

SYNTHESIS OF DISPERSE DYES AND THEIR APPLICATIONS ON SYNTHETIC FABRICS



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Abstract:	bstract: Seven diazo disperse dyes based on 2-chloroaniline and 4-methoxy-2-nitroaniline components were synthes the traditional diazotization coupling strategy. The dyes were characterized using proton nuclear m resonance (¹ H NMR), ¹³ C nuclear magnetic resonance (¹³ C NMR), Fourier transform infrared (FTIR) and visible spectroscopy analyses to confirm the structures. The dyes were applied onto polyester and nylon					
	The results of the fastness tests on polyester substrate were observed to be technically better compared to those on nylon 66 fabric in terms of light fastness (grade 6-7). However, the results of the wash, sublimation and rubbing fastness on both fabrics were similar (grade 4 to 4/5) with little variations. From the technical point of view, it can be concluded that the dyes could meet the requirements to be utilized for dyeing textile fabrics meant for window blinds (curtains) as well as in automobile seat covers.					
Keywords:	Diazo disperse dyes, synthesis, textile fabrics					

Introduction

During the past decades disperse azo dyes have attracted considerable attention by researchers due to their inherent advantages and wide applications. They are the only type of dyes most suitable for dyeing polyester and acetate fibres. Disperse dyes have also found use in non-textile areas such as non-linear optics, chemosensors, radical reaction initiators, liquid crystal displays and optical storage media (Park *et al.*, 2010; Zhang *et al.*, 2017; Choi *et al.*, 2013). It is also worth mentioning that disperse azo dyes have been applied in the dyeing of nylon and acrylic fibres and most recently polybutylene fibres (Zhang *et al.*, 2013). Nevertheless, the solubility of disperse azo dyes is relatively low in polar solvents such as water etc and this property impedes their use in dyeing natural fibres (Kim *et al.*, 2010; Metwally *et al.*, 2013).

Consumers of many types of apparel always require that textile goods should possess or exhibit high durability in terms of sublimation and photostability, especially those that are meant to be used as curtains or window blinds. These two key fastness properties of dyes on dyed fabrics are also important for textiles used in the automobile industry as car seat covers (Burkinshaw, 1994).

In one of the previous papers in the series, the application of disperse azo dyes prepared from 4-merthoxy-2-nitroaniline applied onto textile fibres using the aqueous based exhaust dyeing method has been reported (Otutu *et al.*, 2014). The aim of the present study was to design and synthesize disperse azo dyes that could meet the aforementioned requirements (high sublimation and light stability) when applied onto textile fibres. In doing this, the diazo component was designed to contain electron-withdrawing substituents that could lead to improved light fastness. Furthermore, efforts were made to include polar groups in the dye structure to discourage their tendency to sublime. Finally, the dyes were applied onto polyester fabrics using thermofixation process and aqueous exhaust dyeing method for nylon 66 fabrics.

Material and Methods

The starting materials, 4-methoxy-2-nitroaniline, 2chloroaniline and the coupler compounds were purchased from Sigma-Aldrich (Germany) and were used as obtained. Synthesized compounds were purified by recrystallization from ethanol and identified and characterized by proton nuclear magnetic resonance (¹H NMR) and carbon thirteen nuclear magnetic resonance (¹C NMR) (Varian 200BB series spectrometer) using DMSO-d6 and Fourier transform infrared (FTIR). The UV-visible absorption spectra were measured using a Jasco V-670 spectrophotometer (Jasco, Japan). The purity of the dyes was verified using thin layer chromatography (TLC), Merck silica-gel 60 (Germany). Infrared spectra were recorded on a Nicolet 5700 FTIR (Fourier transform infrared) instrument (Thermo Scientific, USA) (with a spectra range of 4000 - 400 cm⁻¹ and spectral resolution of 0.25 cm⁻¹ using solid samples dispersed in KBr pellets.

Synthesis of dyes

The amine for diazotization, 4-methoxy-2-nitroaniline (7 mmol, 2.0 g) was mixed with water (20 mL) and concentrated sulphuric acid (5 mL) and cooled to 0-5 °C in an ice bath. Sodium nitrite (8 mmol, 1.0 g) was dissolved in cold water and added drop wise to the reaction mixture for 30 min under mechanical stirring. The residual nitrous acid (HONO) was destroyed by adding a small amount of sulphamic acid to obtain the diazonium salt solution. The freshly prepared active diazonium salt was added drop wise for 30 min to the coupling compound, 2-chloroaniline (2.5 mL) dissolved in acetic acid (5 mL) under vigorous stirring. After further stirring for 3 h the reaction mixture was neutralized with ammonia to pH 5-6, and the precipitated crude was filtered and dried after thorough washing with distilled water. The crude product was recrystallized with hot acetonitrile and the brown solid was collected in a yield of 87%.

Compound 3a to 3g were synthesized by diazotizing the intermediate dye 3 as described above using different couplers such as 4-bromoaniline (7 mmol, 1.12 g), 4,6-dihydroxypyrimidine (7 mmol, 0.73 g), 3-aminophenol (7 mmol, 1.04 g), 2-hydroxybenzaldehyde (7 mmol, 1.12 g), 6-hydroxyquinoline (7 mmol, 1.04 g) and 8-hydroxyquinoline (7 mmol, 1.04 g). The retention factors of the dyes were evaluated using thin layer chromatography.



Scheme 1: The chemical structures of the synthesized azo dyes 3a to 3g



Scheme 2: Thermofixation dyeing process of polyester fabric

Dye Application Polyester fabrics

Each dye (1.0 g) was dissolved in dimethylformanide (DMF) (5 mL) and a dispersing agent, Dianix ECO as added. The final volume of the dispersion was made up to 100 mL. The

dye dispersion was used to impregnate the polyester fabric which was slowly passed under the surface of the dye dispersion. This process was repeated for each fabric sample. Using a laboratory padding mangle, the fabric was padded at a pressure of 2.0 kgcm⁻¹ and padding rate of 3 m min⁻¹. At the end of this, the padded fabrics were transferred to the oven unit for thermal fixation of the dye using dry heat method to enable the dye to diffuse into the fibre. The temperature was optimized at $150 - 230^{\circ}$ C for a period of 30 - 60 sec. after this, the dyed sample was removed and rinsed with cold water. This was followed by a reduction-clearing process, using an aqueous solution containing sodium hydroxide (2.0 gl⁻¹) and a non-ionic surfactant (synperonic BD 100, 1.0 gl⁻¹), to remove unfixed dye molecules, the sample was then rinsed with water several times and air dried.

Nylon 66 fabric

The dye bath was prepared as described for polyester dyeing. The pH of the dye dispersion was adjusted to 4.5 using a 2% acetic acid. The nylon 66 fabric (2.0 g) was wetted and dipped into the dye dispersion in the dye at 4°C. As dyeing commenced, the temperature of the dye bath was raised to 102°C for over 15 min and then maintained for 3 min. To the dye bath, 1.5 mL of 10% formic acid was added to raise dye bath exhaustion. The dyes sample was then removed from the dye bath, rinsed with water and reduction-clearing with iodet T (2.0 gl⁻¹) in water, in a bath of liquor ratio 1:2 at 45°C for 15 min. The treated dyed nylon 66 fabrics was then thoroughly rinsed with cold water and dried in air.

Fastness tests

The colour fastness of the dyes on the dyed fabrics were tested according to International Standard Organization methods

including fastness to light using ISO 105-B02; 2014 (ISO, 2013), the dyed samples were exposed to Xenom arc lamp for 72 h under standard testing conditions, washing fastness test was assessed in accordance with ISO 105-C06; 2010 (ISO, 2010). A crock meter was used to measure the both the dry and wet rubbing fastness of the dyes samples according to ISO 105-X12; 2013 by comparison with the greyscale (ISO, 2013). The sublimation fastness test was performed at 150 °C for 10 sec in accordance with ISO 105-P01 (ISO, 2014). The samples were pressed between two plates at 150 °C for 10 sec. after which the adjacent fabrics were then assessed for staining using grey scale. Fastness to sublimation was evaluated using an electronic T-10 thermotester (Japan).

Results and Discussion

5-(bromo-2-aminophenylazo-2'-chlorophenylazo-2"-nitro-4methoxybenzene (3a)

Obtained from compound **2** and 4-bromoaniline as brown crystals (62%, mp: 187-189 °C) IR (KBr); v_{max} (cm⁻¹); 3479 (br) and NH₂ 2868-3052 (ArC-Hstr), 1669(s) (ArC=C), 1393(s) (NO₂ asymmetric), 1093(s) (C-O), 866, 660(m), 548(m) (substituted benzene rings). ¹H NMR (200 MHz DMSO-d₆) $\delta_{\rm H}$ (ppm); 1.28 (s, 2H, NH₂), 3.65 (s, 3H, OCH₃), 7.52-7.68 (m, 3H, ArH), 8.82 (m, 1H, ArH). ¹³C NMR (50 MHz, DMSO-d₆) $\delta_{\rm C}$ (ppm); 165.7, 156.6, 155.3, 147.8, 144.7, 144.5, 133.4, 133.2, 132.6, 132.3, 130.7, 124.8, 122.6, 121.8, 115.9, 112.6, 110.7, 45.6.

4,6-dihydroxypyrimidineazo-2-chlorophenylazo-2"-nitro-4methoxylbenzene (3b)

Obtained from compound **2** and 4,6-dihydroxypyrimidine as brown solid, powder (59%, mp: 158-160 °C), IR (KBr) v_{max} (cm⁻¹) 3519(br) (O-H), 2868(s), 3062(m), (ArC-H), 1673(s) (NO₂, symmetric) 1496(s) 1443(s) (ArC=C), 1393 (NO₂ asymmetric stretch), 1093(s) (C-O) 861(w), 660(m), 551(m) (substituted aromatic rings). ¹H NMR (200 MHz, DMSO d₆) $\delta_{\rm H}$ (ppm); 3.67 (s, 3H, OCH₃), 4.56 (s, 1H, OH) (2), 7.25-7.36 (d, 2H, J=8.2Hz, ArH), 8.03 (d, 2H, J=8.3Hz, ArH), 8.25-8.35 (m, 1H, ArH), 8.41 (m, 1H, ArH). ¹³C NMR (50 MHz DMSO-d₆) $\delta_{\rm C}$ (ppm); 164.8, 163.9, 162.5, 161.5, 161.2, 160.8, 160.6, 155.9, 155.5, 154.3, 154.0, 148.7, 148.5, 142.7, 132.5, 131.5, 46.6.

4-hydroxy-2-aminophenylazo-2'-chlorophenylazo-2"-nitro-4-methoxybbenzene (3c)

Obtained from compound **2** and 3-aminopehnol. (5%, mp: 190 °C). IR (KBr) v_{max} (cm⁻¹) 3494(br) (OH, -NH₂), 2873(m), 2932(m), 3035(w) (ArC-H), 1671(s) (NO₂ symmetric stretch) 1496(s) 1443m (ArC=C), 1393(s) (NO₂ asymmetric stretch) 1093s (C-O), 861(w), 659(m), 548(m) (substituted aromatic rings). ¹H NMR (200 MHz DMSO-d₆) δ_{H} (ppm): 1.32 (s, 2H, NH₂), 3.66 (s, 3H, OCH₃), 7.29-7.50 (m, 3H, ArH), 7.61-7.65 (m, 2H, ArH), 8.01 (m, 1H, ArH), 8.32 (m, 1H, ArH). ¹³C NMR (50 MHz DMSO-d₆) δ_{C} (ppm); 163.5, 163.2, 161.8, 161.6, 160.5, 156.8, 155.9, 155.7, 154.7, 154.4, 153.4, 140.3, 140.1, 113, 6, 111.8, 103.7, 101.8, 50.5.

2-ethoxy-4-hydroxybenzaldehydeazo-2'-chlorophenylazo-2"nitro-4-methoxybenzene (3d)

Obtained from compound 2 and 2-ethoxy-4hydroxybenzaldehyde as brown crystals (55%, mp: 118-120 °C) IR (KBr) v_{max} cm⁻¹; 3483(br) (O-H), 2868, 2932(s), 3062(w) (Arom.C-H), 2710(w) (C=O), 1670(s), (NO₂, symmetric stretch) 1496(s), 1437(s) (Arom.C=C), 1393(s) (NO2 asymmetric stretch), 1094(s) (C-O) 866(w), 660(m), 548(m) (substituted aromatic rings). ¹H NMR (200 MHz DMSO-d₆) $\delta_{\rm H}$ (ppm): 1.24 (t, 3H, -OCH, CH₃), 3.70 (s, 3H, OCH3), 4.11 (q, 2H, OCH2CH3), 4.21 (s, 2H, Arom.H), 8.20 (d, 2H, J=8.2Hz, Arom.H), 8.22-8.34 (d, 2H. J=7.82 Arom.H), 8.55 (m, 1H, Arom.H). ¹³C NMR (50 MHz, DMSO-d₆) δ_C (ppm); 148.5, 163.5, 162.4, 156.8, 155.9, 155.6, 148.7, 148.5,

144.9, 144.6, 135.6, 135.6, 135.5, 132.4, 131.8, 131.6, 125.7, 124.6, 124.5, 121.8, 45.6, 41.7, 40.6.

2-hydroxy-7-methoxynaphthylazo-2-chlorophenylazo-2nitro-4-methoxybenzene (3e)

Obtained from compound 2 and 2-hvdroxy-7methoxynaphthalene (67%, mp: 240 °C), IR (KBr) v_{max} (cm⁻ ¹); 3509(br) (O-H), 2868(m) 2931(s) 3056(w) (Arom.C-H) 1672(vs) (NO₂ symmetric stretch), 1496(s), 1443(s) (Arom.C=C), 1392(s) (NO₂ asymmetric stretch), 1093(s) (C-O), 864(m) 659(m), 543(m) (substituted aromatic ring). ¹H NMR (200 MHz DMSO-d₆) δ_H (ppm): 3.67 (s, 6H, OCH₃), 4.42 (s, 1H, OH), 7.21-7.10 (m, 5H, Arom.H), 7.33-7.41 (m, 2H, Arom.H), 8.25 (d, 2H, J=8.2Hz, Arom.H), 8.41 (m, 1H, Arom.H). ¹³C NMR (50 MHz, DMSO-d₆) δ_C (ppm); 162.9, 162.8, 156.8, 155.7, 155.4, 154.9, 153.4, 149.6, 148.7, 146.5, 145.4, 135.6, 135.3, 134.9, 134.6, 133.5, 131.5, 130.8, 130.7, 130.0, 50.2.

6-hydroxyquinolylazo-2-chlorophenylazo-2-nitro-4mmethoxybenzene (3f)

Obtained from compound **2** and 6-hydroxyquinoline as a brown solid (62%, mp: 223 °C). IR (KBr) v_{max} (cm⁻¹); 3517(br) (Arom.OH), 2868(m), 2931(s), 3062(w) (Arom.C-H), 1673(vs) NO₂, symmetric stretch), 1254(m), 1445, 1496(s) (Arom.C=C), 1391(s) (NO₂ asymmetric stretch), 1093(s) (C-O), 874(w), 659(m), 543(m) (substituted aromatic ring). ¹H NMR (200 MHz DMSO-d₆) δ_{H} (ppm); 3.67 (s, 3H, OCH₃), 4.51 (s, 1H, OH), 6.10-7.73 (m, 5H, quinolyl H), 7.40-7.45 (m, 2H, Arom.H), 8.10 (d, 2H, J=8.21Hz Arom.H), 8.32 (m, 1H, Arom.H), 8.45 (m, 1H, Arom.H). ¹³C NMR (50 MHz CDCl₃) δ_{C} (ppm); 165.3, 165.1, 164.6, 63.5, 156.6, 155.7, 154.8, 153.6, 151.9, 151.8, 148.9, 145.6, 145.3, 138.4, 137.7, 135.6, 134.9, 133.4, 131.7, 121.6, 116.3, 115.3.

8-hydroxyquinolylazo-2-chlorophenylazo-2-nitro-4methoxybenzen (3g)

Obtained from compound **2** and 8-quinoline (53%, mp: 235 °C). IR (KBr) v_{max} (cm⁻¹); 3519(br) (Arom.OH), 2868(s), 2931(s), 3064(s) (Arom.C-H), 1673(vs) (NO₂ symmetric stretch), 1496(s), 1443(s), 1255(m), (Arom.C=C), 1392(s) (NO₂ asymmetric stretch), 1092(s) (C-O), 865(w), 695(m) (substituted aromatic ring). ¹H NMR (200 MHz DMSO-d₆) $\delta_{\rm H}$ (ppm); 3.70 (s, 3H, OCH₃), 4.25 (s, 1H, OH), 6.78-7.56 (m, 5H, quinolyl H), 7.60-7.72 (m, 2H, Arom.H), 8.10-8.42 (d, 2H, J=8.0Hz, Arom.H), 8.4 (m, 1H, Arom.H). ¹³C NMR (50 MHz CDCl₃), $\delta_{\rm C}$ (ppm); 164.8, 163.5, 157.8, 154.9, 154.6, 153.7, 153.5, 151.8, 151.5, 148.6, 145.5, 143.7, 142.1, 141.2, 140.9, 133.6, 132.5, 131.6, 122.8, 120.8, 116.7, 113.8, 112.3.

Table 1: UV-Vis Absorption	n Characteristics	of	dyes	3a	to
3f measured in dimethylfori	namide				

Dye	Molecular weight	Absorption maximum nm (absorbance)	Absorption coefficient (dm ³ M ⁻¹ cm ⁻¹)
3a	C ₁₉ H ₁₄ N ₆ O ₃ ClBr	402 (0.899)	84,700
	465.5	41.3(s) (0.767)	73,700
3b	C17H12N7O5Cl	422 (0.922)	99,000
	429.5	425(s) (0.918)	98,600
3c	$C_{19}H_{15}N_6O_4Cl$	413 (0.492)	52,400
	426.5	401(s) (0.463)	49,400
3d	C22H18N5O6Cl	425 (0.433)	52,900
	483.5	405(s) (0.405)	49,000
3e	$C_{24}H_{18}N_5O_5Cl$	401 (0.716)	88,000
	491.5	488 (0.667)	82,000
3f	C22H15N6O4Cl	486 (0.511)	59,000
	462.5	488(s) (0.509)	58,900
3g	C22H15N6O4Cl	402 (0.302)	34,900
	462.5	411(S) (0.292)	33,800

As illustrated in scheme 1, disazo disperse dyes were synthesized by the traditional diazotization and coupling reactions in between 4-methoxy-2-nitroaniline as the diazo unit and 2-chloroaniline as the coupling second diazo unit. The crude dyes were purified by recrystallization from acetonitrile until a constant molar coefficient and TLC purity were obtained in good yield (51-67%). The melting points of the synthesized dyes were determined and results show variable values depending on the various factors which affects melting point of compounds, including the nature of the substituents. The chemical structures of the dyes were verified by ¹H NMR, ¹³C NMR spectroscopy and FTIR spectral data.

The absorption spectral results of the dyes are presented in Table 1. The dye derivatives exhibited different absorption maximum bands. The position of the visible absorption bands depends on the character of the substituents. The presence of electron withdrawing halogens (Cl, Br) in the structures of the dyes in addition to the nitrogen atoms in the ring of the final coupling components of dye 3b caused a bathochromic shift in its Λ_{max} compared to other dyes. As has been shown in Table 1, the dyes exhibited two to four separated bands with some bands appearing in the ultraviolet region and others in the visible ranges. But only two bands in the visible range were shown on Table 1. The absorption spectra of the dyes showed one absorption maximum at the visible range and one shoulder. It is evident that the position of the substituents such as OH results in a red shift within dye 3f compared with dye 3g due to higher conjugation. There were also significant changes in the molar coefficient values between compounds 3f (δ = 59,000) and 3g (ϵ = 34,900). This indicates that dye 3f showed higher intensity compared with dye 3g.

Fastness properties of dyes 3a to 3g on dyed fabrics

Fastness evaluations were carried out for all the dyed samples. Results are presented in Tables 2 and 3.

 Table 2: Light, Sublimation and Rubbing Fastness

 Properties of Dyes Fabrics (1% owf)

	Fabrics			Rubbing		
Dye		Light fastness	Sublimation	Dry	Wet	
3a	Polyester	7	4/5	4/5	4	
	Nylon	6	4	4	4	
3b	Polyester	6/7	4/5	4	4	
	Nylon	6	4	4/5	4/5	
3c	Polyester	7	4/5	4/5	4	
	Nylon	6	4/5	4/5	4/5	
3d	Polyester	6/7	4//5	4	3/4	
	Nylon	6/7	4	3/4	3/4	
3e	Polyester	7	4/5	4	4	
	Nylon	6	4	4	4	
3f	Polyester	7	4/5	4/5	4	
	Nylon	6	4/5	4/5	4/5	
3g	Polyester	7	4	4/5	4	
2	Nylon	6/7	4/5	4/5	4/5	

Blue wool scale 1-8 for light fastness (where 1-2 = poor, 3 = fair, 4 = moderate, 5 = good, 6-7 = very good, 8 = excellent)

The result presented in Table 2 for light fastness shades, show that the fastness ratings are grades 6 to 6/7 for nylon fabrics and grades 6/7 to 7 for polyester fabrics which implies good to very good for nylon fabric and very good for polyester fabrics. It is therefore evident that the light fastness ratings of the dyes on polyester fabrics were technically higher compared with those of nylon fabrics. This outstanding light fastness performance of the dyes on polyesters may be attributed to the electron-withdrawing –NO₂, and –Cl groups around the hydrazo group in the two diazo components as well as concentration of the dye molecules in the dyed fabrics. All the compounds showed very good to excellent (grade 4 to 4/5) sublimation and rub fastness results on both textile fabrics except dye 3d which showed good (grade 3/4) results.

Dye	Fabric	Colour change	Acetate	Wool	Cotton	Aryclic	Nylon	Shade on dyed fabrics
3a	Polyester	5	5	5	5	4/5	5	Orange-brown
	Nylon	4/5	5	5	5	4/5	5	Orange-brown
3b	Polyester	5	5	5	5	4/5	5	Orange-brown
	Nylon	4/5	5	5	5	4/5	5	Orange-brown
3c	Polyester	5	5	3	5	4/5	5	Orange-brown
	Nylon	5	5	4/5	5	5	5	Orange-brown
3d	Polyester	5	5	5	5	5	5	Brown
	Nylon	5	5	5	5	4/5	5	Orange-brown
3e	Polyester	4/5	5	5	5	5	5	Brown
	Nylon	4/5	5	5	5	4/5	5	Brown
3f	Polyester	5	5	5	5	4/5	5	Scarlet
	Nylon	4/5	5	5	5	5	5	Yellow
3g	Polyester	5	5	5	5	4/5	5	Scarlet
	Nylon	5	5	5	5	5	5	Yellow

Table 3: Wash fastness properties of dyed fabrics (1% owf)

Grey scale 1-5 (where 1 = poor, 2 = fair, 3 = good, 4 = very good, 5 = excellent)

Table 3 shows the wash fastness results of the dyed samples. Results show that the tested samples yielded acceptable wash fastness ratings of 4/5 to 5 for polyester and nylon fabrics which indicates very good to excellent performance. The wet fastness results in terms of staining of adjacent fabrics were observed to be excellent (grades 4/5 to 5). This shows that all the dyes may exist in the hydrazine tautomeric form in the solution phase. This assertion has been confirmed by the results of the change in colour value of the dyed samples

(Table 3). From Table 1, it is evident that dye 3b exhibited the highest absorption coefficient (ϵ =99,000) followed by dye 3e (ϵ =88,000).

Conclusion

Seven disperse azo dyes based on 4-methoxy-2-nitroaniline and 2-chloroaniline systems were successfully synthesized and characterized using ¹H NMR, ¹³C NMR and FTIR spectroscopy. The spectroscopic properties of the synthesized compounds clearly confirmed their structures. The results of the light fastness tests clearly indicate that the presence of electron-withdrawing groups such as $-NO_2$ and -Cl in the dye structures leads to higher light fastness ratings on the polyester fabrics compared with those of nylon 66 fabrics. Furthermore, the high concentration of the dye molecules in the dyed polyester fabrics, due to the dyeing method used could also be responsible for the higher light fastness performance. The wet, sublimation and rubbing fastness of the dyes were also observed to be satisfactory.

Conflict of Interest

Author declares that there are no conflicts of interest

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