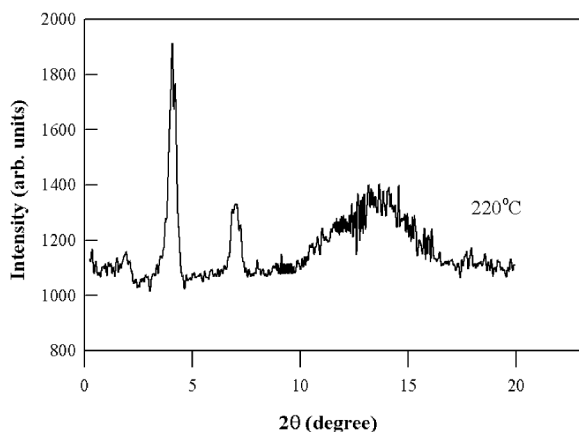


Figure 5. Plot of the intensity versus diffraction angle 2θ for the three-benzene-ring-containing banana-shaped compound C_6 . The temperature was 220°C .

molecules within the layers. All phases designated as B_1 are characterised by X-ray pattern with two or more reflections in the small-angle regions [6]. We see the two sharp reflections at low angles $2\theta = 4.1^\circ$ and 7.0° . The corresponding d -spacings are 2.1 nm and 1.26 nm, respectively. These spacings can be indexed as (002) and (103) of a two-dimensional rectangular lattice. The cell parameters in this case are $a = 4.2$ nm and $b = 3.98$ nm. According to the indexing for the B_1 phase, the two sharp peaks in Figure 5 suggest that the mesophase formed by C_6 has a two-dimensional rectangular packing.

Along with the physical properties of DSC and X-ray diffraction, the electro-optical behaviour is of particular interest and can provide some information about the structure of the banana phases. The most interesting phases are those with ferroelectric or anti-ferroelectric properties, whereas many banana-shaped compounds form two-dimensional phases, which seem to be specific for banana-shaped molecules. To obtain more information on the properties of the observed mesophase, we performed electro-optical studies on the banana-shaped compound C_6 . The electro-optical response was studied by using a triangular waveform of 30 Hz whose peak-to-peak voltage (V_{pp}) ranged from 0 to 140 V. By applying a triangular voltage up to $15 V_{pp} \mu\text{m}^{-1}$, we recorded no current peaks. The absence of the current peaks suggests that the mesophase formed by C_6 is not electrically switchable. This is understandable for a B_1 phase because this phase does not exhibit any electro-optical switching properties even at high voltages [6].

As frequently observed in banana series, the lengths of the terminal chains sometimes influence the occurrence of different B phases. In order to check if the 12 homologues show the same type of B

phase, we characterised them with X-ray diffraction and electro-optical studies. Our results show that all the 12 homologues exhibit quite similar X-ray diffraction patterns and electro-optical properties to those of C_6 . Consequently, our preliminary investigations suggest that the 12 homologues show the same type of B phase, although the carbon atom number in their terminal chains varies from 1 to 12. The exact influence of the terminal chains on the formation of the different B phases requires detailed studies, since only powder-like patterns are considered. Therefore, the identification of mesophase is rather speculative and must be proven by further experiments.

4. Summary

In conclusion, we have reported the synthesis and characterisation of a series of three-benzene-ring-containing banana-shaped compounds N,N-bis(4-alkylcarbonyloxybenzylidene)benzene-1,3-diamine.

As the number of carbon atoms in the terminal tails increases from 1 to 12, 12 novel banana-shaped compounds have been synthesised and their mesomorphic properties have been characterised by means of polarised optical microscopy, DSC, X-ray diffraction and electro-optical response studies. Our results have demonstrated that the three-benzene-ring-containing banana-shaped compounds can form mesophases if their molecular structures are appropriate.

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