

SYNTHESIS OF NON-BENZIDINIC HOMOLOGUE OF CONGO RED (DIRECT RED 28) AND EFFECT OF TEMPERATURE AND TIME ON ITS FASTNESS PROPERTIES

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Abstract: Benzidine substituted azo dye was prepared using 4,4'- Diaminobenzanilide and Sodium Naphthionate (1-amino naphthalene-4-sulphonic acid sodium salt). Dye was applied to different varieties of fabrics at different temperatures and time. It was found that there was marked effect on fastness properties of dye with increase in temperature and time period of dyeing. Results show that dyes have fair to good fastness properties for cotton, whereas poor fastness for acetate, acrylic, wool and very poor properties for polyester.

Keywords: 4,4'- diaminobenzanilide, fastness Properties

Introduction

The manufacture of Benzidine based dyes is prohibited due to carcinogenicity of the precursor. The problem of replacing benzidine has not been fully resolved although a number of approaches have been made, based on two concepts. The first concerns the use of other classes of dyes for dyeing cellulose fiber, and the second approach involves the use of other diamines in dye synthesis. The first solution does not usually ensure obtaining all possible hues from the benzidine based dyes. Also, it is a rather expensive way and often requires alternative dyeing technology. The second concept is of interest, since it could yield new harmless dyes having the same colours and application properties as the benzidinic dye. 4,4'-diaminocarbanilide [1], diaminostilbenedisulphonic acid [2], and diaminodiphenylamine sulphonic acid [3] have been investigated in this context, and more recently further suggestions for replacing benzidine with other diamines have been described [4-8]. The causes of carcinogenicity of benzidine have not been fully rationalized, but it has been noted that lake of carcinogenicity is likely to occur in di-

amines (and derived dyes) whose metabolic decomposition results in the formation of water soluble products [9,10].

In the present study, 4,4'-diaminobenzanilide has been used, which is not a benzidine congener, for the synthesis of direct dye having dyeing properties similar to their benzidine homologues. 4,4'-diaminobenzanilide has already been used as benzidine substitute in the synthesis of some direct dyes [11-13]. In this regard, 4,4'-diaminobenzanilide (DABA) and Sodium Naphthionate have been used to prepare direct red dye. The synthesized dyes have been used to dye different varieties of fabrics at different temperatures and times. The effect of time and temperature on the dyeing and fastness properties such as washing, light, rubbing and perspiration have also been investigated.

Materials and Methods

Synthesis of Direct Red Dye:

A red symmetrical bis-azo dye was prepared by the coupling reaction of bis-diazonium salt of

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4,4'-diaminobenzanilide with 1-naphthylamine-4-sulfonic acid, in a 1:2 molar ratio. The synthesis of the red dye consisted of two steps: (1) Bis-diazotisation of 4,4'-Diaminobenzanilide, and (2) Coupling of Bis-diazotised product with Sodium Naphthionate.

Bis-diazotisation Step

An amount of 5.0 g (0.021806 moles) DABA 99%, (supplied by Vardhman Chemicals, 203, Hari Krishna Complex, Pritam Nagar Ahmadabad, India), 50.0 ml of distilled water and 7.5 ml of 37% fuming HCl were mixed in a 1000 ml beaker (large sized beaker to avoid overflow due to foaming in the subsequent steps). The contents of the beaker were stirred vigorously and the temperature of the contents of the beaker was reduced to 0-5 °C with the help of ice around the beaker. Between 0-5 °C 3.01g (0.043187 moles) sodium nitrite 99% in 25ml water was added--70% at once and 30% at the rate of consumption. The rate of consumption was checked by using starch iodide paper. Stirring was continued for 2 hr keeping the temperature between 0-5 °C with crushed ice until starch iodide-congo red paper positive. Excess of HNO₂ was removed using 5 ml of 10% sulphamic acid solution.

Coupling Step:

An amount of 14.44g (0.0436 molar) Sodium Naphthionate 74% (supplied by Vivil export pvt. Ltd. Mumbai India) was dissolved in 60.0 ml water. This solution was added drop-wise

over 1/2 hr period to the bis-diazotised solution of DABA, and then the reaction mixture was stirred for further three hr at 0-8 °C at a pH of 1.8-2.2. The pH of this solution was increased with soda ash (20% solution) to a range of 6.5-6.8 over 2 hours, keeping the temperature of the contents of the beaker "between" 3-8 °C. In case of foaming, a few drops of antifoam (Ethyl Hexanol) were added. The product was stirred vigorously for further 5 hours. The dye was precipitated by the addition of NaCl (5% w/v). Stirring was continued for a further 1 hr followed by filtering of the dye through a Buckner funnel using a vacuum pump. The paste was dried in an oven at 60-65 °C. The dye obtained was ground and stored in an air tight container. The yield of the dye obtained was 95%. The structure of the dye is shown in Figure 1.

Dyeing of Fabrics

Depth of shade	1%
Dye concentration	1%
Liquor ratio	1:20

Dyeing was performed in IR dyeing machine at different temperatures and time intervals. The wavelength of maximum absorption was recorded on a Unicam Helios spectrophotometer.

Temperature range: The Fabrics were dyed with 1.0% dye (calculated by weight of fabrics) by keeping the dyeing machine at temperatures that ranged from 50 to 95 °C at a liquor ratio of

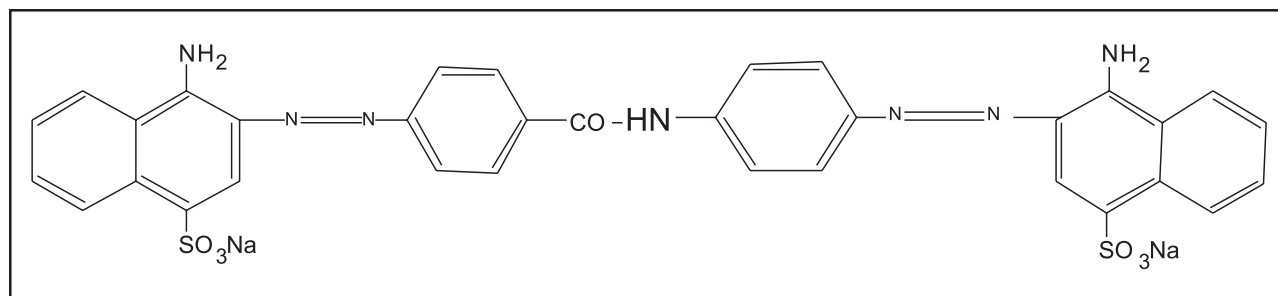


Fig. 1. Structure of Direct Red dye.

Fastness to Artificial Light (using weather-o-meter)

The colour fastness to artificial light for the fabric obtained after treatment at different temperature was determined using ISO-BO2 standard procedure. The test specimen of the dyed fabrics was exposed to artificial light (using weather-o-meter) under standard conditions, using a sample of blue wool as reference. The colour fastness was then assessed by comparing the change in colour with that of reference blue wool. The results thus obtained are shown in Table 1.

Fastness to Perspiration

This test was carried out using ISO-EO4 standard procedure. In this test, a specimen measuring 40 mm x 100 mm was cut and placed between two single fiber adjacent fabric also of the same size and sewn along one of the shorter sides. The composite specimen was placed smoothly in a dish containing freshly prepared acidic solution of L-histidine monohydrochloride monohydrate. The specimen was thoroughly wetted out in the acidic solution at a liquor ratio of 50:1, and was allowed to remain in the solution at room temperature for 30 minutes. The solution was poured off and the excess liquor was wiped off the specimen. The composite was then placed between two acrylic resin plates, under a pressure of 12.5 K Pa. The test device containing the composite specimen was placed in an oven for 4 hours at 37 °C. The fabric was opened out, dried and the change in colour was assessed with the grey scale. The same procedure was repeated with another piece of fabric using freshly prepared alkaline solution of L-histidine monohydrochloride monohydrate. The change in colour of each specimen and the staining of the adjacent fabric was assessed with grey scale. The results are summarized in Table 1.

Results and Discussion

When both the dyeing time and temperature of dyeing were increased, the absorbance of the remaining aliquot in the dyeing bath decreased accordingly. The results obtained to see the effect of temperature and time on dye exhaustion are shown in Figs. 3 & 4, respectively. There was gradual decrease in the absorbance of the remaining aliquot in both cases. This confirms that there is gradual uptake of dye on fabrics with increase of both temperature and time.

Similarly, the effect of temperature on the fastness properties was obtained on different fibers. The results show that with the increase of temperature the fastness properties moved from poor to fair and good side on grey scale. The results obtained are shown in Table 1.

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