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Synthesis of disazo disperse dye compounds derived from 4-bromoaniline and 3-aminophenol as potential near infra-red absorbers.

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Abstract

In attempt to find new near infra-red absorbing azo compounds, the synthesis and characterization of six disazo compounds are reported. The azo compounds were synthesized via diazotization of 4-bromoaniline and coupled with 3-aminophenol to afford 4-bromophenylazo-4¹-amino-2-hydroxybenzeneazo dye intermediate. This intermediate was further diazotized and coupled with various aryl-amine and phenolic couplers to give a series of disazo disperse dyes. The UV-VIS spectral properties of the dyes were evaluated and some of the dye derivatives bearing electron withdrawing groups in their couplers showed absorptions at 772nm to 786nm in the near infra-red region in dimethylformamide solution. This, in addition to their relatively high thermal stability would enhance their use as potential organic photoconductors. The results of the fastness tests of the dyes on nylon fiber and polyester fiber showed excellent sublimation, washing, good rubbing and light fastness ratings. This also indicates that the azo compounds could be of commercial importance in the textile industry. The dyes were further characterized using proton nuclear magnetic resonance (¹HNMR), carbon- 13 nuclear magnetic resonance (¹³CNMR) and infra- red (IR) analyses.

Keywords: Disazo dyes, spectral properties, disperse dyes, fastness, nylon fiber, polyesterfiber.

Introduction

The use of dyestuffs in coloring textile fibers including polyesters and other materials of industrial importance, led to the constant effort to find specific dye or a particular class of dyes for application to such diverse materials as textile fibers, aluminum sheets, inhibition of metal corrosion, leather, electro-optical devices, ink-jet printers and organic photoconductors (Otutu, 2012; Dixit *et al.*, 1994; Ortega *etal.*, 2007; Yildiz and Bortepe, 2008; Kirkan and Gramazan, 2008). In textile applications, azo disperse dye types have been explored for a long time (Otutu *et al.*, 2012; Towns, 1999). And in the area of non-textile applications, so many different classes of dyestuffs such as azos, perylenes, phthalocyanines, vat types, and cyanine dyes have been claimed to be useful organic photoconductors. For instance, many azo dye compounds especially those in form of pigments have been covered by many patenting companies which use them as organic photo-conductors. Some of these azo compounds are dis- ortrisazo types (Loutgy *et al.*, 1988; Bawans and Sawant, 2003). Thus, literature review revealed that no publication yet describe the use of these two intermediates for the synthesis of disazo dyes.

In this study, we report the synthesis and characterization of disazo dyes derived from 4- bromoaniline and 3-aminophenol as diazo components. The main purpose of the study is to describe the spectral properties of the disazo dyes from which the potential for application as organic photoconductors can be deduced. Also, the fastness properties of the dyes on nylon fabrics and polyester fabrics are described.

Experimental

Synthesis of compound 3

Concentrated sulphuric acid (9.0ml) was added to a mixture of 4-bromoaniline (10g, 57.5mmol) and 50ml water. Sodium nitrite (5.0g, 63.mmol) in water (10ml) was added drop wise to the above mixture at 0-5^oC and stirred for 30mins. Excess of nitrous acid was removed by the addition of urea (15g, 0.03mmol). The resultant diazonium salt solution was added to the solution of 3-aminophenol (6.3g, 57.8mmol) in 1.5gsodium hydroxide solution (20ml) and stirred vigorously for one hour. The precipitate formed was isolated by filtration, washed with distilled water and dried. The crude product was recrystallized from hot carbontetrachloride to afford compound 3 in 16.52g (97%) yield.



Figure 1: Synthesis scheme for the preparation of compound 3 (i) $H_2SO_4/NaNO_2$, 0-5 0 C (ii) $C_6H_7N_1O$

Synthesis 2, 4-dihydroxyphenylazo-2¹- hydroxyphenylazo- 4'-bromobenzene (5a).

Compound 3 (3.0g, 10.3mmol) was diazotized in 5ml of concentrated sulphuric acid and 30ml water by adding sodium nitrite solution (2.5g, 31.6mmol) in 10ml water at 0.5° C with stirring for 25mins. The diazonium salt solution obtained was added to a solution of 1, 3- dihydroxybenzene (1.13g, 10.3mmol) in (1.0g, 25mmol) solution of sodium hydroxide dissolved in 20ml water and stirred for one hour. The product was filtered, washed with distilled water and dried. The crude product was purified using carbontetrachloride to give brown solid of compound 5a.m.p: 258- 260 $^{\circ}$ C, yield 50%, IR (Nujol/cm $^{-1}$)v_{max}: 3522, 3404(OH), 3119, 2812 (C- H_{str}), 1654, 1597, 1508 (Ar-ring), 1489, 1388 (-N=N-) 1313, 1211, 1134 (C-O_{str}), 661.30 (C- Br_{str}). 1 HNMR (50.28 MHzDMSO-d₆): δ_{H} /ppm:6.21 (1H, d, J = 8.2Hz,ArH), 6.42- 7.40 (5H, m, ArH), 7.60- 7.65 (2H, m, ArH), 10.60 (1H, s, OH). 13 CNMR (50.28 MHz,DMSO-d₆) δ_{C} /ppm: 143.03, 141.21, 131.65, 123.43, 118.43, 117.61, 105.02, 41.27, 40.56.

4- Hydroxynaphthaleneazo-2-hydroxyphenylazo-4-bromobenzene (5b)

Compound 5b was synthesized using the above procedure with 1- naphthol as coupling component. Brown solid m.p: 214-216, yield 68%, IR (Nujol/cm⁻¹) ν_{max} : 3446 (OH), 3095, 2968, 2864 (C-H_{str}), 1647, 1624 (Ar-ring) 1595(naphthalene-ring) 1458, 1384 (N=N-), 1220, 1111 (C-O_{str}), 660 (C-Br_{str}). HNMR (50.28MHz,DMSO-d₆) δ_{H} /ppm: 6.23 (2H, d, J = 8.2 Hz, ArH) 6.40- 7.44 (6H, m, naphthalene) 7.62- 7.73 (5H, m, ArH) 7.83 (2H, m, ArH) 10. 62 (1H, s, OH). CNMR (50.28MHz,DMSO-d₆) δ_{C} /ppm: 142.76, 141.32, 132.45, 131.60, 131.52, 131.46, 125.44, 123.64, 121.00, 118.53, 41.17, 40.56, 40.36.

2, 4- dihydroxyphenylazo-2¹- hydroxyphenylazo-4-bromobenzene (5c)

Compound 5c was synthesized using the procedure above with 1,5-dihydroxybenzene as coupling component.Brown solid yield 74%, mp: $210\text{-}212^{0}$ C, IR (Nujol/cm⁻¹)v_{max}:3649, 3446, 3387 (OH_{str}), 3101 (C-H_{str}), 1616, 1575, 1541, 1508 (Ar-ring), 1458 (-N=N-), 1251 (C-O_{str}), 669.32 (C-Br_{str}). ¹HNMR (50.28MHz,DMSO-d₆) δ_{H} /ppm: 6.22 (2H,d, J = 8.3Hz, ArH), 10.61 (1H, S, OH) ¹³CNMR (50.28MHz,DMSO-d₆) δ_{C} /ppm: 146.18, 134.96, 132.71, 131.51, 129.70, 124.54, 119.44, 118.42, 66.46, 41.14 40.59, 40.30, 40.20, 39.38, 39.55.

$\hbox{\bf 4-chloro-2-} a min ophenylazo-\hbox{\bf 2-hydro} xyphenylazo-\hbox{\bf 4-bromobenzene} \ (5d)$

Compound 5d was synthesized using the procedure described for dye 5a with 2-chloroaniline as coupling component, dissolved in 5ml acetic acid to afford brown solids. Yield 50%, mp: 105° C. IR(Nujol/cm⁻¹) v_{max} : 3423 (NH₂, OH) 3095 (C-H_{str}), 1616, 1581, 1508 (Ar-ring), 1487, 1404 (-N=N-), 1211 (C-O_{str}), 827 (C-Cl_{str})726, 646 (C-Br_{str}) 4.00 (IH, s, OH), 2.45 (2H, s, NH₂), HNMR (50.28MHz,DMSO-d₆) δ_{H} /ppm: 6.20 (2H, d, J = 8.3 Hz, ArH), 6.60 – 7.40 (4H, m, ArH), 7.45- 7.70 (2H, m, ArH). 13 CNMR (50.28 MHz,DMSO-d₆) δ_{C} /ppm: 145.02, 141.61, 132.75, 124.47,118.53, 117.51,105.00,41.17,40.76,40.34,39.93,39.09,38.67.

4-Amino-2-hydroxyphenylazo-2¹-hydroxyphenylazo-4-bromobenezene (5e)

Compound 5e was synthesized using the procedure similar to that described for compound 5a with 3-aminophenol as the coupling component to give brown crystals. Yield 64% mp: 145° C, IR (Nujol/cm⁻¹) v_{max} : 3520, 3404 (NH₂, OH_{str}),3173, 3061, 2816, (C-H_{str}), 1660, 1595, 1510(Ar- ring), 1487, 1390 (-N=N-), 1211.34 (C-O_{str}), 646.17 (C- Br_{str})¹HNMR (50.28MHz,DMSO-d₆) δ_{H} /ppm: 2.46 (2H, S, NH₂), 6.20 (2H, d, J = 8.2Hz, ArH), 6.40- 7.26 (5H, m, Arh), 7.41- 7.80 (3H, m, ArH), 10. 60 (1H, s,OH), ¹³CNMR (50.28MHz,DMSO-d₆) δ_{C} /ppm: 156.19, 134.94, 132.81, 131.41, 129.80, 124.55, 119.54, 118.52, 66.56, 41.15, 40.79,40.90, 39.48, 39.07, 38.65.4-amino-2-cyanophenylazo-2¹-hydroxyphenylazo-4-bromobenzene (5_f)



Compound 5_f was synthesized by using the procedure similar to that described for compound 5d with 2-aminonitrile as coupling component to give brown solid yield: 55° Cmp: 130° C; IR (Nujol/cm⁻¹) ν_{max} : 3423 (NH₂, OH_{str}) 3095 (C-H_{str}), 2225 (C=N), 1614, 1581, 1510 (Ar-ring), 1467, 1404 (-N=N-), 1220 (C-O_{str}) 646 (C-Br_{str}). 1 HNMR (50.28 MHz,DMSO- d₆), δ_{H} /ppm: 2.81 (2H, s, NH₂), 3.98 (1H, s, OH) 6.20 (2H, d, J = 8.1Hz, ArH), 6.40- 7.21 (5H, m, ArH) 7.42- 7.65 (2H, m, ArH), 7.81 (1H, m, ArH). 13 CNMR (50.28 MHz,DMSO-d₆) δ_{C} /ppm: 145.04, 141.62, 132.92, 132.76, 124.48, 124.09, 118.54, 41.18, 40.76, 40.35, 4035, 39.92, 39. 50,9.09, 38.68.

Materials

4-Bromoaniline, 3-amninophenol, 3-chloroaniline, 2-aminonitrile, 1, 3-dihydroxy benzene, 1-naphthol and 1, 4-dihydroxybenzene were obtained from Merck (Pure) and used without further purification. All the other reagents, such as $conc.H_2SO_4$, $NaNO_2,NaOH$ Pellets, and acetic acid were commercially available as pure samples, and used as received.

Analytical Methods

Melting points were uncorrected and obtained on an Electrothermal9100 apparatus. Infrared spectra (IR) were recorded using a Shimadzu (84005) FT-IR spectrometer. Thin Layer Chromatography (TLC) was used to follow the course of the reactions (Chloroform-Methanol (95:5),in order to monitor the dye purity,(R_f values) using silica gel 60 coated plates F_{254} (Merck aluminum sheets), H (50.28MHz) and 13 C(50.28MH₂)NMRs were recorded on a Mercury 200BB series spectrometer. DMSO-d₆ was used as NMR solvent. Chemical shifts were reported in parts per million (ppm) downfield from internal tetramethylsilane (TMS). All the ultraviolet-visible absorption spectra were recorded on Genesys10sV1.20 series spectrophotometer.

Dye Application Polyester (Tetron)

The dye baths were prepared with the disazo dyes 5a- 5_f using Lignosulphonate (Sulphite cellulose-Liquors) as the dispersing agent. The dyes (1.0g) were dissolved in dimethylformamide followed by adding the dispersing agent at Liquor to material ratio of 20:1. The pH of the dye Liquor was adjusted to 5 with 2% acetic acid. The polyester fabric was dipped into the dye bath, and dyeing was continued for 60 minutes at 130^0 to 135^0 C under increased pressures. The dyed polyester fabric was subjected to reduction- rinsing treatment at 85^0 C for 10 minutes in a solution of sodium hydroxide ($2gl^{-1}$),hydrosulphite ($2gl^{-1}$) and a betaine amphoteric surfactant ($2gl^{-1}$) in water, washed with water and then dried.

Polyamide (nylon 66)

The dye bath was first prepared by mixing the dye solution in dimethylformamide and with Lenacid NL (1.2 ml of 10%) as an acid buffer (pH) 5 to 5.5) and water (18ml). The nylon 66 fabric (2.0g) was wetted and dipped into the dye bath at 45° C. The dye bath temperature was increased to 102° C over 15 minutes and maintained for 30 minutes. At this temperature, formic acid (1.5ml of 10%) was added to the dyebath to achieve good exhaustion (Ukponmwan *et al.*, 1999). The dye bath was then cooled, and the dyed fabric was rinsed and reduction- rinsed twice with Iodet (2gl⁻¹) in a bath containing water (material to Liquor 1:2) at 40° C for 15 minutes. The dyed fabric was then washed thoroughly with water and air-dried.

Fastness Standard Tests

Wash Fastness

The wash fastness was evaluated according to International Standard Organization (ISO) washing test number 3 using Atlas Lintiest machine (ISO, 105-CO6, 1990). The dyed fabric samples measuring (10 x 5cm) were composited and treated with a solution consisting of $5g\Gamma^1$ – soap and sodium carbonate $2g\Gamma^1$ of Liquor ratio 50:1 for 30 minutes at 60 0 C. The changes in shades and staining to adjacent multi fiber were related to the standard grayscale rating whose indications are 1 to 5 (where 1 is poor and 5 is excellent).

Light Fastness

Light fastness was tested by exposing the dyed fabrics to the Xenon arc fading Lamp of an Alas 3 SUN weather-Ometer, according to the following conditions (ISO, 1994). Black panel temperature 63°C, dry bulb temperature, 43°C, relative humidity 30%, duration of test is 24h. The change in shades under artificial light were evaluated according to standard blue wool fabrics (grade 1 to 8) where 1 is poor and 8 is excellent.



Sublimation Fastness

The heat (Sublimation) fastness was assessed according to ISO. 105-PO1 1993 protocol (ISO, 1993). The dyed samples were treated in a sublimation testing machine at 177° C for 30sec and the change in shades were related to the standard grayscale rating (grade 1-5) where 1 is poor and 5 is excellent.

Rubbing Fatness

The fastness to rubbing was tested using Atlas rubbing fastness tester according to ISO 105- X12 method. The changes in shades were also related to the standard grayscale rating (where 1 is poor and 5 is excellent).

Results and Discussion

Synthesis and characterization

The disazo disperse dyes 5a- 5_f were obtained in 50- 74% yield as brown, solids from the coupling reactions of the diazonium salt solutions of the azo compound 3 with a variety of coupling compounds as illustrated in figure 2.

The assumed structures of the disazo compounds were established by FT-IR, 1 HNMR 13 CNMRand UV-Vis spectra. The FTIR spectra of the dyes showed strong broad bands at 3404- 3649cm $^{-1}$ region due to N-H and OH stretching vibrations.In the IR spectrum of compound 5_f , the v C \equiv N vibrations occurred as strong absorption at 2225 cm $^{-1}$, indicating the nitrile group. The FT-IR spectra of the dyes also showed strong bands at 646 – 736cm $^{-1}$ due to C- Br stretch bonds.

Figure 2: Synthesis Scheme for the preparation of dyes 5a - 5f (i) HONO, 0-5°C, (ii) CsH₂O (iii) C₁₀H₂O, (iv) CsH₂O (v) CsH₂NCl (vi) C₃H₃NO (vi) C₁H₂NO (vi) C₁H₂NO

The structures of the prepared compounds were also, confirmed by ¹HNMR spectra. The proton signals displayed chemical shifts and multiplicities corresponding to their surroundings. The ¹HNMR spectra displayed the signals of pairs of protons in the 6.20-6.23ppm range with coupling constants of 8.1-8.2H_Z. In agreement with the presence of aromatic rings, the ¹HNMR spectra of the compounds display the signals of 6.42-7.81 ppm range, Moreover, as expected, the ¹HNMR spectra of the dyes showed a distinctive signal at 10.60-10.62 ppm for the OH proton for compounds 5a-5d, and at 3.98-4.00ppm for the same proton for compounds 5d-5_f. The difference in the chemical shifts is due to the concentration effects (Paula, 1995; Ukponmwan*et al.*, 1999). The complete chemical shifts for all compounds (¹HNMR and ¹³CNMR) are listed in thematerials and methods section. All the results were consistent with the predicted structures given in figure 2.

UV-Vis Absorption Spectra

UV-Vis absorption spectra of these disazo dye molecules in diluted dimethylformamidesolutions are listed in Table 1. As shown, the absorption spectra of 5d, 5e and 5_f gave two peaks at 584 and 785nm, 584 and



785nm, and 488 and 738nm respectively. This indicated that these colorants absorbed in the infra-red region. Moreover, the maximum absorption peaks of dyes 5a, 5b and 5c were at 401, 406 and 410nm respectively. Therefore, the presence of sufficiently electronegative substituents in both the diazocomponents and the couplers, conducive to bathochromism contributed to the push of absorptions into the near infrared in compounds 5d, 5e and $5_{\rm f}$. Actually, compared with compounds 5a, 5b and 5e which possess a less extended π -conjugated system, leading to the lower maximum absorptions observed. The values of the logarithm of the molar extinction coefficient (Log ϵ) of the colorants are 4.41, 4.46, 4.47 and 4.95, 4.30, 4.80 which are consistent with their respective intensities.

The retention factor (R_f) values of the dyes are summarized in Table 1.The method of establishing the distance travelled by a dye under the influence of a given solvent system, is provided by the R_f value. This refers to the ratio of the distance travelled by the dye to that covered by the solvent-system, as measured from the starting line. The R_f value of a compound lies between zero and unity. The disazo dyes as shown by the assumed structures have the same basic skeleton except for the substituents on the coupling components that are having different characters. Thus, the R_f values of dyes5a, 5b and 5e with Br and OH as the substituents are 0.34, 0.25, 0.32 respectively. The positions and characters of the substituents in these dyes are quite similar hence the similarly low R_f values. Whereas dyes 5d, 5e and S_f , which contain other substituent groups such as -NH₂, -CNand -Cl in different positions, in addition to the -Br and -OH groups showed higher R_f values of 0.48, 0.42, 0.51 respectively. This indicated that they may be having smaller dipole moments hence the higher R_f values.

Wash Fastness

In Table 2, the wash fastness results are summarized and all the dyes showed colour change of 5, that is excellent ratings on the polyester and nylon fabrics except dyes 5e and 5_f which gave good to excellent ratings (4/5) on nylon. In general, this performance is an indication that the dyes have strong affinity for the two substrates. This may be attributed to the large molecular size of the dyes.In addition to this, is the fact that the shades of the dyes on the nylon fabrics were observed to be deeper compared to the shades on polyester fabrics. This indicates that the dyes showed more exhaustion on the nylon fabric than on polyester. As seen from the results in Table 2, there wasslight staining of the adjacent cotton in the case of dye 5a composited with dyed polyester fabric and also slight staining of adjacent cotton of dyed nylon with dyes 5b and 5c. No staining on the adjacent multi-fiber fabric was observed in adjacent dyed nylon, polyester and acrylic.

Light Fastness

The light fastness of the dyes on polyester fabrics and nylon fabrics are listed in Table 3. And as seen from the results, the photo stability of the nylon fabric (grade 6) tends to be higher than that of polyester (grade 5/6). This is due probably to the position of the hydroxyl substituent group which is ortho to the azo groups in the dye structure. This has been found to increase light fastness of nylon dyed disperse dyes and also found to decrease those of polyester fiber (Otutu *et al.*, 2008). Dye 5_f , however, gave a good light fastness of grade 6 on both fibers. This may be due to the presence of the $C \equiv N$ group in the dye structure which has been found to increase the photostability of disperse dyed polyester fabric and nylon fabric.

Table 1: Absorption maxima and intensities of dyes $5a - 5_f$ in dimethyl formamide solution (DMF).

Dye	λ _{max} (nm)	$\mathbf{R_f}$	$\varepsilon_{\text{max}} (\text{dm}^3 \text{mol}^{-1} \text{cm}^{-1})$	Intensity (log ε)		
5a	401	0.34	26146	4.42		
5b	406	0.25	29046	4.46		
5c	401	0.32	29693	4.47		
5d	584, 785	0.48	8709	4.94		
5e	584, 786	0.42	20178	4.30		
$5_{\rm f}$	488, 738	0.51	63789	4.80		



Table 2: Wash fastness ratings of dyed polyester fabrics and nylon 66 fabrics

Dye	Types of fabric	Light	Sublimation	Rubbing		
				Wet	Dry	
5a	Polyester	5/6	5	4	4/5	
	nylon	6	4/5	4	4/5	
5b	Polyester	5	5	4	4/5	
	nylon	6	4/5	4	4	
5c	Polyester	5/6	5	4	4/5	
	nylon	6	5	4	4	
5d	Polyester	5/6	5	4	4/5	
	nylon	6	3/4	4	4	
5e	Polyester	5/6	5	4	4/5	
	Nylon	6	5	4	4/5	
$5_{\rm f}$	Polyester	6	4/5	4	4/5	
	nylon	5	5	4	4	

Table 3: Light, Sublimation and rubbing fastness rating of dyed polyester fabrics and nylon fabrics.

Sublimation Fastness

The sublimation fastness results of the dyes are presented in Table 3. The results showed excellent ratings of grade 5. This is attributed to the large molecular size and increase in polarity of substituents in the dye structures which enhance high sublimation fastness.

Rubbing Fastness

Table 3, show the results of the rubbing fastness of the dyes on polyester fabric and nylon fabric. The dry rubbing fastness properties of the dyed samples varied generally between 4 and 4-5 while the wet rubbing

Dye	Type of fabric	Colour	Staining			Colour on	
		change	Cotton	Nylon	Polyester	Acrylic	fabric
5a	Polyester	5	4/5	5	5	5	Brown
	nylon	5	5	5	5	5	
5b	Polyester	5	5	5	5	5	Brown
	nylon	5	4/5	5	5	5	
5c	Polyester	5	5	5	5	5	Orange-
	nylon	5	4/5	5	5	5	brown
5d	Polyester	5	5	5	5	5	Brown
	nylon	5	5	5	5	5	
5e	Polyester	5	5	5	5	5	Brown
	nylon	4/5	5	5	5	5	
$5_{\rm f}$	Polyester	5	5	5	5	5	Brown
1	nylon	4/5	5	5	5	5	

fastness properties varied between 3/4 and 4. The slight difference may be attributed to the fact that in wet rubbing the dyed samples are likely to lose some colour than when dry as water molecules do assist in the colour removal during rubbing.

Conclusion

The synthesis and characterization of disazo dyes using spectral methods, have been reported. The present work indicated, from the UV-visspectral results, that some of the dyes absorbed in the infrared region and are therefore potential organic photoconductors. The results of the fastness tests of the dyes on polyester



fabric and nylon fabric also demonstrate that the best of these dyes could be of commercial importance in the textile industry.

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