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Short communication

Synthesis of dis-azo black dyes for electrowetting displays

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ABSTRACT

This study was to design and synthesize a series of dis-azo dyes derived from p-n-alkyl aniline by introducing different alkyl group resulted in having high solubility in linear alkanes solvents, even absorption intensity of visible wavelengths. Results indicated that elementary properties of black oil ink were (1) non-polar; (2) low viscosity (<3.0 cps); (3) specified surface tension (<30 mN/m); (4) intensity of visible absorption uniformly covering 400–800 nm; (5) FoM equal to 100–1000; (6) hue close to standard black ($L=0$, $a=0$, $b=0$). We can conclude that dis-azo black dyes fulfills elementary conditions of colored oil ink for electrowetting display.

1. Introduction

Currently, the development of flexible displays is still in research and development stage. There are a lot of developed technologies which mainly contain technologies of liquid crystal display (LCD), electrophoretic display (EPD), organic light-emitting diode (OLED), and so on. Electronic paper based on the electrophoretic motion of particles inside small capsules has been demonstrated and commercialized. The electrophoretic technology with high reflectivity, high contrast, and bistable states predominantly led and developed by E-ink and SiPix .The response speed of such a system is rather slow because the velocity of the particles is limited [1,2]. Much research in recent years have demonstrated in electrowetting technology that is an attractive technology for the rapid manipulation of liquids on a micrometer scale [3–6]. They show that electrowetting technology could use to form a reflective display whose response speed is significantly faster than electrophoretic display, so that video content can be displayed.

A low cost, full color display can be fabricated with electrowetting using an RGB color filter. Liquavista proposes a possible display mode of color electrowetting display which belong to single-layer structure which is manufactured by using black oil ink and color filters in coordination. In this case, a black oil ink is required as an absorbing switch [7]. The colored oil should have specified surface tension and good compatibility with insulator [8]. Black oil ink consists mainly of black dye and non-polar solvent. Commercial black dyes are obtained by mixing red, yellow, and blue dyes or orange, navy, and etc., whose disadvantages are poor color reproducibility. Conventional dis-azo black dyes possess poor solubility in non-polar solvents and not enough broad and even absorption intensity of visible wavelengths at 400–800 nm. Few commercial dyes are satisfied for the requirements such as neutral hue, high oil solubility, high molar absorption coefficient and low viscosity. Besides, when increasing the concentration of dye in oil ink, the oil–water interfacial tension decreases [9] and the viscosity of oil ink increases steeply. This effect may influence either the amplitude of the driving voltage or the speed of oil moving when the voltage is switched on. Therefore, these results could not satisfy the application for light and thin electrowetting displays.

Previous researches have focused only on designing of electrowetting devices [10]. Fewer studies have addressed on black oil ink. Obtaining an ideal black shade is not easy. There are many shade of black ranging from reddish blacks to neutral blacks to greenish blacks. The purpose of this study was to synthesize a series of dis-azo dyes derived from p-n-alkyl aniline by introducing different alkyl group resulted in having high solubility in linear alkanes solvents and even absorption intensity of visible wavelengths. The black oil inks had advantages of neutral hue and color reproducibility and specified surface tension, and low viscosity. The results indicated these dis-azo black dyes were applicable significantly to electrowetting displays.

2. Experiment

2.1. Chemicals and reagents

1-Naphthylamine, 1,8-diamino naphthalene, and p-n-decylaniline were obtained from Aldrich (Buchs, Switzerland), sulfamic acid (99.5%) from Tokyo Chemical, hydrochloride (37%),

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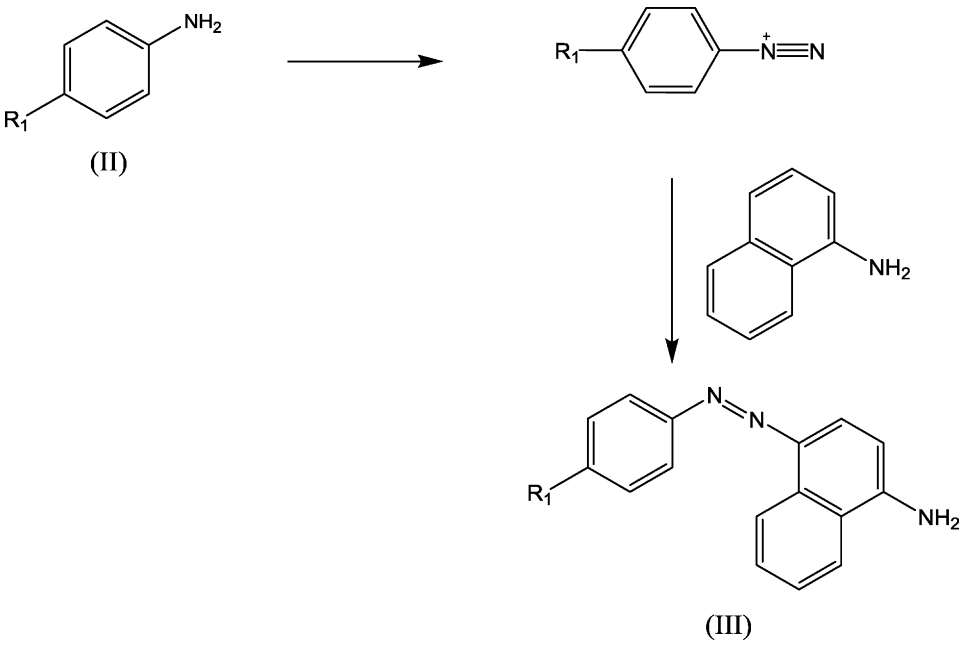


Scheme I.

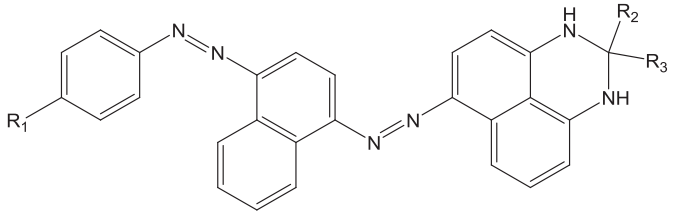
sodium acetate, glacial acetic acid and sodium nitrite (99%) from First Chemical. All the other reagents were of analytical grade from Merck.

2.2. Equipments

UV-Vis Spectroscopy (U-3010, Hitachi), Nuclear magnetic resonance (NMR, Bruker AV500), Fourier transform spectroscopy (FTIR-460 Plus) and LC/MS (Waters UPLC/ZQ-4000) system. The properties of oil were measured with Sivne-wave Vibro Viscomerter (SV-10) and SITA pro line f10 tensiometer.

3. The preparation of dis-azo black dyes and oil inks

The dis-azo compound provided by the study was represented by the following formula (I):



(I)

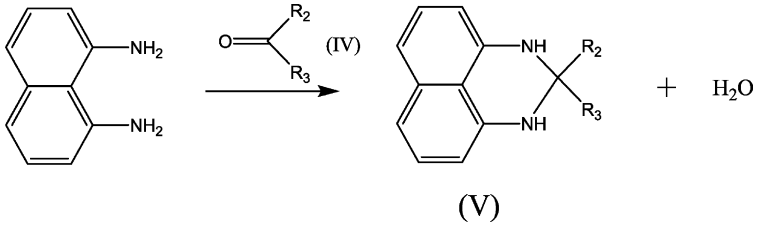
wherein R_1 was substituted C4–C12 alkyl, and R_2 and R_3 were independently substituted or unsubstituted C2–C4 alkyl.

As shown in those schemes, the dis-azo compound represented by the formula (I) was synthesized by following process. Firstly, the compound of the formula (II) was diazotized, and then reacted with α-naphthylamine to obtain a monoazo compound of formula (III), as shown in Scheme I. Subsequently, 1,8-diaminonaphthalene was reacted with the reagent of formula (IV) to obtain a intermediate of formula (V), as shown in Scheme II. Finally, the monoazo compound of formula (III) was diazotized, and then reacted with the intermediate of formula (V) to give the dis-azo compound of the formula (I) of the study, as shown in Scheme III.

3.1. Synthesis of mono-azo compound

To p-n-decylaniline (2.9 g) dissolved in IPA (6 mL). The solution was stirred at room temperature till dissolution completely. After that 5 N HCl (20 mL) was introduced; cooled to below 5 °C by ice container, and 40% $NaNO_2$ (70 mL) was added and stirred for 2 h. When the color of solution became yellow, sulfamic acid (2.0 g) was added. The resulting solution was a diazo component-1.

To α-naphthylamine (1.4 g) dissolved in H_2O (50 mL) and 5 N HCl (3 mL). The mixture was heated to 60 °C and stirred for 30 min. After α-naphthylamine was dissolved completely, the solution was cooled to 40 °C as a coupling component-1. One-third of the coupling component-1 was quickly added to the diazo component-1 prepared above. The reaction temperature was controlled to below 5 °C, then sodium acetate solution (10 mL) was added. After the reaction solution was stirred for 10 min, the remaining coupling component-1 (i.e. two-thirds of the coupling component-1) was slowly dropped therein. The reaction end was confirmed by TLC,



(V)

Scheme II.