Available online at www.sciencedirect.com
ScienceDirect
Dyes and Pigments 75 (2007) 550–555
DYES and PIGMENTS
www.elsevier.com/locate/dyepig

Dyeing of wool with natural anthraquinone dyes from *Fusarium oxysporum*

F.A. Nagia [a], R.S.R. EL-Mohamedy [b,*]

[a] *Dyeing and Printing Department, National Research Center, Dokki, Cairo, Egypt*
[b] *Plant Pathology Department, National Research Center, Dokki, Cairo, Egypt*

Received 14 December 2005; received in revised form 12 March 2006; accepted 1 July 2006
Available online 18 September 2006

Abstract

Two anthraquinone compounds are described which were produced by liquid cultures of *Fusarium oxysporum* (isolate no. 4), isolated from the roots of citrus trees affected with root rot disease. These anthraquinone compounds are 2-acetyl-3,8-dihydroxy-6-methoxy anthraquinone or 3-acetyl-2,8-dihydroxy-6-methoxy anthraquinone. Dyeing of wool fabrics with these new anthraquinone compounds as natural dyes has been studied. The values of dyeing rate constant, half-time of dyeing and standard affinity have been calculated and discussed. The effect of dye bath pH, salt concentration, dyeing time and temperature were studied. Colour strength values and the dye uptake were high. The results of fastness properties of the dyed fabric were good.

Keywords: Natural dyes; Anthraquinone; Dyeing; Wool; Kinetic; Fungi; *Fusarium oxysporum*

1. Introduction

Recently a revival interest in the use of natural dyes in textile coloration has been growing. This is a result of the stringent environmental standards imposed by many countries in response to the toxic and allergic reactions associated with synthetic dyes [1]. Natural dyes are friender to the environment than synthetic dyes and can exhibit better biodegradability and generally have a higher compatibility with the environment [2–4].

The production and evaluation of microbial pigments as textile colorants is currently being investigated [5]. Fungi are more ecological interesting source of pigments, since some fungal species are rich in stable colorants such as anthraquinone [6–9]. Anthraquinone derivatives were previously isolated from the fungus *Dermocybe sanguinea* [10]. A number of anthraquinone derivatives, Fig. 1, have been identified from various species of fungi and Lichens [11]. These metabolites are of interest because many of them possess significant antibiotic activity, primarily against Gram-positive bacteria and *Pseudomonas aeruginosa*. Anthraquinones are also reported to have antiprotozoal and cytotoxic activities [12,13].

The purpose of this research is to evaluate the anthraquinone dyes isolated from the fungus *Fusarium oxysporum* in dyeing wool and their effectiveness on different factors affecting dyeability and fastness properties.

2. Experimental

2.1. Materials

Anthraquinone compounds extracted from the culture of *F. oxysporum* are used as natural dyes.

Scoured and bleached wool fabric with the following characteristics was purchased from Misr for Spinning and Weaving Company, Mahalla El-Kobra, Egypt; weight 205 g m^{-2}, 72 ends per inch, 64 picks per inch. Before using, the fabric

* Corresponding author.
E-mail address: riadelmohamedy@yahoo.com (R.S.R. EL-Mohamedy).

0143-7208/$ - see front matter © 2006 Elsevier Ltd. All rights reserved.
doi:10.1016/j.dyepig.2006.07.002

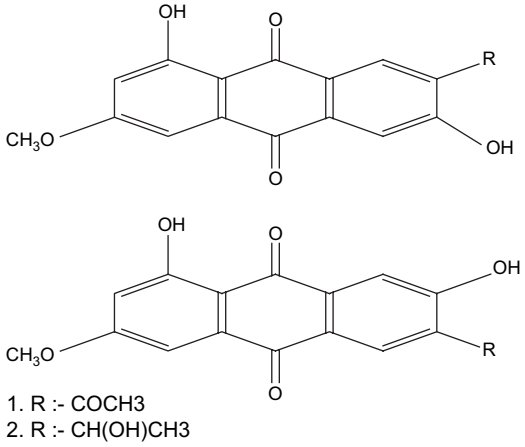

Fig. 1. Structure of *F. oxysporum* anthraquinone.

was treated with a solution containing 5 g L^{-1} nonionic detergent (Hostapal CV, Clariant), at 50 °C for 30 min.

Then, the fabric was thoroughly washed with water and air dried at room temperature.

2.2. Methods

2.2.1. Fungal isolates

Five isolates of *F. oxysporum* were isolated from naturally infected feed roots of citrus trees affected by wilt and root rot disease [14,15]. *F. oxysporum* (isolate no. 4) with highly pinkish purple pigment on PDA medium was used in this study to evaluate their ability in producing anthraquinone dyes.

2.2.2. Cultures grown

Cultures of *F. oxysporum* (isolate no. 4) were grown on a defined mineral salts–glucose medium [16]. This medium contains the following, in ppm; $NaNO_3$, 848; KCl, 300; $MgSO_4 \cdot 7H_2O$, 165; NaH_2PO_4, 100; $CaCl_2 \cdot 2H_2O$, 40; H_3BO_4, 5.7; $FeSO_4 \cdot 7H_2O$, 5.0; $ZnSO_4 \cdot 7H_2O$, 4.4; $MnSO_4 \cdot H_2O$, 3.1; $Na_2MoO_4 \cdot 2H_2O$, 25; $CuSO_4 \cdot 5H_2O$, 0.4; and glucose, 20,000. Mineral salts and glucose solutions were autoclaved separately and combined after cooling in culture flasks in a sterile laminar-flow hood. All flasks were inoculated by a mycelial disk (5 mm diam.) from PDA culture of *F. oxysporum* which were grown at 27 °C in the dark as stationary cultures for 4–6 weeks.

2.2.3. Extraction

Cultures were filtered after 4–6 weeks, and the filtrate was adjusted to pH 3 with HCl. The acidified filtrate was then passed through a column of Amberlite XAD-7 for absorption of the pigments.

Compounds were removed from the column by elution with acetone, the acetone being removed using a Buchi rotary evaporator and the aqueous phase extractions were combined and reduced using the rotary evaporator for chromatographic analysis.

2.2.4. Chromatography

Column chromatography was performed on a 5 × 20 cm column packed with Kieselgel 60 reinst (70–230 mesh ASTM) that had been deactivated with acetic acid and H_2O, washed with acetone and then chloroform. The sample was loaded in chloroform, then eluted with chloroform, followed by consecutive elutions with chloroform containing acetone at 3, 8 and 20%; fractions eluted from the column were purified by TLC on 250 μm silica gel GF plates, using (A) benzene–nitro methane–acetic acid (75:25:2) or (B) chloroform–methanol–acetic acid (195:5:1). The R_f values (15 cm) for compound **1** in solvent system A and B were 0.65 and 0.55; for compound **2**, 0.30 and 0.08.

2.2.5. Toxicity assay

Filtrate extract (10 mL) and 100 mL of different concentrations of eluated anthraquinone pigment were prepared for testing by diluting with 0.2 mL of ethanol, applied to sterile 9 cm diam. Whatman No. 1 filter paper disks in Petri dishes, and drying a sterile laminar-flow hood. After all solvent was removed, 5 mL of sterile water was added. Then 10 surface disinfected seeds of sour orange, Rangpur lime and Volkamer lime were placed on the wetted paper. Ten Petri dishes were used as replicates for each treatment. After 21 days of incubation at 27 ± 1 °C, total root growth (germination) was measured and compared to control (untreated), and expressed as root growth inhibition %.

2.2.6. NMR and mass spectra

^{1}H NMR spectra at 270 MHz with tetramethyl silane as an internal standard in deuterated chloroform and mass spectra (MS) were obtained through Central Lab of Service in National Research Center.

2.2.7. Dyeing procedure

Dye bath containing different amounts of sodium chloride (0–20 g L^{-1}) and the calculated amount of the dye with liquor ratio 40:1 was heated at different durations (12–120 min) and at different temperatures (30–100 °C).

The dyed samples were rinsed with cold water, washed in a bath of liquor ratio 40:1 using 3 g L^{-1} nonionic detergent (Hostapal CV, Clariant) at 50 °C for 30 min, then rinsed and finally dried at ambient temperature.

The pH values were recorded with Hanna pH meter and adjusted with dilute solutions of sodium carbonate.

2.2.8. Dyeing rate

The wool fabric samples were cut into pieces approximately 1 cm^2 and dyed at pH 2.5 in a beaker with 160 mL aqueous solution containing 1 g nonionic wetting agent (Hostapal CV, Clariant) and the calculated amount of the dye at liquor ratio 40:1 and at 100 °C with frequent shaking.

2.3. Colour strength

The reflectance of the soaped samples was measured on a Perkin–Elmer Lambda 3B UV/vis spectrophotometer.