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Yang-Chu Lin[a]; Kung-Lung Cheng[a]; Woan-Shiow Tzeng[a]; Chih-Lin Su[a]; Long-Je Lee[b]; Ling-Yung Wang[b]; Chun-Ming Yang[b]; Long-Li Lai[b]
[a] Union Chemical Laboratory, Industrial Research Institute, Hsinchu, Taiwan 300, ROC [b] Department of Applied Chemistry, National Chi Nan University, Puli, Taiwan 545, ROC

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Synthesis and study of *N*,*N*-disubstituted 4-[(4-aminophenyl)diazenyl]benzylidene-4′-alkylanilines

YANG-CHU LIN†, KUNG-LUNG CHENG†, WOAN-SHIOW TZENG†, CHIH-LIN SU†, LONG-JE LEE‡, LING-YUNG WANG‡, CHUN-MING YANG‡ and LONG-LI LAI*‡

†Union Chemical Laboratory, Industrial Research Institute, Hsinchu, Taiwan 300, ROC
‡Department of Applied Chemistry, National Chi Nan University, Puli, Taiwan 545, ROC

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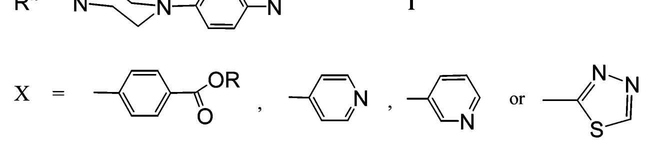
A series of *N*,*N*-disubstituted-4-[(4-aminophenyl)diazenyl]benzylidene-4′-alkylanilines (azo dyes) were synthesized from the reaction of the corresponding benzaldehyde with alkylanilines. These azo dyes exhibit nematic and SmC phases on heating. Their order parameter, photo-stability and miscibility were studied by investigation of a representative sample.

1. Introduction

A high level of interest in the application of liquid crystalline materials continues, particularly after the successful mass production of LCDs [1]. Among these materials, azo dye liquid crystals provide promising candidates for industrial application owing to their easy availability and special properties [2]. In particular, azo dyes have a reasonable dichroic ratio and are suitable materials for use in lower power consumption reflective liquid crystal devices. These include three-layered guest–host (GH) systems, which, with subtractive colour mixing of yellow, magenta and cyan, are expected to be used for developing full colour reflective displays [3].

Previously, we successfully synthesized several azo dye materials having structure **I** [4], most of which show smectic phases. However, nematic liquid crystals are more valuable in practical use, as they offer lower viscosity and faster response under applied electrical switching. Compound **II** was therefore prepared and, as expected, exhibited a nematic phase on heating [5*a*, *b*]. It is regarded that a strong polar group at the *para*-position may interrupt the layer arrangement, thus favouring the formation of a nematic phase. However, compound **II**, containing a CHO functional group, was found not to be photostable, decomposing rapidly under UV radiation. To avoid such a problem, azo dyes having structure **5** (see scheme 1) were prepared. Surprisingly, azo dyes **5a**–**d** not only exhibit the desired nematic phase on heating, but also possess good miscibility, photostability and order parameter by studying a representative sample.



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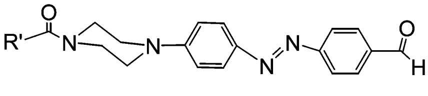

II

2. Experimental

Starting chemicals were commercially available and used without further purification. The mesogenic behaviour and phase transitions were characterized by polarizing optical microscopy (POM) and differential scanning calorimetry (Perkin-Elmer DSC 7). Powder X-ray diffraction (XRD) patterns were obtained from a Siemens D-5000 X-ray diffractometer equipped with a TTK 450 temperature controller, using Cu radiation with wavelength λ=1.5406. The absorbance of the azo dyes was recorded with a Jasco-V530 spectrometer. Compounds **5a**–**d** were prepared according to scheme 1, checked by t.l.c. for purity and characterized by ^{1}H NMR (Varian 300 MHz FT NMR spectrometer) at room temperature using $CDCl_3$ (7.24 ppm) as internal standard (s=singlet, d=doublet, t=triplet, b=broad), IR (Perkin -Elmer RX-I spectrometer; KBr, cm^{-1}) and high resolution mass spectrometry (VG70-250; EI, 70 ev).

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*Corresponding author. Email: lilai@ncnu.edu.tw

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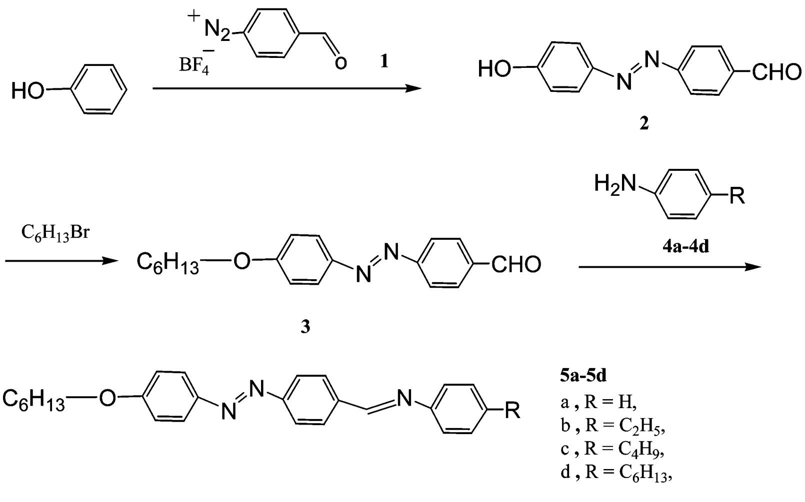


Scheme 1

2.1. *Synthesis of p-hydroxyazobenzaldehyde, 2*

Poly(aminobenzaldehyde) (1.21 g, 10 mmol) was added to HBF_4 (50 wt% in water, 8 ml), which was then diluted with hot water (about 90°C, 7 ml). $NaNO_2$ (1.03 g, 15 mmol) was gradually added at 0°C and the solution then maintained in the refrigerator overnight. The precipitated diazonium salt **1** was filtered off, washed with dry ether and then dried; it was isolated in quantitative yield. Phenol (0.94 g, 10 mmol) and potassium carbonate (2.07 g, 15 mmol) in 20 ml water was added to the previously prepared salt **1**, and the solution was stirred at room temperature for 2 h. Concentrated HCl was added and the precipitated solid was filtered off and then recrystallized from acetone/dichloromethane (1/5) to give compound **2** in almost quantative yield. **2**: m.p. 216–217°C. ^1H NMR $\delta(CDCl_3)$: 6.96(d, 2H, J=9.0 Hz, 2 × Ar–H), 7.91(d, 2H, J=9.0 Hz, 2 × Ar–H), 7.99(s, br., 4H, 4 × Ar–H), 10.07(s, 1H, CHO).

2.2. *Synthesis of p-hexyloxyazobenzaldehyde, 3*

Freshly prepared compound **2** and hexyl bromide (1.65 g, 10 mmol) were added to DMF (5 ml) containing potassium carbonate (2.07 g, 15 mmol), and the resulting mixture was heated at 100°C for 24 h. Water (200 ml) was added and the precipitated solid filtered off and washed with ethanol (5 ml × 2) to give compound **3** in 80.8% yield (3.39 g). **3**: m.p. 77–78°C [5*c*]. ^1H NMR δ ($CDCl_3$): 0.90(t, J=5.4 Hz, 3H, CH_3), 1.32–1.87(m, 8H, $4CH_2$), 4.04(t, J=5.1 Hz, 2H, Ar–OCH_2), 7.00(d, 2H, J=8.7 Hz, 2 × Ar–H), 7.94(d, 2H, J=8.7 Hz, 2 × Ar–H), 8.01(s, br, 4H, 4 × Ar–H), 10.07(s, 1H, CHO).

2.3. *Synthesis of p-hexyloxyazobenzylideneanilines, 5a–d*

Compound **3** (0.84 g, 2 mmol), followed by aniline (0.21 g, 2.2 mmol) in benzene (20 ml) were consecutively added to methanol (40 ml). The resulting mixture was heated under reflux for 3 h, while removing the generated water by a Dean–Stark apparatus. The precipitated solid was filtered off and then recrystallized from ethanol/dichloromethane (3/1); compound **5a** was thus prepared in 65.8% yield (0.50 g). **5a**: ^1H NMR δ ($CDCl_3$): 0.92(t, J=5.4 Hz, 3H, CH_3), 1.34–1.85(m, 8H, $4CH_2$), 4.06(t, J=6.6 Hz, 2H, Ar–OCH_2), 7.02(d, 2H, J=9.0 Hz, 2 × Ar–H), 7.20–7.28(m, 4H, 4 × Ar–H), 7.40(d, 2H, J=8.7 Hz, 2 × Ar–H), 7.93–7.97(m, 5H, 5 × Ar–H), 8.53(s, 1H, Ar–CHN–Ar). HRMS for $C_{25}H_{28}N_3O$ (M+1), 386.2234; found, 386.2232. IR: 3057, 2952, 2939, 2868, 2887, 1618, 1602, 1580, 1498, 1473, 1248. UV (λ_{max} in CH_2Cl_2): 377 nm.

Compounds **5b–d** were prepared similarly in comparable yields. **5b**: ^1H NMR δ ($CDCl_3$) 0.92(t, J=5.4 Hz, 3H, CH_3), 1.24–1.85(m, 11H, $4CH_2$+$1CH_3$), 2.69(quart, 2H, Ar–CH_2), 4.05(t, J=6.6 Hz, 2H, Ar–OCH_2), 7.02(d, 2H, J=9.0 Hz, 2 × Ar–H), 7.20–7.26(m, 4H, 4 × Ar–H), 7.93–7.98(m, 4H, 4 × Ar–H), 8.03(d, 2H, J=8.7 Hz, 2 × Ar–H), 8.54(s, 1H, Ar–CHN–Ar). HRMS for $C_{27}H_{32}N_3O$ (M+1), 414.2546; found, 414.2545. IR: 3026, 2955, 2934, 2871, 1622, 1602, 1583, 1499, 1474, 1248. UV (λ_{max} in CH_2Cl_2): 380 nm. **5c**: ^1H NMR δ ($CDCl_3$) 0.92(t, J=5.4 Hz, 3H, CH_3), 1.34–1.85(m, 15H, $6CH_2$+$1CH_3$), 2.64(t, J=7.5 Hz, 2H, Ar–CH_2), 4.06(t, J=6.6 Hz, 2H, Ar–OCH_2), 7.02(d, 2H, J=9.0 Hz, 2 × Ar–H), 7.20–7.26(m, 4H, 4 × Ar–H), 7.93–7.98(m, 4H, 4 × Ar–H), 8.03(d, 2H, J=8.7 Hz, 2 × Ar–H), 8.54(s, 1H, Ar–CHN–Ar). HRMS for $C_{29}H_{36}N_3O$ (M+1), 442.2856; found, 442.2858. IR: 3025, 2956, 2930, 2858, 2872, 1618, 1601, 1582, 1500, 1466, 1419. UV (λ_{max} in CH_2Cl_2): 380 nm. **5d**: ^{1}H NMR δ ($CDCl_3$) 0.92(t, J=5.4 Hz, 3H, CH_3), 1.32–1.86(m, 19H, $8CH_2$+$1CH_3$), 2.63(t, J=7.5 Hz, 2H, Ar–CH_2), 4.06(t, J=6.6 Hz, 2H, Ar–OCH_2), 7.02(d, 2H, J=9.0 Hz, 2 × Ar–H), 7.20–7.26(m, 4H, 4 × Ar–H), 7.93–7.98(m, 4H, 4 × Ar–H), 8.03(d, 2H, J=8.7 Hz, 2 × Ar–H), 8.55(s, 1H, Ar–CHN–Ar). HRMS for $C_{29}H_{36}N_3O$ (M+1), 470.3169; found, 470.3171. IR: 3025, 2927, 2932, 2854, 1622, 1602, 1582, 1498, 1472. UV (λ_{max} in CH_2Cl_2): 381 nm.

2.4. *Photostability*

Compound **5b** or compound **II** was exposed in a RPR-100 photochemical reactor which was equipped with eight 3 500 Å lamps (24 watts × 8). The concentration of the azo dye in a pair of quartz UV cuvettes was maintained at the same concentration in toluene (5×10^{-5} g ml^{-1}). One was kept in the dark for reference and the other was continuously exposed to light in the photochemical reactor. To measure the percentage decomposition of the azo dye after exposure, the exposed and reference cuvettes were simultaneously investigated by UV-Vis spectrometer. The absorption of the exposed quvette after being corrected from the reference quvette was recorded. The decaying percentage of the azo dye v.s. the exposing time was thus obtained and demonstrated in table 1.

2.5. *Order parameter*

The order parameter of azo dye **5d** was measured by dissolving 1% of the dye material in a host liquid crystalline material (ZLI-5100-100) in an ITO-glass cell with inner uniform parallel alignment (4.7 µm thickness). The dye- and liquid crystal-contains ITO-glass cell was inserted in the light path of a Jasco-V530 spectrometer and located behind a linear polarizer (figure 1). The polarizer was adjusted to the parallel alignment of the liquid crystal in the cell, and the maximum absorption for $A^{\parallel}$ (1.03) at λ_{max} (388 nm) was thus recorded. After realigning the polarizer to be orthogonal to the liquid crystal alignment in the cell, a second minimum absorption, $A^{\perp}$ (0.11) at λ_{max} (388 nm) was also obtained. The order parameter of azo dye **5d** was calculated to be 0.74 according to equation (1) [6]. To calibrate our measuring method, the order parameter of dichroic dye SI-426 was similarly measured and calculated to be 0.73, consistent with the value reported by the Mitsui company (0.74).

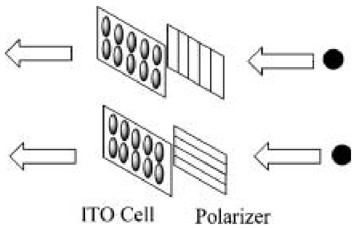


Figure 1. The simple device for measuring the order parameter of the dichroic dye.

$$S = A^{\parallel} - A^{\perp} / A^{\parallel} + 2A^{\perp} \tag{1}$$

Table 1. The decomposition percentage of compounds **5b** and **II**; the error percentage was estimated within 3% on the basis of repeated experiments (three times).

	Decomposition %	
UV exposure time/hr	**5b**	**II**
2	8.54	10.2
4	10.20	20.18
6	10.98	26.75

3. Results and discussion

3.1. *Synthesis and mesogenic study*

The desired dichroic azo dye liquid crystals were prepared according to scheme 1. The salt **1** [5*a*, *b*], prepared from poly(aminobenzaldehyde), was allowed to react with phenol to give compound **2** in almost quantitative yield. Further reaction of compound **2** with hexyl bromide gave **3** in about 80% yield; this was allowed to react with aniline **4a** to give azo dye **5a** in refluxing benzene/methanol (60 ml, 2/1) for 3 h. During the course of reaction, the reaction mixture gradually became clear and then crude solid precipitated. Recrystallization of the solid from ethanol/dichloromethane give compound **5a** in about 60% yield; it's purity was confirmed by t.l.c. and identified by ^{1}H NMR, IR spectroscopy and high resolution mass spectrometry. Compounds **5b–d** were prepared similarly. These compounds have a UV absorbance (λ_{max} in CH_2Cl_2) at about 380 nm, and a typical absorbance of compound **5d** is shown in figure 2. The absorbance is slightly different from that in ZLI-5100-100 (388 nm), which may result form the varying extend of interaction with the media. Although benzylidene aniline derivatives are easy to hydrolysis, compounds **5b–d** are reasonably stable. On checking their purity by thin layer chromatography, they are not found to decompose on silica, which may be the result of the stable conjugation of the system.

Azo dyes **5a–d** show nematic and SmC phases during heating (table 2), which were characterized from their

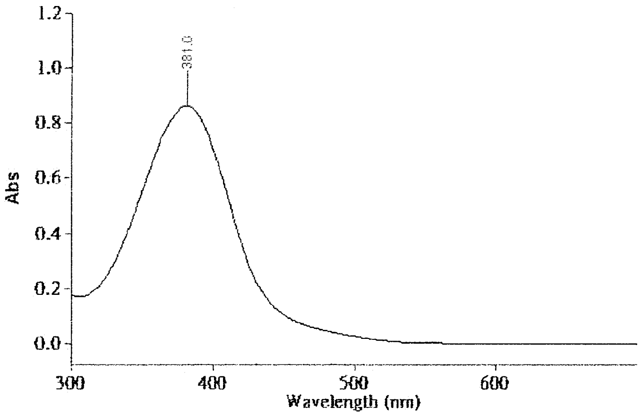


Figure 2. The UV absorbance ($\lambda_{\max}$) of compound **5d** in CH_2Cl_2.

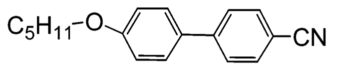
schlieren textures and the coexistence of focal-conic and schlieren textures, together with their low viscosity. Excluding **5a**, the mesogenic (N and SmC) ranges for compounds **5b–d** are about 90–110°C during thermal processing. To confirm the mesogenic phases of compounds **5a–d**, a representative compound **5a** was further studied by powder XRD. As there is no layer structure in the nematic phase range, no reflection was observed at the small angle area for compound **5a** at 170°C. However, the layer distances were measured to be 26.94, 26.33, 25.99 and 25.83 Å at 135, 130, 125 and 120°C, respectively in the SmC range. As the molecular axis of liquid crystals in the SmC phase tilts at an angle along the *Z*-axis, the tilted angle will decrease with increasing temperature, and the *d*-spacings (the fully extended molecular length on the *z*-axis) of the molecules consequently increases at elevated temperature, which is consistent with the experimental results.

Table 2. Phase transition temperature (°C) and corresponding enthalpies ($\mathrm{J\,g^{-1}}$) of compounds **5a–d**, **6** and mixture **7**, determined on 2nd DSC scans 7 at heating and cooling rates of $5^\circ\mathrm{C\,min^{-1}}$ between 50 and 240°C. Cr=crystalline, SmX=unidentified phase, SmC=smectic C phase, N=nematic phase, I=isotropic liquid.

5a	$\mathrm{SmX\ or\ Cr}\ \underset{119.1\ (5.6)}{\overset{119.8(82.5)}{\rightleftharpoons}}\ \mathrm{SmC}\ \underset{143.5\ (3.4)}{\overset{145.3\ (3.6)}{\rightleftharpoons}}\ \mathrm{N}\ \underset{184.9\ (2.1)}{\overset{186.3\ (1.8)}{\rightleftharpoons}}\ \mathrm{I}$
5b	$\mathrm{SmX\ or\ Cr}\ \underset{119.1\ (5.6)}{\overset{139.3^{a}}{\rightleftharpoons}}\ \mathrm{SmC}\ \underset{131.5\ (5.5)}{\overset{143.5}{\rightleftharpoons}}\ \mathrm{N}\ \underset{237.7\ (2.1)}{\overset{247.3\ (2.6)}{\rightleftharpoons}}\ \mathrm{I}$
5c	$\mathrm{SmX\ or\ Cr}\ \underset{132.3(4.4)}{\overset{139.1(4.9)}{\rightleftharpoons}}\ \mathrm{SmC}\ \underset{158.4\ (1.8)}{\overset{163.9\ (3.4)}{\rightleftharpoons}}\ \mathrm{N}\ \underset{237.4\ (2.1)}{\overset{241.9\ (2.1)}{\rightleftharpoons}}\ \mathrm{I}$
5d	$\mathrm{SmX\ or\ Cr}\ \underset{133.8\ (4.1)}{\overset{137.6(3.0)}{\rightleftharpoons}}\ \mathrm{SmC}\ \underset{166.2\ (1.2)}{\overset{172.1\ (1.6)}{\rightleftharpoons}}\ \mathrm{N}\ \underset{225.5\ (2.2)}{\overset{229.6\ (2.5)}{\rightleftharpoons}}\ \mathrm{I}$
6	$\mathrm{SmX\ or\ Cr}\ \underset{30.1^{a}}{\overset{52.3\ (134.9)}{\rightleftharpoons}}\ \mathrm{N}\ \underset{67.0\ (2.3)}{\overset{68.0\ (1.5)}{\rightleftharpoons}}\ \mathrm{I}$
7	$\mathrm{SmX\ or\ Cr}\ \underset{\mathrm{r.t.}}{\overset{\mathrm{r.t.}^{a}}{\rightleftharpoons}}\ \mathrm{SmC}\ \underset{91.7\ (1.0)}{\overset{91.7\ (1.1)}{\rightleftharpoons}}\ \mathrm{N}\ \underset{133.8\ (0.01)}{\overset{141.2\ (0.05)}{\rightleftharpoons}}\ \mathrm{I}$

[a]The phase temperature was determined by POM; r.t.= room temperature.

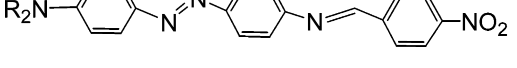
To investigate the miscibility of azo dyes **5a–d** in a host liquid crystal, compound **6**, prepared according to the literature [7], was mixed with a representative sample **5d** (9/1,w/w) to give mixture **7**. The resulting mesogenic behaviour is shown in table 2. Compound **6** and mixture **7** show nematic and SmC phases on heating. The clearing temperature for the nematic phase of mixture **7** is about 80° lower than that of azo dye **5d**, and its SmC phase is observed even at room temperature. No phase separation is observed for mixture **7** under POM, indicating that the miscibility of azo dye **5d** with compound **6** is reasonably good, even though azo dye **5d** contains a large and rigid conjugation moiety.



6

3.2. *Study of order parameter and photostability*

To verify the applicability of the prepared azo dyes to the LCD industry, the order parameter of azo dye **5b** was also measured according to the literature process and calculated to be 0.74 [6]. Usually, the optical order parameter (*S*), defined as in equation (1), should be in the range of 0.7–0.8 or higher for application. The order parameter of mono-azo dyes is in the range of 0.45–0.60 and that of bis-azo dyes 0.60–0.72 [6 *b*]. Attachment of a strong electron-withdrawing group, such as NO_2, at the end of the phenyl moiety increases the order parameter of the azo dye, but decreases its photostability. From the literature [6 *c*], the order parameter of compound **8**, containing an analogous conjugation moiety to the azo dye liquid crystal **5b**, is about 0.61 and is much less than that of azo dye **5b**. Apparently, the introduction of suitable alkyl chains into the long conjugation moiety of liquid crystalline molecules increases the order parameter of azo dye compounds significantly (about 10%). This should result from the self-assembly properties of liquid crystals. Additionally, this approach also not only improves the order parameter but also improves photostability when compared with compound **II** (table 1). According to Jones and Reeve's study [8], the instability of azo dyes under UV radiation arises from the transfer of hydrogen from the aryl group to the



8 R = C_4H_9

N=N moiety. Thus, the more acidic the hydrogens of aryl groups, the worse the photostability of azo dyes. The poor photostability of azo dye **II** may thus arise both from the strong electronegativity of the CHO group, resulting in the more acidic aryl Hatoms, and from oxidation of H on the CHO moiety. Conversion of CHO to CH=N reduces the electron-withdrawing on the aromatics and hence reduces the acidity of aryl Hatoms. The photostability of azo dye **5a–d** is thus increased.

4. Conclusions

We have successfully synthesized and characterized a series of yellow azo dye liquid crystals. Surprisingly, these azo dyes not only possess nematic and SmC phases during thermal processing, but also show good solubility in a host liquid crystal, good photostability and good optical order parameter. It is regarded that the introduction of suitable linear alkyl chains into a long-conjugation moiety not only increases the solubility of azo dyes in the hosts but also improves the order parameter because of the self-assembly property of liquid crystals. Additionally, the acidity of aryl Hatoms of azo dyes **5a–d** was reduced when compared with those of compound **II**, and thus their photostability is increased.

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