

Synthesis and Application to Polyester and Nylon 6 Fabrics of Hetaryl Bis-Azo Disperse Dyes Based on 6-Amino-2,4-Dihydroxypyrimidine and 4-Methoxy-2-Nitroaniline Moieties

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Received 1 November 2018, accepted in final revised form 6 March 2019

Abstract

Six new bis-azo disperse dyes were synthesized by linking various aryl amino and hydroxyl derivatives to 4-methoxy-2-nitroaniline and 6-amino-2,4-dihydroxypyrimidine moieties through diazo coupling reactions. The structures of the bis-azo dyes were identified by Fourier transform infrared, proton and carbon 13 nuclear magnetic resonance data. The prepared dyestuffs were applied onto polyester and nylon 6 fabrics and subsequently their fastness properties in terms of light, washing, sublimation and rubbing were determined. Compared with the light fastness of polyester fabrics, the light fastness of the dyed nylon 6 fabrics were slightly lower. The technical performance of the dyes on the two textile substrates used for the study, demonstrated their useful applications in the dyeing of synthetic fibres.

Keywords: Bis-azo dyes; Synthetic fibres; Heterocyclic compounds; Fastness properties; Characterization.

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doi: <http://dx.doi.org/10.3329/jsr.v11i2.38734> J. Sci. Res. **11** (2), 215-224 (2019)

1. Introduction

Synthetic colorants are widely utilized in industrial textile dyeing processes [1]. Azo compounds accounts for over 65% of synthetic dye production in the world today. The major groups of azo dyes includes; reactive, disperse and acid dyes. The success achieved by these groups of dyes is due to the following factors: the simplicity of production, the numerous possibilities available for structural changes to suit the diverse applications and the high molar extinction coefficient exhibited [2-4].

The development in azo disperse dyes prepared from heterocyclic diazo components and coupling components have been summarized by Towns [5]. Disperse dyes containing the azo group and heterocyclic compounds have attracted much attention and this is

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expected to increase in future due to their healthy contributions. For instance, many heterocycles possess anti-microbial activities and they do not persist for a long time before being degraded in the environment. Thus, they do not pose ecological problems. In addition, the deep bright colors produced by the heterocyclic compounds could be explained using resonance theory, in which case, the ring system often has resonance stabilization energy to lose excitation compared with those based on the benzene rings [6-9].

The first starting material for this work, 4-methoxy-2-nitroaniline is a versatile intermediate in the synthesis of dyes, pigments and other chemical compounds [10]. The second diazo/coupling components, 6-amino-2,4-dihydropyrimidine, contain the pyrimidine nucleus whose derivatives have been found to be widely applied in the field of pharmacy and medicine. They have also been found useful in the detection of cancer, and as a chemotherapeutic agent. Furthermore, they are important precursors in the synthesis of many fused heterocyclic compounds of biological importance [11].

A combination of these moieties is yet to be utilized in the synthesis of bis-azo dispersed dyes. Hence the aim of this study was to synthesize and apply the bis-azo disperse dyes. Finally, the coloration properties of the dyes on polyethylene terephthalate (polyester or PET) and nylon 6 fabrics were determined.

2. Materials and Methods

The solvents and reagents used in this study were of spectroscopic and pure grades respectively. 4-methoxy-2-nitroaniline and 6-amino-4,6-dihydropyrimidine were purchased from Sigma-Aldrich (Germany). All other chemicals were used as received, and the dyes were characterized using ^1H and ^{13}C nuclear magnetic resonance spectroscopic analysis on a 200 BB series spectrometer. DMSO- d_6 and CDCl_3 were used as NMR solvents. All chemical shifts were expressed in part per million (ppm) using tetramethyl silane (TMS) as the internal reference standard. All reactions were monitored by thin-layer chromatography (TLC, R_f values). Absorption spectra were measured using UV-2450 UV/vis spectrometer (Shimadzu Corp. Japan). The infrared (IR) spectra of the dyes were recorded on a 8400S FTIR spectrometer.

2.1. Synthesis of intermediate dye 2

Sodium nitrite (NaNO_2 , 0.017 mol) solution in distilled water (10 mL) was added to a mixture of 4-methoxy-2-nitroaniline (3.0 g, 0.017 mol) and concentrated sulphuric acid (98%, 5 mL). the mixture was stirred for 1h and a test for the presence of nitrous acid (HNO_2) was performed with starch iodide paper at 0–5 °C. The excess of HNO_2 was then eliminated by adding a few drops of sulphamic acid to obtain the active diazonium salt solution. Then a solution of 6-amino-4,6-dihydropyrimidine (2.27 g, 0.017 mol) in sodium hydroxide (1.0 g, 20 mL) was slowly added to the above solution at 0–5 °C. The mixture was stirred for 2 h, after which the crude azo product was isolated by filtration and washed with distilled water. The product was recrystallized from ethanol to afford compound 2 in 95% yield.

2.2. Synthesis of bis-azo dyes 3a to 3f.

The intermediate compound 2 (2.0 g, 0.007 mol) was diazotized in concentrated sulphuric acid (5 mL) in the presence of water (3.0 mL) with a solution of sodium nitrite (1.0 g, 0.006 mol) dissolved in 8 mL of water at 0–5 °C stirred for 1 h and a positive test for nitrous acid on starch iodide paper was obtained. The residual HNO₂ was removed by adding a few drops of sulphamic acid to obtain the active diazonium salt solution. The coupling reactions were then performed by adding the diazonium solution to the solution of 6-hydroxyquinoline (0.95 g, 0.007 mol) in sodium hydroxide (1.0 g, 20 mL) and stirred for 2 h at room temperature. The precipitated crude dye was collected through filtration and washed with water three times and dried. The pure dye was recrystallized from ethanol to give compound 3a. in 58% yield. Dyes 3b to 3f were similarly synthesized using each of the following coupling components; 8-hydroxyquinoline (0.007 mol), 2-chloroaniline (2 mL), 4,6-dihydropyrimidine (0.0065 mol), 7-methoxy-2-naphthol (0.01 mol), 3-ethoxy-2-hydroxybenzaldehyde (0.01 mol) respectively (Scheme 1).

2.3. Dyeing and reduction-clearing processes

2.3.1. Nylon 6 fabric

Nylon 6 fabrics were wetted and immersed into the dyebath containing 1% of owf (liquor ratio 1:20) at 45 °C using an Ahiba Texomat dyeing machine. The pH of dyebath was adjusted to 4 using 2% acetic acid. The dyeing procedure was carried out according to Fig. 1. Initially the dyeing commenced at 45 °C and then increased to 100 °C at 2 °C min⁻¹. After maintaining the temperature for 30 min the dyed nylon fabrics were removed and rinsed with tap water. The rinsed fabrics were reduction-cleared with iodet T (2 gL⁻¹) in a bath containing water (liquor ratio 1:2) at 45 °C for 15 min. This was followed by thorough rinsing of the treated fabrics with water and air dried (Figure 1).

2.3.2. Polyester fabric

The dye-bath was prepared by dissolving 1.0 g of each dye in 5 mL of dimethyl formamide (DMF) followed by adding of a dispersing agent (Lignosulphonate liquor) and the final volume of disperse dye solution was made up to 100 mL. Acetic acid (2%) was used to bring the pH of the disperse dye solution to 4.5. Dyeing of the polyester fabric (10 × 5 cm) was performed in a high-temperature, high-pressure dyeing machine Galvanin (St Marin Q, Italy). Dyeing initially commenced at 60 °C and the temperature gradient was increased at 3 °C min⁻¹ until it reached 130 °C.

This temperature was kept constant for 60 min. after which the dyeing vessel was allowed to cool to 50 °C and the dyed samples were collected from the dye bath and rinsed with cold water. A solution containing sodium hydrosulphite (2.0 gL⁻¹) and sodium hydroxide (2.0 gL⁻¹) was used to treat the dyed fabrics for 30 min at 30 °C to remove dye molecules on the surface of the dyed fabrics followed by further treatment with betaine amphoteric

surfactant. The reduction-cleared dyed fabrics were rinsed with distilled water and air dried (Figure 2).

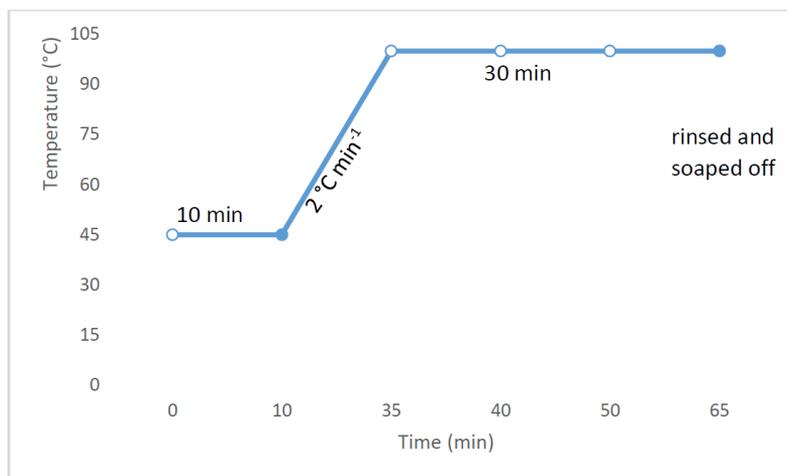


Fig. 1. Outline of the dyeing procedure for dye 3a – 3f on nylon 6 fabric.

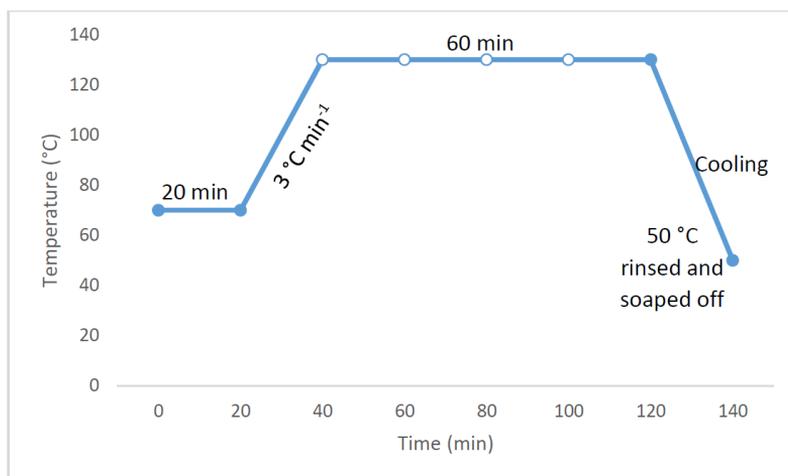
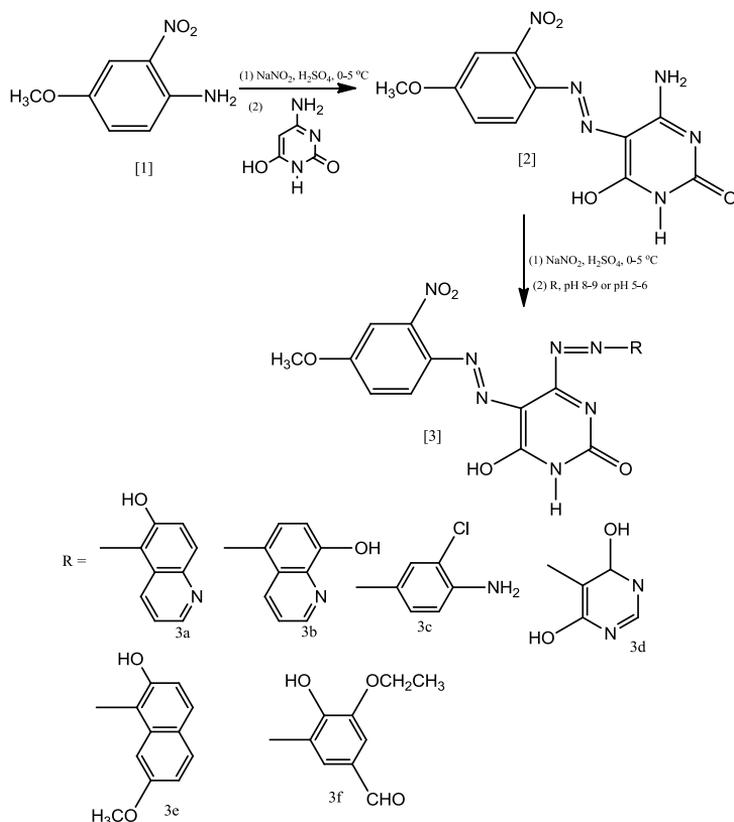


Fig. 2. Outline of the dyeing procedure for dye 3a – 3f on polyester fabric.

2.4. Colour fastness measurement

Colour fastness of dyed fabrics was tested according to the International Standardization Organization (ISO) methods, including fastness to washing ISO 105-CO6 2010 [12], light ISO 105-B02 [13], rubbing (ISO 105-X12: 2016) [14]. In the case of rubbing, the test was carried out using a Crockmeter (Taber@crockmeter, USA) during which the dyed fabric sample was subjected to rubbing with a standard cotton fabric in order to assess the extent

of colour transfer. Two tests were carried out, one with dry cotton fabric, the other with wet cotton fabric. Sublimation test was evaluated in accordance with ISO 105-PO1; 1993 [15]. The dyed fabric samples were subjected to sublimation treatment in a sublimation testing machine at 177 °C for 30 sec. The tested fabrics were then related against standard grey scale 1–5 for alteration of color.



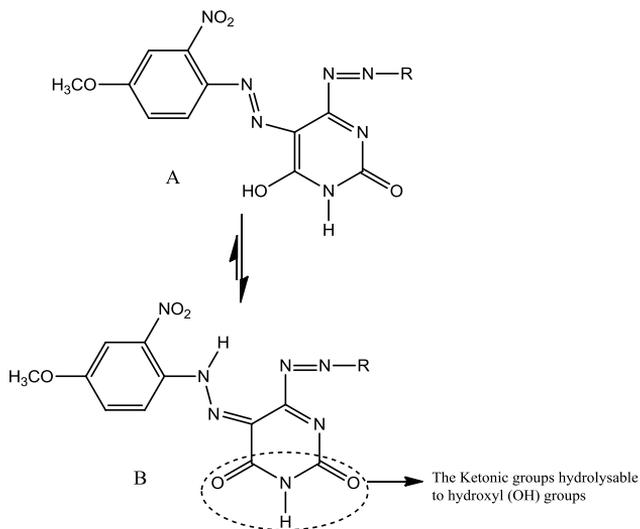
Scheme 1. Synthesis procedure of the studied bis-azo dyes 3a to 3f.

3. Results and Discussion

3.1. Synthesis and spectroscopic studies

Scheme 1 outlines the synthesis of the bis-azo disperse dyes. 4-methoxy-2-nitroaniline was diazotized in an acidic medium and the active diazonium salt solution obtained was coupled with 6-amino-2,4-dihydropyrimidin-5(1H)-one to give the intermediate compound 2 [16]. This was made to react with HONO that was generated in situ in the presence of a mineral acid in a diazotization reaction, at very low temperature. The resultant product was reacted with various coupling compound in coupling reactions to afford the bis-azo

disperse dyes 3a to 3f [17]. The dye compounds were purified by recrystallization from ethanol or methanol until a constant molar extinction coefficient was obtained. TLC was used to confirm the purity of the synthesized compounds. The bis-azo disperse dyes 3a to 3f were synthesized in 52 to 88% yields. The structures of the prepared azo compounds were confirmed using proton (^1H) and ^{13}C NMR and IR spectra.



Scheme 2. (A) = Equilibrium between the azo form and the hydrazone form (B) of the dyes.

Table 1 shows the electronic properties of the dyes. The studied dyes showed absorption bands at 403 – 477 nm range, which is associated with $n - \pi^*$ transitions. The multiple absorption bands observed is as a result of the presence of several chromophoric systems present in the dye structure. Dye 3c and 3e gave a broad shoulder around 449–477 nm which might be due to the Internal Charge Transfer (ICT) between the electron-donating $-\text{OCH}_3$ and electron-withdrawing $-\text{NO}_2$. It is also evident that the presence of carbonyl or formyl group in dye 3f causes a blue shift in the absorption band of 3f in contrast to 3a and 3d. Dye 3d also exhibited a blue shift because it contains electron-donating and electron-withdrawing groups; $-\text{OH}$ and $-\text{NO}_2$ both in the ortho position to the hydrazo group.

Table 1. Spectroscopic data of the studied dyes in dimethylformamide (DMF).

Compounds	Molecular formula	λ_{max} (nm)	$\epsilon \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$
3a	$\text{C}_{20}\text{H}_{14}\text{N}_8\text{O}_6$	404, 445, 454	71,850
3b	$\text{C}_{20}\text{H}_{14}\text{N}_8\text{O}_6$	416, 442, 446	54,630
3c	$\text{C}_{17}\text{H}_{13}\text{N}_8\text{O}_5\text{Cl}$	403, 449, 477	46,110
3d	$\text{C}_{15}\text{H}_{11}\text{N}_9\text{O}_7$	407, 466, 468	81,110
3e	$\text{C}_{21}\text{H}_{17}\text{N}_7\text{O}_7$	434, 456, 471	80,670
3f	$\text{C}_{21}\text{H}_{17}\text{N}_7\text{O}_8$	408, 436, 468	90,790

3.1.1. Dye 3a

Yield: 52%; mp: 190 °C; FTIR $\bar{\nu}_{\max}$ (KBr) cm^{-1} : 3487 (O-H_{str}), 2952 – 3167 (C-H_{str}), 2352 (quinolyl) 1671 (C=O_{str}), 1496 – 1397 (NO₂ symmetric and asymmetric stretch), 1440 (C=C_{str}), 1254 (C-O-C_{str}), 1097 (C-O_{str}), 864 (disubstituted aromatic ring). ¹H NMR (DMSO-d₆ 200 MHz) δ_{H} (ppm): 3.25 (s, 3H, OCH₃), 4.40 (s, 1H, OH x 2), 6.82–7.57 (m, 5H, quinolyl H), 7.62–8.20 (m, 3H, ArH), 9.83 (s, 1H, NH). ¹³C NMR (DMSO-d₆ 50 MHz) δ_{C} : 175.6, 168.7, 165.5, 156.9, 156.7, 155.6, 154.2, 153.8, 153.4, 145.6, 145.4, 145.2, 133.8, 133.6, 131.5, 131.3, 130.9, 130.7, 126.7, 126.5, 30.8.

3.1.2. Dye 3b

Yield: 59%; mp: 290 °C; FTIR $\bar{\nu}_{\max}$ (KBr) cm^{-1} : 3488 (O-H_{str}), 2932 – 3098 (C-H_{str}), 2352 (quinolyl), 1670 (C=O_{str}), 1496, 1397 (NO₂ symmetric and asymmetric stretch), 1440 (C=C_{str}), 1254 (C-O-C_{str}), 1094 (C-O_{str}), 864 (disubstituted aromatic ring). ¹H NMR (DMSO-d₆ 200 MHz) δ_{H} (ppm): 3.25 (s, 3H, OCH₃), 4.41 (s, 1H, OH x 2), 6.82–7.57 (m, 5H quinolyl-H), 7.61–8.10 (m, 3H, Ar-H), 9.82 (s, 1H, NH). ¹³C NMR (DMSO-d₆ 50 MHz) δ_{C} (ppm): 175.2, 165.9, 165.7, 165.2, 156.8, 156.5, 155.6, 155.5, 154.7, 154.3, 148.6, 148.3, 145.9, 145.8, 145.5, 133.6, 133.4, 131.7, 131.6, 125.5, 30.9.

3.1.3. Dye 3c

Yield: 49%; mp: 250 °C; FTIR $\bar{\nu}_{\max}$ (KBr) cm^{-1} : 3495 (O-H_{str}, NH_{str}), 2932 – 3098 (C-H_{str}), 2352 (quinolyl ring), 1671 (C=O_{str}), 1496 – 1393 (NO₂, symmetric and asymmetric stretch), 1440 (C=C_{str}), 1255 (C-O-C_{str}) 1093 (C-O_{str}), 864 (disubstituted aromatic ring), 659 (C-Cl_{str}). ¹H NMR (DMSO-d₆, 200 MHz) δ_{H} (ppm): 2.10 (s, 2H, NH₂), 3.25 (s, 3H, OCH₃), 4.30 (s, 1H), 7.41–7.50 (m, 3H, ArH), 7.61–8.10 (m, 3H, ArH), 9.82 (s, 1H, NH). ¹³C NMR (CDCl₃, 50 MHz) δ_{C} (ppm): 174.8, 172.5, 165.5, 165.1, 155.8, 155.4, 147.6, 147.3, 145.5, 142.8, 142.6, 141.7, 133.6, 133.4, 131.8, 131.5, 30.7.

3.1.4. Dye 3d

Yield: 78%; mp: 220 °C; FTIR $\bar{\nu}_{\max}$ (KBr) cm^{-1} : 3501 (O-H_{str}), 2931–3109 (C-H_{str}), 2351 (quinolyl ring), 1672 (C=O_{str}), 1496, 1394 (NO₂, symmetric and asymmetric stretch), 1443 (C=C_{str}), 1255 (C-O-C_{str}), 1093 (C-O_{str}), 869 (disubstituted aromatic ring). ¹H NMR (DMSO-d₆, 200 MHz), δ_{H} (ppm): 3.25 (s, 3H, OCH₃), 4.34 (s, 1H, OH x 3), 7.61–8.10 (m, 3H, ArH), 9.82 (s, 1H, NH). ¹³C NMR (DMSO-d₆, 50 MHz) δ_{C} (ppm): 167.3, 166.8, 166.4, 156.5, 156.2, 155.2, 154.8, 153.7, 148.3, 148.0, 133.9, 132.7, 125.4, 34.8, 30.5.

3.1.5. Dye 3e

Yield: 66%; mp: 150 °C; FTIR $\bar{\nu}_{\max}$ (KBr) cm^{-1} : 3509 (O-H_{str}), 2868–3062 (C-H_{str} aromatic), 2352 (quinolyl ring), 1672 (C=O_{str}), 1496 and 1392 (NO₂ symmetric and asymmetric stretch), 1443 (C=C_{str}), 1254 (C-O-C_{str}), 1093 (C-O_{str}), 863 (disubstituted aromatic ring), 659 (C-H_{bending}). ¹H NMR (DMSO-d₆ 200 MHz), δ_{H} (ppm): 3.51 (s, 3H,

OCH₃ x 2), 4.32 (s, 1H, OH x 2), 6.87 – 7.22 (m, 5H, maphthylH), 7.62 – 8.12 (m, 3H, ArH), 9.83 (s, 1H, NH). ¹³C NMR (CDCl₃, 50 MHz) δ_C (ppm): 174.6, 165.3, 164.0, 154.9, 154.7, 150.4, 146.8, 146.6, 145.7, 145.5, 143.8, 143.6, 134.8, 134.6, 125.2, 122.5, 122.2, 121.9, 120.5, 30.7.

3.1.6. Dye 3f

Yield: 74%; mp: 196 °C; FTIR $\bar{\nu}_{\max}$ (KBr) cm⁻¹: 3504 (O-H_{str}), 2873 – 3063 (C-H_{str}), 2352 (quinolyl ring), 1672 (C=O_{str}), 1496, 1392 (NO₂ symmetric and asymmetric stretch), 1445 (C=C_{str}), 1254 (C-O-C_{str}), 1093 (C-O_{str}), 864 (disubstituted aromatic ring), 659 (C-H_{bending}). ¹H NMR (DMSO-d₆, 200 MHz) δ_H (ppm): 2.38 (t, 3H, CH₃), 3.52 (s, 3H, OCH₃), 4.34 (s, 1H, OH x 2), 5.21 (q, 2H, CH₂), 7.34–7.51 (m, 2H, ArH), 7.61–8.20 (m, 3H, ArH), 9.82 (s, 1H, NH), 12.51 (s, 1H, CHO). ¹³C NMR (DMSO-d₆, 50 MHz), δ_C (ppm): 175.7, 164.4, 163.6, 155.1, 154.5, 148.4, 140.5, 140.1, 130.8, 128.6, 128.4, 120.8, 118.9, 118.7, 117.5, 110.7, 35.6, 31.5

Table 2. Washing and rubbing fastness properties of dyed fabrics (1% owf).

Dyes	Fabrics	Wash fastness			Rubbing		Shade of colour on fabrics
		Alt	SW	SC	Dry	Wet	
3a	Nylon	4/5	5	5	4/5	4/5	Green
	Polyester	5	5	5	4/5	4/5	Orange-green
3b	Nylon	5	5	5	4	4	Orange
	Polyester	4/5	5	5	3/4	4	Orange
3c	Nylon	5	5	5	3/4	4	Orange
	Polyester	5	5	5	4	4/5	Orange
3d	Nylon	5	5	5	3/4	4/5	Orange-brown
	Polyester	5	5	5	3/4	4/5	Orange-brown
3e	Nylon	5	5	5	4	4/5	Green
	Polyester	5	5	5	4	4/5	Orange-brown
3f	Nylon	5	5	5	4/5	4	Green-brown
	Polyester	4/5	5	4/5	4/5	4	Orange brown

SW = Staining on wool SC = Staining on cotton, Alt = Alteration

Table 3. Light and sublimation fastness properties of dyes (1% owf).

Dye	Fabrics	Light fastness	Sublimation
3a	Nylon	5	4
	Polyester	5/6	4
3b	Nylon	5	4/5
	Polyester	6/7	4/5
3c	Nylon	5/6	5
	Polyester	6/7	5
3d	Nylon	5/6	4
	Polyester	5/6	4
3e	Nylon	6	4/5
	Polyester	6/7	4
3f	Nylon	5	4
	Polyester	5/6	4

3.2. Assessment of fastness properties

After dyeing with dyes 3a to 3f, the color fastness values of the dyed textile substrates were measured according to standard methods. The results are listed in Tables 1 and 2. The results of the wash fastness gave ratings of grade 4/5 to 5 for the two textile substrates used for the study with little variations especially for dyes 3a, 3b and 3f. The variations may be due to the nature of the fibre and that of the substituent groups. The wet fastness in terms of the staining of the adjacent fabrics gave excellent rating of grade 5, which implies no staining. This can be explained from the point of view of the large molecular size of the dye structures. The results of fastness of the dyes on the two textile substrates also show that wet rubbing performance gave ratings of grade 4 to 4/5 whereas the dry rubbing gave ratings of 3/4 to 4/5. This indicates that the wet rubbing performances are better when compared with the dry rubbing grades.

The results obtained show that the fastness to light could be quite satisfactory (5/6 and 6), such results implies that there is relative stability of the dyes under the influence of light. However, the light fastness is significantly dependent on the nature of the substituents which tend to change the electron density around the hydrazo (-N=N-) group [18]. The high light fastness of the dyes on PET and nylon fabrics could be due to electron donating and the electron-withdrawing groups of the diazo components especially in the vicinity of the hydrazo group respectively. This agrees with the view that azo dyes containing electron-withdrawing groups such as -NO₂ ortho to the hydrazo (-N=N-) group, tend to significantly decrease the electron density, and has have been reported to enhance the photostability [19] of dyed polyester fabric. The slightly better photostability of the dyes on polyester fabric [20] could be as a result of the compact crystalline nature of the fibre compared with that of nylon fabrics. The sublimation fastness results gave ratings of 4 to 4/5, which indicates acceptable performance. Generally, the synthesized dyes exhibited satisfactory fastness to light on both polyester and nylon fabrics (grade 5 to 6/7) according to the international geometric scale.

4. Conclusion

The synthesis and utilization of a series of bis-azo disperse dyes 3a to 3f which are based on 4-methoxy-2-nitroaniline and 6-amino-2,4-dihydroxypyrimidine moieties have been described. The spectroscopic data of the prepared dyes have provided enough evidence to confirm their suggested structures. Finally, the synthesized bis-azo disperse dye compounds were used to dye PET and nylon fabrics and their fastness properties in terms of sublimation, washing, light and crocking (rubbing) were evaluated. All these properties were observed to be acceptable except the dry rubbing fastness which showed moderate ratings. It may therefore be concluded that the bis-azo disperse dyes are potential disperse dyestuffs given the all-round properties observed on polyester and nylon 6 fabrics, especially with regard to wet fastness.

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