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THE APPLICATION OF DISPERSE DYES DERIVED FROM 4-BROMOANILINE AND 3-CHLOROANILINE ON TO POLYESTER FABRIC

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ABSTRACT. A series of new disazo disperse dye compounds based on 4-amino-2-chlorophenylazo-4-bromobenzene intermediate were prepared by linking various arylamines and phenolic derivatives to this intermediate through diazo coupling reactions. The structures of the azo compounds were confirmed by FT-IR, ¹H NMR, ¹³C NMR and UV-visible spectral data. The results of the UV-vis absorption spectra of some of the dyes showed near-infrared absorptions around 573-800 nm. The results of the colour fastness to washing and sublimation gave an excellent value of grade 5. The light fastness values were found to be technically acceptable with the grey-scale grade of 5 to 6-7. Also, the rubbing fastness was observed to be grade 3/4 to 4.

KEY WORDS: Disazo dyes, Spectral absorption, Polyester, Fastness, Near-infrared absorption

INTRODUCTION

Organic compounds that absorb light in the near-infrared region of the electromagnetic spectrum represent a group of emerging compounds which show great potential for use in many areas of human endeavour [1-3]. Some of these include security-printing such as bank notes, credit cards, passports, invisible inks, and identity cards. Compounds of this type are all considered to contain a conjugated π -electron system. And for a chromophore to absorb at wavelengths in the near-infrared, the π -electrons must be delocalised especially along the conjugated chain [4-7]. Near-infrared absorbing organic molecules are compounds that interact with near infrared light and can also emit such near infrared light when under external stimulation like photoexcitation, chemical reactions and electric field [8-10].

Various classes of organic molecules that can be classified as near-infrared absorbers are metal dithiolene complexes, phthalocyanines, naphthacyanines and polymethines. Most of these compounds exhibit extreme stability to high temperatures, chemical stability, photostability and resistant to weather effect. Thus, some of them have been reported to be suitable for applications in the automobile and architectural sectors [4, 8, 11-13].

Recently, the synthesis, characterisation and application of a series of some near-infrared absorbers based on 4-bromoaniline and 3-aminophenol were discussed [13]. The findings obtained from the study, encouraged the authors' to undertake the present work as a continuation of an on-going research program, aiming to synthesize, characterize and apply another series of azo compounds based on 4-bromoaniline and 3-chloroaniline on to synthetic textile substrate. Organic compounds of this type can be used as chromophoric systems for many kinds of applications such as in the dyeing of polyesters, polyamides and acrylics. In addition, they could be used as organic photoconductors, in liquid crystal displays, photosensitizers and other non-textile applications [11, 14, 15].

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EXPERIMENTAL

Materials and instruments

4-Bromoaniline and 3-chloroaniline were purchased from Merck (Germany). All other chemicals and solvents were of analytical reagent grades and were used without further purification. A plain woven polyester fabric was used (density 136 x 88, weight 68-70 gm⁻²). The melting points of the dyestuffs were determined on a melting point instrument (Thermo Fisher Scientific, USA) and were reported uncorrected in °C. Fourier transform-infrared (FT-IR) spectra were recorded on a Shimadzu IR-8400S spectrometer. Ultraviolet-visible (UV-Vis) absorption spectra were recorded using a Shimadzu UV-2400 double-beam PC spectrophotometer. The ¹H NMR spectra and ¹³C NMR spectra were recorded using a Varian mercury 200BB series spectrometer, respectively, in dimethylsulphoxide (DMSO)-d₆, using tetramethylsilane (TMS) as the internal standard.

Synthesis of compound 3

Compound **1** (10.0 g, 85 mmol) was mixed with water (50 mL) and concentrated sulfuric acid (8 mL) and cooled to 0-5 °C. A cold aqueous solution of sodium nitrite (4.0 g, 58 mmol) in water was then added slowly over a period of 20-30 min, while maintaining the temperature at 0-5 °C with vigorous stirring. The reaction mixture was further stirred for 70 min, and excess nitrous acid produced was destroyed by the addition of urea (0.2 g). The clear diazonium salt solution was then added dropwise with stirring to a well-cooled (0-5 °C) and stirred solution of 3-chloroaniline (6 mL) in glacial acetic acid (7 mL). Stirring was continued for 2 h at room temperature and the precipitated product obtained was filtered, washed with water several times, dried and purified by column chromatography (using 100-200 mesh silica gel, ca 100 g) and a gradient eluent system with chloroform-methanol (95:5, v/v) to afford compound **3** with a yield of 85%.

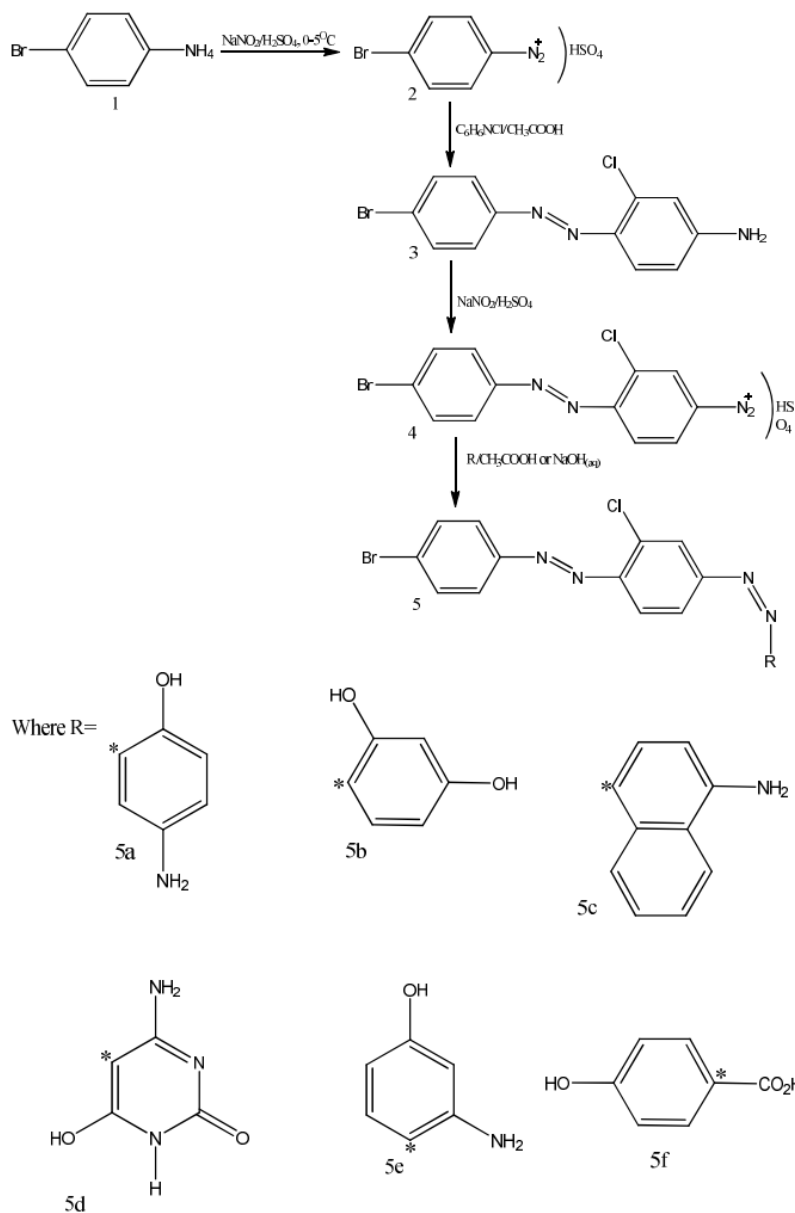
Synthesis of compounds 5a to 5f

A well-stirred mixture of compound **3** (2 g, 0.006 mol), concentrated sulfuric acid (5 mL), and water (30 mL) was thoroughly stirred and cooled in an ice-bath for 3 min. Sodium nitrite (2.3 g, 0.058 mol) solution in water (10 mL) was cooled to 0-5 °C and then added dropwise to the mixture with vigorous stirring for about 20 min. The cold diazonium salt solution obtained was then added dropwise to a well-stirred solution of 4-aminophenol (1.05 g, 0.10 mol) in sodium hydroxide (1.0 g) dissolved in water (20 mL) with vigorous stirring. The reaction mixture was further stirred for 2 h at room temperature and the precipitated product obtained was filtered, washed with water thrice, dried, and recrystallised from ethanol to give compound **5a**. Other compounds such as 1,3-dihydroxybenzene, 1-naphthylamine, 6-aminouracil, 3-aminophenol and 4-hydroxybenzoic acid were used in a coupling reaction with the diazonium salt solution of compound **3** to afford compounds **5b**, **5c**, **5d**, **5e** and **5f**, respectively.

Dye 5a. Yield 43%; mp 170 °C; ¹H NMR (200 MHz, DMSO-d₆) δ, ppm 2.72 (s, 2H, NH₂), 4.31 (s, 1H, OH), 6.64 (m, 2H, ArH), 6.92 (m, 1H, ArH), 7.23-7.70 (m, 3H, ArH), 7.85-8.20 (m, 2H, ArH), 8.51-8.75 (m, 2H, ArH), ¹³C NMR (50 MHz, DMSO-d₆) δc, ppm 140.2, 145.4, 144.7, 132.5 (all C), 132.5, 122.0, 121.9, 117.5 (all C), 41.3, 40.6, 40.4, 39.8, 39.2, 38.6, 38.4 (all CH), IR (ν cm⁻¹), 35.20-3400 (ArO-H_{str} and NH_{str}), 3163-3069 (ArC-H_{str}), 1696-1579 (ArC=C_{str}), 1487 (N=N), 1031 (C-O_{str}), 848 (C-H_{bend}), 788 (C-Cl_{str}), 657 9C-Br_{str}).

Dye 5b. Yield 65%, mp 150 °C; ¹H NMR (200 MHz, DMSO-d₆), 5, ppm: 4.22 (s, 1H, OH_{str}), 6.62 d, 1H, J = 7 Hz, ArH), 7.24-7.45 (m, 2H, ArH), 7.73 (m, 1H, ArH), 7.80-8.20 (m, 4H,

ArH), 8.42 (m, 2H, ArH), ^{13}C NMR (50 MHz, DMSO- d_6), δ_{C} , ppm: 147.2, 135.9, 133.4, 131.6, 128.4, (all C), 123.5, 116.7, 67.5, 41.5, 41.1, 40.8, 40.2, 40.6, 39.5 (all CH); IR (ν cm^{-1}): 3471-3441 (Ar O-H_{str}), 3058-2931 (Ar C-H_{str}), 1614-1583 (Ar C = C_{str}), 1479 (-N=N), 1064 (C-O_{str}), 855 (C-H_{bend}), 759 (C-Cl_{str}), 677 (C-Br_{str}).



Scheme 1. Synthetic route to compound 3 and dyes 5a to 5f.

Dye 5c. Yield 52%, mp 240 °C; ^1H NMR (200 MHz, DMSO- d_6), δ_{H} , ppm: 2.51 (s, 2H, ArH), 6.50-7.53 (m, 6H, naphthyl-H), 7.80-8.22 (m, 3H, ArH), 8.45-8.62 (m, 4H, ArH); ^{13}C NMR (50 MHz, DMSO- d_6), δ_{C} , ppm: 143.7, 141.4, 133.5, 131.7, 131.4, 125.0 (all C), 123.7, 122.2, 118.6, 116.7, 41.8, 41.5, 40.1, 39.7, 39.5, 39.2, 38.6, 38.4 (all CH); IR (ν cm^{-1}): 3423-3288 (Ar-NH $_2$), 3186-3093 (Ar C-H $_{\text{str}}$), 1678-1589 (Ar C=C $_{\text{str}}$), 1479 (-N=N-), 1086 (C-O $_{\text{str}}$), 885, 833 (C-H $_{\text{bend}}$), 786 (C-Cl $_{\text{str}}$), 677 (C-Br $_{\text{str}}$).

Dye 5d. Yield 68%; mp 180 °C; ^1H NMR (200 MHz, DMSO- d_6), δ_{H} , ppm: 2.41 (3, 2H, NH $_2$), 3.84 (s, ^1H , Ar O-H $_{\text{str}}$); 6.72-7.24 (d, 2H, $J = 8$ Hz ArH), 7.44-7.67 (m, 2H, ArH), 7.82-8.24 (m, 3H, ArH) 13.01 (s, ^1H NH); ^{13}C NMR (50 MHz, DMSO- d_6) δ_{C} , ppm: 167.8, 167.7, (all C=O) 145.8, 143.4, 135.2, 132.3, 127.8, 125.5, 118.6 (all C), 76.5, 41.2, 40.8, 40.6, 40.1, 39.6, 38.6, 38.3 (all CH); IR (ν cm^{-1}): 3471-3416 (O-H $_{\text{str}}$ and NH $_{\text{str}}$) 3088 (Ar C-H $_{\text{str}}$), 1726 (C=O $_{\text{str}}$) 1666-1583 (Ar C=C $_{\text{str}}$), 1064 (C-O $_{\text{str}}$), 885, 835 (C-H $_{\text{bend}}$), 788(C-Cl $_{\text{str}}$), 677 (C-Br $_{\text{str}}$).

Dye 5e. Yield 42%; mp 270 °C; ^1H NMR (200 MHz, DMSO- d_6), δ_{H} , ppm: 2.41 (s, 2H, NH $_2$), 4.01 (s, ^1H , Ar OH), 6.54-7.23 (m, 2H, Ar H), 7.50-7.73 (m, 2H, ArH), 7.85-8.10 (m, 4H, ArH); ^{13}C NMR 50 MHz, DMSO- d_6) δ_{C} , ppm: 146.5, 142.6, 132.8, 131.7, 125.6, 124.7, (all C), 116.4, 41.8, 40.6, 40.3, 39.9, 39.7, 39.5, 38.7, 38.2 (all CH); IR (ν cm^{-1}): 3521, 3401 (Ar O-H $_{\text{str}}$, N-H $_{\text{str}}$), 3163-3059 (Ar C-H $_{\text{str}}$) 1695, 1597 (Ar C=C $_{\text{str}}$), 1487 (-N=N-) 1064, 1031 (C-O $_{\text{str}}$), 846 (C-H $_{\text{bend}}$), 786 (C-Cl $_{\text{str}}$) 657 (C-Br $_{\text{str}}$).

Dye 5f. Yield 54%; mp 234 °C, ^1H NMR (200 MHz, DMSO- d_6), δ_{H} , ppm: 3.45 (s, ^1H , Ar OH) 6.74 (m, 2H, ArH), 7.26-7.65 (m, 2H, ArH), 7.87-8.26 (m, 2H, ArH); ^{13}C NMR (50 MHz, DMSO- d_6), δ_{C} ppm: 160.2, 152.5, 143.7, 134.0, 133.6, 124.7, 122.4, 121.8, 115.3, (all C); 41.7, 40.9, 40.5, 40.1, 39.6, 38.8, 38.5 (all CH); IR (ν cm^{-1}): 3423 (Ar O-H $_{\text{str}}$), 3188-3093 (Ar C-H $_{\text{str}}$), 1678-1589 (Ar C=C $_{\text{str}}$), 1479, 1431 (-N=N-), 1066 (C-O $_{\text{str}}$), 885, 833 (C-H $_{\text{bend}}$), 759 (C-Cl $_{\text{str}}$), 677 (C-Br $_{\text{str}}$).

Preparation of dye dispersions

Each disperse solution of the dye sample was prepared by dissolving the dye (1.0 g) in dimethylformamide (DMF) (5 mL) and adding sodium lignin sulphonate (0.5-1.0 gL $^{-1}$) as the dispersing agent. The appropriate volume of the disperse solution was adjusted to 100 mL.

Dye application

Dye dispersion (10 mL) was made up to 100 mL with the dispersing agent and placed in a dyebath. The liquor ratio used was 1:50. The pH of the dye liquor was adjusted to 5.5 by adding a few drops of acetic acid. Polyester fabric (2 g) was dipped inside the dye bath and dyeing was carried out according to high temperature and high pressure method [16]. The dyebath temperature was raised from 20 to 135 °C at a rate of 3 °C min $^{-1}$ and this temperature was maintained for 1 h before cooling rapidly to 50 °C. The dyed fabrics were at this stage rinsed with cold water and reduction cleared in aqueous solution containing sodium hydroxide (2.0 g L $^{-1}$) and sodium hydrosulphite (1.0 gL $^{-1}$). The dyed fabric was then soaped off (washing the dyed fabric with detergent to remove residual dye molecules) with 2% non-ionic detergent and ammonia (pH 8.5) at 50 °C for 30 min.

Colour fastness to washing test

The test for colour fastness to washing was determined using the ISO 105-CO6:2010 test method [17]. The cut dyed samples were sewed between two bleached pieces of wool and cotton fabric and placed into a bath containing an aqueous solution of 5 gL $^{-1}$ of a detergent (non-ionic)

of liquor ratio 1:50. The bath was maintained thermostatically at 60 °C for 30 min. After which, the samples were removed, rinsed and hand squeezed twice and dried. The washing fastness was then evaluated by comparing with the standard greyscale for change of color.

Color fastness test to rubbing

Color fastness to rubbing was measured according to ISO 105-X12: 2001 method [18]. The test is designed to ascertain the amount of color that could be transferred from the surface of the colored specimen to adjacent white fabric surface by rubbing. This test can be done on wet and dry fibres.

Wet rubbing test

The dyed polyester fabric specimen was thoroughly wetted with water and placed flat at the base of the tester. The white testing cloth was also mounted and lowered unto the test specimen. The finger of the tester carrying the weight of 2-6 N was placed in contact with the test specimen and was made to slide back and forth several times until ten complete turns was made at a rate of 1 turn s⁻¹. The staining on the white test sample was then evaluated using the standard greyscale.

Dry rubbing test

The dyed polyester fabric specimen was placed flat at the base of the tester. The procedure was then run as described for wet rubbing fastness test. The white test sample, 60 mm square, was removed and evaluated for staining using the grey scale (grade 1-5).

Colour fastness to sublimation test

The dyed polyester fabric specimen was subjected to sublimation treatment according to ISO 105-POI 1993 method [19]. The test was carried out at 177 °C for 30 s. The polyester fabrics were then evaluated for colour change using the standard greyscale (grade 1-5) where 1 (poor), 5 (excellent).

Colour fastness to light

The dyed polyester samples were exposed to xenon arc lamp involving the use of continuous light for 35 h. The change in color of the tested samples was recorded by comparing with the standard blue scale for color change. This test was carried out in accordance with ISO 105-BO2: 1988 method [20].

RESULTS AND DISCUSSION

The compounds yield range from 42-68 % and the melting points of the dyestuffs vary from 150-270°C. The dyes with one hydroxyl group exhibited high melting points (170-270°C) while the lowest melting point was found for dye 5b (150°C). The reason for this low melting point of dye 5b could be attributed to the substituents on the benzene ring of the coupling components. All the dyes possess good solubility in dimethylformamide.

The FTIR spectra of the synthesised compounds showed a strong band between 3423 and 3520 cm⁻¹ region, which corresponds to O-H or N-H stretching vibrations [21]. Absorption bands of C-H in the IR spectra were observed between 3093 and 3186 cm⁻¹. In the ¹H NMR spectra, the broad singlet peaks which appeared at 6.72-7.73 ppm suggest the presence of OH in the synthesised dyes [22]. Absorption bands of C=C stretching vibration were observed at 1583-1675 cm⁻¹ region, suggesting the presence of aromatic rings.

All the sp^2 carbons attached to the more electronegative atoms of the benzene rings such as $-Br$, $-Cl$ and $-OH$, showed absorptions at δ 141–167 ppm. This indicates that the sp^2 carbons gave absorption down field due to the de-shielding effect of the carbons by the electronegative atoms. The other sp^2 carbons of the aromatic compounds showed absorptions at δ 115–130 ppm. The absorption peaks observed at δ 38–67 ppm were assigned to sp^3 carbons.

UV-Visible spectroscopic studies

The results of the UV-vis spectroscopic analysis are presented in Table 1. The UV-vis spectra of the dyes exhibited typical electronic spectra with multiple absorptions in the UV region at about 274–400 nm also referred to as the B-band, and the others were observed in the visible region at 450–800 nm (Q-band). The Q-band is ascribed to $n-\pi$ transition from the highest occupied molecular orbital (HOMO) to the lowest unoccupied molecular orbital (LUMO) of the π -conjugated chain of the dye molecules [23, 24]. The absorption maximum of dye **5a** occurred at 573 nm with no shoulder. That of dye **5b** was observed at 646 nm with an intense and broad shoulder over 750 nm. Dye **5c** gave an absorption maximum at 596 nm and a shoulder extending into the near infrared region over 650 nm. The absorption maximum of dye **5d** is 604 nm and a red-shift shoulder at 784 nm, extending and increasing beyond 800 nm. Dye **5e** gave absorption maximum at 464 nm with an intense shoulder, extending over 800 nm. Dye **5f** showed absorption maximum at 496 nm which extended beyond 600 nm. Thus, the absorption maxima observed for the dyes appear to have been influenced by the aromatic substitution pattern of the rings. The multiple absorptions are attributed to the non-conjugative effects of the various chromophores present in the molecules [24, 25]. And the near-infrared absorption maxima observed in the dye compounds is a function of extensive electron delocalisation within the ring system along the chain, as well as the interaction of this delocalisation with the available heavy bromine atom.

Table 1. Absorption maxima of disazo disperse dyes **5a** to **5f** in dimethylformamide solution.

Dye	B-Band (λ_{max})	Q-Band (λ_{max})	R_f
5a	274	573	0.60
5b	370	646, 750	0.59
5c	267	596, 650	0.62
5d	345	604, 784	0.74
5e	352	464, 800	0.82
5f	400	496, 576	0.70

Fastness to washing

All the dyes showed excellent fastness to washing of grade 5 (Table 2). Generally, the dyes are characteristically hydrophobic so high washing fastness values were expected according to standard greyscale. This may be attributed to an increase in the molecular size of the dyes and sufficient intra-fibre diffusion of molecules of the dye within the fabrics.

Table 2. Wash fastness properties of dyed polyester fabric (2% owf).

Dye	Change in color	Staining		
		Cotton	Polyester	Nylon
5a	5	5	5	5
5b	5	5	5	4-5
5c	5	5	5	4-5
5d	5	5	5	5
5e	5	5	5	5
5f	5	5	5	4-5

Fastness to rubbing

The test was actually carried out to determine the degree of color transfer from the surface of the dyed fabric to another adjacent undyed fabric surface by rubbing. The results in Table 3 showed that most of the dyes exhibited good rubbing fastness values of grade 4 and 3-4 which implies good, or very good results based on the standard greyscale. This performance may be attributed to sufficient dye diffusion into the fabrics.

Table 3. Sublimation, light and rubbing fastness properties of dyed polyester fabric.

Dye	Sublimation	Light	Rubbing	
			Dry	Wet
5a	5	5-6	3-4	3-4
5b	5	5	4	4
5c	5	6	3-4	3-4
5d	5	5	3-4	3-4
5e	5	5-6	4	3-4
5f	5	6-7	4	4

Fastness to light

The prepared dyes showed satisfactory fastness to light, ranging from grade 5 to 6-7 according to the standard blue wool for change of color. The light fastness performance could be attributed to the presence of electron withdrawing groups in the vicinity of the azo group.

Fastness to sublimation

The fastness to sublimation treatment of the synthesized dyes was expressed in terms of color staining on undyed polyester pieces. The results (Table 3) show good to excellent ratings (grade 4 to 5). Generally, fastness to sublimation depends upon the substituents on the coupling component ring as well as those on the diazo component ring. Thus, substituents such as OH, NH₂ and other polar groups contributed to the high sublimation fastness of the dyes on polyester fabrics [26].

CONCLUSION

This study presented the synthesis and characterization of new sets of azo dyes and their fastness properties on polyester fabrics. The UV-Vis data of the synthesized azo compounds showed that they are potential near infrared absorbers and could be used as organic photoconductors and similar applications. The synthesized compounds showed good fastness properties on polyester fabrics and could be found useful in the production of textiles.

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