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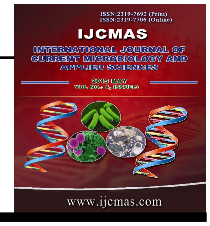

Original Research Article

Dyeing of polyester with disperse dyes: Part 1. Antimicrobial activity and dyeing performance of some disperse dyes

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A B S T R A C T

Keywords

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3-Dimethylamino-1-*p*-arylpropenones 3a,b coupled with phenyl diazonium salt to give the corresponding 3-oxo-2-(phenylhydrazono)-3-*p*-arylpropionaldehydes disperse dyes. Fastness properties of the dyes were examined against light, rubbing, perspiration and washing fastness. The biological activities of the synthesized dyes against Gram positive bacteria, Gram negative bacteria, yeast and fungi were also evaluated.

Introduction

Azo disperse dyes are a versatile class of coloured organic dyes and receive a considerable attention, as a consequence of their exciting biological properties and their applications in various fields, e.g., textiles, leathers, papers, additives, and cosmetics (Abdou *et al.*, 2013; Zollinger, 2003; Klaus, 2003). Considerable studies have been devoted to azo dyes derived from 3-dimethylamino-1-*p*-arylpropenones (El-Apasery, *et al.*, 2011; Hajós, *et al.*, 2008; Al-Saleh, *et al.*, 2004). The azo disperse dyes based on enaminones result in brighter and deeper shades than their benzene analogues. Al-Etaibi *et al.* (Al-Etaibi *et al.*, 2012; and 2011) reported the syntheses of different azo disperse dyes based on enaminones for polyester fabrics. Recently, other studies reported the application of synthesized azo dyes for dyeing and printing polyester fabrics (El-Molla *et al.*, 2013; Elattar, *et al.*, 2013). Thus, we have initiated a program of applying the synthesized dyes derived from enaminones to polyester as disperse dyes to study their colour measurement, fastness properties and antimicrobial activities.

Experimental

General procedure for the synthesis of azo disperse dyes (5a,b)

A cold solution of the diazonium salt (10 mmol), prepared by adding a cold solution of sodium nitrite (0.7 g) in water (10 mL) to a solution of aniline (10 mmol) in conc. hydrochloric acid (4 mL), was added to a cold solution of 3-dimethylamino-1-*p*-arylpropenones **3a** or **3b** (10 mmol) in ethanol (15 mL) containing sodium hydroxide (1.2 g). The resulting mixture was stirred at room temperature for 30 min. The precipitate that formed was collected by using filtration and crystallized from ethanol. Dyes **5a,b** was confirmed by the reported data (Al-Shiekh, *et al.*, 2008; Hajós, *et al.*, 2008; Al-Awadi, *et al.*, 2001)..

High temperature dyeing method (HT)

Materials

Scoured and bleached 100% polyester fabric (149 g\m^2) was supplied by El-Mahalla El-Kobra Company. The fabric was treated before dyeing with a solution containing non-ionic detergent 5 g/L (Hostapal, Clariant) and sodium carbonate (2 g/L) in a ratio of 50:1 at 60 °C for 30 min, thoroughly washed with water, and air dried at room temperature.

Dyeing

A dispersion of the dye was produced by dissolving the appropriate amount of dye (2% shade) in 1 mL dimethylformamide and then added drop wise with stirring to the dye bath (Liquor ration 50:1) containing sodium lignin sulfonate as dispersing agent. The ratio of dispersing agent to dyestuff is 1:1. The pH of the dye bath was adjusted to 4.85 using aqueous acetic acid and the wetted-out polyester fabrics were added. Dyeing was performed by raising the dye bath temperature to 130 °C for 60 min under pressure in an infra red dyeing machine. After dyeing, the fabrics were thoroughly washed and subjected to surface reduction clearing ((2 g NaOH + 2 g sodium hydrosulphite)/L, and soaped with 2% nonionic detergent to improve washing fastness). The samples were heated in this solution for 45 min at 80 °C. Rinse well in cold water and neutralize with 1g/L acetic acid for 5 min at 40 °C, the dyed samples were removed, rinsed in tap water and allowed to dry in the open air.

Colour measurements

The colorimetric parameters of the dyed polyester fabric was determined on a reflectance spectrophotometer. The colour yields of the dyed samples were determined by using the light reflectance technique performed on UV-Vis spectrophotometer. The colour strengths, expressed as K/S values, were determined by applying the Kubelka-Mink equation (Al-Etaibi *et al.*, 2014a).

$$K/S = [(1 - R)^2 / 2R] - [(1 - R_o)^2 / 2R_o]$$

where R = decimal fraction of the reflectance of the dyed fabric; R_o = decimal fraction of the reflectance of the undyed fabric; K = absorption coefficient; S = scattering coefficient.

Fastness tests

Fastness to washing

After washing using 5 g/L of the non-ionic detergent (Hostapal, Clariant) and 2 g/L of sodium carbonate, the dyed fabrics were tested by using ISO standard methods (Chrysler *et al.*, 1990). A specimen of dyed polyester fabric was stitched between two pieces of undyed cotton and wool fabrics, all of equal length, and then washed at 50 °C for 30 min. The changes in color were assessed according to the gray scale (Al-Mousawi *et al.,* 2014).

Fastness to perspiration

The samples were prepared by stitching a piece of dyed polyester fabric between two pieces of cotton and wool fabrics and then immersed in the acid or alkaline solution for 30 min. The acid solution (pH=4.5) contains sodium chloride (10 g/L), sodium dihydrogen orthophosphate (1 g/L) and L-histidine monohydrochloride monohydrate (0.25 g/L). The alkaline solution (pH=8.7) contains sodium chloride (10 g/L), disodium orthophosphate (1 g/L) and L-histidine monohydrochloride monohydrate (0.25 g/L). The changes in color were assessed according to the gray scale.

Fastness to rubbing

The dyed polyester fabric placed on the base of a crock meter, so that it rested flat on the abrasive cloth with its long dimension in the direction of rubbing. A square of white testing cloth forced to slide on the tested fabric back and forth twenty times. For the wet rubbing test, the test square was wetted in distilled water. The rest of the procedure was the same as the dry test. The staining on the white testing cloth was assessed using gray scale.

Fastness to light

Light fastness was determined by exposing the dyed polyester on a Xenotest 150 Original Hanau, chamber temperature 25-30 °C, black panel temperature 60 °C, relative humidity 50-60%, and dark glass (UV) filter system) for 40 h. The changes in color were assessed according to the blue scale (Chrysler, 1990).

Antimicrobial Activities Test

The antimicrobial activities of disperse dyes were tested using Agar-well diffusion technique (Al-Etaibi *et al.,* 2014b), against six different microbial cultures. Pure cultures of *Bacillus cereus* and *Staphylococcus auerus* (Gram positive bacteria), *Escherichia coli* and *Pseudomonas aeruginosa* (Gram negative bacteria), *Candida albicans* (Yeast) and *Aspergillums Niger* (Fungi) were involved in the test. An aliquot of 0.1 mL of each bacterial strain was inoculated and spread on nutrient agar (NA) while 0.1 mL of the yeast and fungi were spread on potato dextrose agar (PDA). The inoculated plates were supplied with 100µl of each of the tested disperse dyes with a total final concentration of 100mg ml^{-1}. The disperse dyes were included in 6 mm wells produced by sterile cork borer. The NA plates were incubated at 37 °C for 24 hours while PDA plates were incubated at 25 °C for 24-48 h. The zones of inhibition around the wells were determined. Picture were taken for some of the plates after 24 and 120 h using digital camera to determine the nature of the dyes if they were cytolytic or cytostatic

Results and Discussion

Synthesis of some dyes based on 3-dimethylamino-1-*p*-arylpropenones moiety has been reported (Al-Shiekh, *et al.,* 2008; Hajós, *et al.,* 2008; Almazroa, *et al.,* 2004). Herein, in an attempt to evaluate their dyeing performance, thus enaminones 3a,b react with phenyl diazonium salt in acidic medium afforded the 3-oxo-2-(phenyl-hydrazono)-3-*p*-arylpropionaldehydes disperse dyes 5a,b (Scheme 1).

Disperse dyes 5a,b were applied to polyester fabrics at 2% (shade), using high temperature dyeing method (HT) at 130 °C. Greenish-yellow and yellowish-orange colour shades were obtained. The dyeing properties on the polyester fabrics were evaluated in terms of their fastness properties (e.g., fastness to rubbing, washing, perspiration and light). The colour of dyeing on polyester fabrics is expressed in terms of CIELAB colour space values (Table 1). The following CIELAB coordinates were measured: lightness or darkness ($L*$); brightness or dullness (chroma ($C*$)); hue angle (h) from 0 to 360 °; $a*$, whose value represents the degree of redness (positive) and greenness (negative); and $b*$, whose value represents the degree of yellowness (positive) and blueness (negative). The surface colour yield K/S was used to explain the amount of dye absorbed on the surface of the fibre. The K/S values listed in (Table 1) show that dyes 5a,b showed high affinity for the polyester fabrics and the K/S were all generally satisfactory. In general, the color hues of the disperse dye 5a on the polyester fabric shifted to the greenish directions; this was indicated by the negative value of $a* = -20.37$ (red–green axis).

The fastness ratings are recorded in (Table 2), shows that the disperse dyes displayed very good fastness levels to washing and excellent fastness levels to both rubbing and perspiration. The light fastness of the dyes 5a,b displayed fair fastness on polyester fabrics. It is of value to mention here that light fastness was obtained by the dye 5b containing a nitro group in the coupling component, the nitro group increases the polarity of the dyes (Al-Etaibi *et al.*, 2014a), which may link them more strongly to the fabric and it opens an extra way for the dissipation of energy after light absorption which decreases fading. Attempts are in hand to improve the light fastness properties of these dyes. It can be seen from Table 2 that the rating nature of substituents on the aromatic moieties of the dye molecules determined the wash fastness for dyed fabrics. Electron-withdrawing group enable stronger Van der Waals forces and hydrogen bonding with the dyed fabrics that increases their stability to washing.

Table.1 Shade and optical measurements of the azo disperse dyes on the polyester fabrics

Dye No	Color shade on polyester	Absorption (λ_{max} (nm))	$L*$	$a*$	$b*$	$C*$	$h*$	K/S
5a	Greenish-yellow	410	77.22	-20.37	53.94	57.66	110.69	17.59
5b	Yellowish-orange	405	67.58	9.29	61.34	62.04	81.39	16.69

Table.2 Fastness properties of azo disperse dyes on polyester fabrics *

Dye No	Rubbing fastness		Wash fastness			Perspiration fastness						Light fastness
						Alkaline			Acidic			
	Dry	Wet	Alt	SC	SW	Alt	SC	SW	Alt	SC	SW	
5a	5	5	4-5	4-5	3-4	5	5	5	5	5	5	3
5b	5	5	4-5	4-5	4	5	5	5	5	5	5	3–4

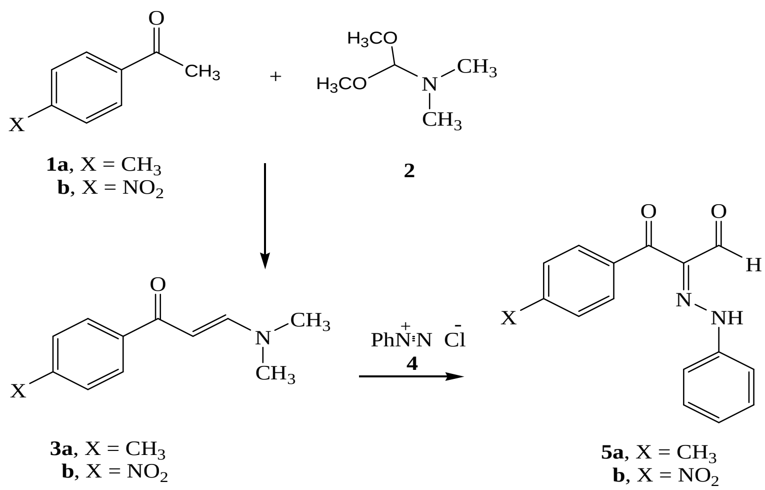
* Alt = alteration; SC = staining on cotton; SW = staining on wool

Table.3 Diameter of the zones of inhibition of the tested disperse dyes against Gram positive, Gram negative bacteria, yeast and fungi

Dye No	Inhibition zone diameter (Nearest mm)					
	G+Ve bacteria		***G-Ve bacteria***		***Yeast***	***Fungi***
	B. cereus	***S. aureus***	***E. coli***	***P. aeruginosa***	***C. albicans***	***A. Niger***
5a	11	11	9	8	NI	NI
5b	18	12	10	14	20	24

(NI) No Inhibition.
Well Diameter: 6 mm

Scheme.1



Antimicrobial activities

The inhibition zone diameter data for the 3-oxo-2-(phenylhydrazono)-3-*p*-arylpropionaldehydes disperse dyes, given in (Table 3), shows that all of the tested dyes showed strong positive antimicrobial activities against at least four of the tested microorganisms. Both of disperse dyes 5a,b showed cytolytic effect even after five dyes of incubation, there were no growth recorded in the inhibited zone for all of the tested microbes. Comparison between conventional and both of microwave, ultrasound dyeing, for these disperse dyes are under investigation.

In conclusion, 3-Dimethylamino-1-*p*-arylpropenones coupled with phenyl diazonium salt to give the corresponding 3-oxo-2-(phenylhydrazono)-3-*p*-aryl-propion aldehydes disperse dyes. The dyes produced in this manner were then applied to polyester fabrics by using high temperature dyeing method at 130 °C. The dyed fabrics displayed greenish-yellow and yellowish-orange on polyester fabric, have fair, very good and excellent fastness levels to light, washing, rubbing and perspiration, respectively. Finally, the antimicrobial activities of the synthesized disperse dyes against Gram positive bacteria, Gram negative bacteria, yeast and fungus were discussed.

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