



Synthesis of N-, S-, O-substituted quinone dyes and their dyeability on polyester fibers



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ARTICLE INFO

Article history:

Available online 4 May 2016

Keywords:

1,4-Naphthoquinone
Polyester fibers
Dyeability
Rubbing fastness
Wash fastness

ABSTRACT

The aim of this study was synthesis of substituted naphthoquinone derivatives and testing of their dyeability on polyester fibers. There are few studies concerned with dyeing properties of heteroatom-substituted-1,4-naphthoquinone compounds in the literature. A series of N-, S-, O-substituted-1,4-naphthoquinone compounds were synthesized via vinylic substitution. Their structures were determined by infrared spectrometry, ^1H and ^{13}C nuclear magnetic resonance spectroscopy, mass spectrometry and elemental analysis. The ultraviolet-visible absorption spectra of the dyes were examined in different solvents. The dyes were applied to polyester fibers and dyeing properties were investigated. The rubbing fastness, wash fastness and optical properties of the synthesized compounds also were measured.

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1. Introduction

A large number of naphthoquinone dyes occur naturally as plant constituents, and in the past many of these have found use as colorants. In contrast to the naphthoquinones, few synthetic benzoquinone dyes are suitable for application as vat dyes. From about 1930 onwards, developments in the field of naphthoquinone dyes concentrated on the use of naphthazarin and naphthoxidine as intermediates for the preparation of violet, blue and green acid and disperse dyes [1]. Recently, there has been interest in the synthesis and color and constitution properties of simple colored naphthoquinones, stimulated by the fact that such dyes have similar tinctorial properties to the anthraquinones but a smaller molecular size. The naphthoquinones provide a useful alternative to the anthraquinones for certain specialized applications [2,3]. There are few studies interested with dyeing properties of heteroatom-substituted-1,4-naphthoquinone compounds in the literature. The color of the naphthoquinone compounds can be produced in the 1,4-naphthoquinone chromogen by introduction amino and hydroxyl groups into the quinoid ring (positions 2 and 3), into the benzenoid ring (positions 5 and 8), or into both rings [4–7]. N-substituted-1,4-naphthoquinone compounds are generally yellow to red, depending on the nature of the substituents attached to the amino group and to the 3-position of the ring. Fluoro- and

trifluoro-methylphenylamino substituted naphthoquinones have been described for coloring wool and polyester [8]. Synthetic fibers are prepared from man-made polymers by extrusion through spinnerets, either from the molten state or from a solution in an organic solvent. They differ from natural fibers especially in the difficulty of dyeing. However, polyester can be dyed by disperse dyes, these are non-ionic dyes with finite, but very limited water solubility. Heteroatom substituted naphthoquinone dyes obtained their name from their application conditions, as they are applied as a very fine dispersion or suspension in water.

Four methods have been used for dyeing polyester fabrics: (a) conventional aqueous dyeing (batch dyeing); (b) aqueous dyeing with a carrier (batch dyeing); (c) high-temperature (above 100 °C usually 130 °C) aqueous dyeing (batch dyeing); (d) “Thermosol” dyeing (continuous dyeing). The high-temperature dyeing method was used for dyeing polyester fabrics in this study.

The present paper is concerned with the synthesis and investigation of dyes based on 2- and/or 2,3-substituted-1,4-naphthoquinones. In addition to synthesizing these proposed candidates, their spectroscopic properties and their application in the dyeing of polyester fabrics were studied.

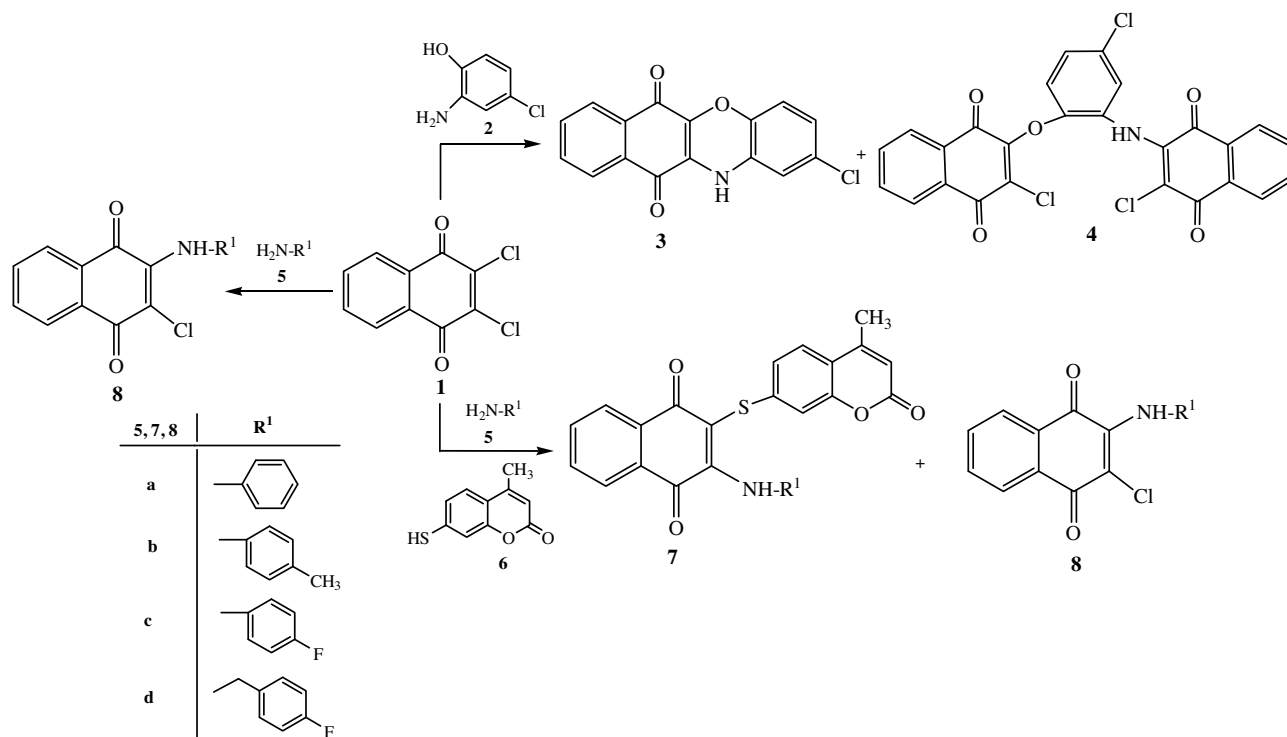
2. Experiment

2.1. General

All chemicals and solvents were obtained commercially and used without purification. Products were isolated by column chro-

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Scheme 1. The synthesis of N, S, O-Substituted-1,4-naphthoquinones.

matography on SiO₂ (Fluka Kieselgel 60, particle size 63–200 μm). TLC plates: Silica 60F₂₅₄ (Merck, Darmstadt), detection with ultraviolet light (254 nm). Compounds **3**, **8a–c** were synthesized from 2,3-dichloro-1,4-naphthoquinone **1** and aromatic amines according to the general procedure and confirmed by melting points, elemental analyses, FT-IR spectroscopy by comparison with published data [9,10]. Melting points were measured on a Büchi B-540 melting point apparatus and are uncorrected. Elemental analyses were performed with a Thermo Finnigan Flash EA 1112 elemental analyzer. Infrared (FT-IR) spectra were recorded in KBr pellets in Nujol mulls on a Perkin Elmer Precisely Spectrum One FTIR spectrometer. NMR spectra were recorded on a VarianUNITY INOVA instrument operating at 500 MHz for ¹H and 125 MHz for ¹³C. Mass spectra were obtained on a Thermo Finnigan LCQ Advantage MAX LC/MS spectrometer according to ESI probe. UV spectra in chloroform and hexane solutions were recorded on Varian 50 bio UV-spectrophotometer.

2.2. Synthesis procedure of dyes

2.2.1. General method

Sodium carbonate (1.52 g) was dissolved (60 mL) in ethanol. 2,3-dichloro-1,4-naphthoquinone and nucleophile compounds were added slowly to this solution for synthesis of known **3**, **8a–c** and previously unknown **4**, **7d** and **8d** compounds. Without heating, the mixture was stirred for 24 h. The color of the solution quickly changed and the extent of the reaction was monitored by TLC. Chloroform (30 mL) was added to the reaction mixture. The organic layer was washed with water (4 × 30 mL), and dried with Na₂SO₄. After the solvent was evaporated the residue was purified by column chromatography on silica gel (Scheme 1).

2.2.2. 2,2'-(2-Aminyl-4-chloro-phenyl)-3,3'-dichloro-1,4-naphthoquinone (4)

Compound **4** was synthesized from the reaction of **1** (1 g, 4.4 mmol) with **2** (0.63 g, 4.4 mmol) according to general method. Red solid. Yield: 1.4 g (60%). R_f: 0.40 (CHCl₃). FT-IR (in KBr pellet, cm⁻¹): 3445 (N–H), 3089 (Ar–H), 2958, 2918, 2849 (C–H), 1629 (C=O), 1582, 1562 (C=C). ¹H NMR (499.74 MHz, CDCl₃): δ = 5.1 (s, 1H, NH), 7.20 (t, J = 5.84 Hz, 1H, H_{arom}), 7.70 (t, J = 6.83 Hz, 1H, H_{arom}), 8.25 (dd, J = 5.32 Hz, J = 5.85 Hz, 1H, H_{arom}), 8.6 ppm (dd, J = 5.32 Hz, J = 5.84 Hz, 1H, H_{arom}). ¹³C NMR (125.66 MHz, CDCl₃): δ = 117.61, 119.99, 120.46, 124.03, 125.66, 127.81, 128.99, 129.72, 129.87, 130.43, 131.32, 131.44, 132.28, 138.44 (CH_{arom}, C_{arom}), 138.90, 140.01 (=C–N), 145.74, 145.80 (=C–Cl), 153.81 (C–O–C), 176.34, 176.44 ppm (C=O). MS[+ESI]: m/z 524 [M]⁺. Anal. Calcd. for C₂₆H₁₂O₅N₁Cl₃ (M, 524.74): C, 59.51; H, 2.30; N, 2.66. Found: C, 59.52; H, 2.31; S, 2.65.

2.2.3. 2-(7-Sulphanyl-4-methyl-coumarinyl)-3-(4-florobenzylaminyl)-1,4-naphthoquinone (7d)

Compound **7d** was synthesized from the reaction of **1** (1.0 g, 4.4 mmol) with **5d** (0.48 g, 4.4 mmol) according to general method. Red solid. M.p.: 195–197 °C. Yield: 0.90 g (43%). R_f: 0.55 (CH₂Cl₂). FT-IR (in KBr pellet, cm⁻¹): 3290 (N–H), 3071 (Ar–H), 2918, 2849 (C–H), 1733, 1720, 1667 (C=O), 1600, 1551 (C=C). ¹H NMR (499.74 MHz, CDCl₃): δ = 2.3 (s, 3H, CH₃), 4.9 (s, 2H, CH₂), 6.1 (s, 1H, NH), 6.7 (s, 1H, CH), 6.8 (t, J = 5.60 Hz, 2H, H_{arom}), 7.2 (dd, J = 7.84 Hz, J = 7.32 Hz, 2H, H_{arom}), 7.1 (t, J = 7.84 Hz, 1H, H_{arom}), 7.3 (dd, J = 6.34 Hz, J = 5.83 Hz, 2H, H_{arom}), 7.6 (t, J = 7.84 Hz, 1H, H_{arom}), 7.7 (t, J = 5.60 Hz, 2H, H_{arom}), 8.00 (t, J = 6.84 Hz, 1H, H_{arom}). ¹³C NMR (125.66 MHz, CDCl₃): δ = 17.56 (CH₃), 47.68 (NH–CH₂), 116.27, 120.63, 121.07, 123.83, 124.25, 126.09, 126.39, 128.27, 128.34, 129.13, 131.76, 132.49 (CH_{arom}, C_{arom}), 114.80 (CH), 114.97

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