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**Abstract:** Six new azo disperse dyes were prepared from 4-phenoxyaniline through diazo coupling reactions. The structures of the azo dyes were identified by Fourier transform infrared, proton and carbon-13 nuclear magnetic resonance. All the compounds were tested for their fastness properties on nylon and polyester fabrics using pressure dyeing method. Results showed that the dyes gave brown colours on the two fabrics. The light fastness on nylon was 5 and 6 better than that of polyester. The sublimation fastness were between 3 and 4. The results of the fastness properties of the dyes indicate that they could be promising dyestuffs in the area of heat transfer printing of modern fibers.

**Keywords:** 4-phenoxyaniline, characterization, coupling reactions, fastness, pressure dyeing

### Introduction

Disperse dyes are coloured organic compounds which are characterized by their low water solubility, low molecular weight (especially monoazo disperse types). They are mainly used for dyeing synthetic hydrophobic fibres such as cellulose acetate, acrylics, polyamides and polyesters (Youssef *et al.*, 2005; Koh *et al.*, 2004; Lee *et al.*, 2005). Most disperse dyes contain the azo group as the chromophore. There are, however, others that contain polar groups such as -NH<sub>2</sub>, and -OH which confer some measure of solubility to them. Some other disperse dyes may also contain reactive groups such as sulphonylazide, triazine, bromacrylic acid and vinyl sulphone (Koh *et al.*, 2004). Of all these groups, only the sulphonyl and vinyl sulphone containing ones, have been found best for dyeing textile fibres that contain amino groups such as wool.

The 4-phenoxyaniline, which is the diazo component in this study has been used in the synthesis of some Schiff bases that have been tested for their ability to disrupt the growth and development of animal and plant parasites (Binder *et al.*, 1992).

To the author's knowledge, there has been no report yet which described the use of 4-phenoxyaniline in the synthesis of azo disperse dyes for the purpose of textile coloration. The present study, therefore, investigates the preparation of monoazo disperse dyes from this intermediate and their application to polyamide fibres. The dyes were also applied to polyester fibres for the purpose of comparison. Thus, the light fastness, wash fastness and rubbing fastness values of the dyeings on polyamide fibres were compared with those of polyester fibres. Disperse dyes, just like any other dye type; do not attain hundred percent fixation and textile substrates, especially during exhaustion dyeing. To this end, a lot of dye molecules are lost or rather remain in the spent dye baths which are subsequently disposed off as dye effluents. The pressure method of dyeing was therefore used in applying the dyes to the polyamide (nylon 6) and polyester fabrics. In order to increase the concentration of the dyes in the polymer matrix

### Experimental

#### General

The proton nuclear magnetic resonance (<sup>1</sup>H NMR) and carbon-13 nuclear magnetic resonance (<sup>13</sup>C NMR) spectra were obtained at room temperature on a mercury 200BB series spectrometer at 200 MHz and 50 MHz respectively, in deuterated dimethyl sulphoxide (DMSO-d<sub>6</sub>), using tetramethylsilane (TMS) as an internal standard. The melting points of the synthesized dyes were determined on a Stuart-SMPP3 apparatus in a sealed capillary tube and are not

corrected. The infrared (IR) spectra (in KBr pellets) were recorded on a Shimadzu 8400 S spectrometer. Ultraviolet-Visible (UV-vis) spectra were measured on a UV 1600 Series Spectrophotometer. The purity of the compounds was monitored by thin layer of Chromatography (TLC), using Merck prepared plates (Silica gel 60 F<sub>254</sub> on aluminium). All the chemicals used in this study were standard analytical grade reagents obtained from Merck, Aldrich and Fluka, and were used as obtained.

#### Synthesis of dyes

##### Diazotization

The amine, 4-phenoxyaniline (10.8 mmol) was suspended in water (50 ml). Sulphuric acid (6 ml, 98%) was added to the suspension. The mixture was cooled and kept at 0-5 °C in an ice bath and diazotised by the addition of a solution of NaNO<sub>2</sub> (43.5 mmol) in water (10 ml) followed by stirring for 0.5 h at 0-5°C. Complete diazotization was confirmed by testing the solution with starch-iodide paper which gave a blue black colour. Excess nitrous acid formed during the process was decomposed by adding 2% urea solution.

##### Coupling reactions

##### Synthesis of 2-hydroxy-7-methoxynaphthylazo-4-phenoxybenzene (3a)

The solution of the diazonium salt 2 was slowly added to a solution of 7-methoxy-2-naphthol (10.1 mmol) in acetic acid (10 ml) at 0-5°C over 30 min. The resulting mixture was stirred at room temperature for 3 h and left overnight. The precipitate was filtered, washed with water (4 x 20 ml), dried at room temperature and purified by column chromatography to give a product yield of 77%, Mp = 159-161°C, λ<sub>max</sub> (in DMF), nm = 438. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 200 MHz), δ<sub>H</sub>(ppm): 3.45 (3H, s, ArH), 3.84 (1H, s, OArH), 6.26(2H, d, J = 8.24:Hz, ArH), 7.16 (2H, d, J=8.20 Hz, ArH), 7.65 – 7.78 (4H, m, ArH), 8.48 (1H, s, OH). <sup>13</sup>C NMR (DMSO – d<sub>6</sub>, 50 MHz) δ<sub>C</sub>(ppm): 161.5, 132.2, 124.7, 123.7, 116.0, 56.0, 41.2, 40.8, 46.3, 39.9, 39.5, 38.7. IR(KBr pellets, cm<sup>-1</sup>): 3494 (O-H<sub>str</sub>), 2931 (C-H<sub>str</sub>) 1671 (Ar C = C str), 1497(-N=N-), 1255, 1093(=C-o-C, symmetric and asymmetric str), 659 (C-H bend).

##### Synthesis of 2,6-dihydroxy-3-hexylphenylazo-4-phenoxybenzene (3b)

The diazonium salt 2, was slowly added to a solution of 4-hexylresorcinol (10.1 mmol) dissolved in 20 ml of sodium hydroxide solution (25 mmol) and stirred for 2 h. The precipitated product was obtained and purified as described in 3a to give a brown solid in 68% yield, mp = 175 -177 °C, λ<sub>max</sub> (in DMF), nm = 433. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 200 MHz), δ<sub>H</sub>(ppm): 2.45 (3H, s, CH<sub>3</sub>), 3.43 (1H, s, O-ArH), 3.87(5H, s, CH<sub>2</sub>), 6.24(2H, d, J = 8.35Hz, rH), 7.06 (2H, m, ArH), 7.16 – 7.88 (5H, m, ArH), 8.42 (1H, s, Ar-OH). <sup>13</sup>C NMR (DMSO –

$\delta_6$ , 50 MHz)  $\delta_c$ (ppm): 161.5, 145.6, 133.2, 124.8, 122.6, 115.9, 55.9, 41.2, 40.8, 40.3, 39.9, 39.5, 39.1, 38.7. IR (KBr pellets,  $\text{cm}^{-1}$ ): 3415 (ArO-H str), 2931 (C-H str), 1673 (Ar C=C str), 1493 (N=N), 1256, 1093 (=C-O-C, symmetric and asymmetric stretch) 666 (C-H bend).

#### Synthesis of 2-hydroxyquinolyl azo-4-phenoxybenzene (3c)

The diazonium solution 2 was slowly added to a solution of 6-quinolinol (0.8 mmol) in sodium hydroxide solution (25 mmol, 20 ml). The resulting mixture was stirred for 1 h and left over night. The crude product was isolated and purified as described in 3a to afford a brown solid in 75% yield, mp = 150 – 152°C,  $\lambda_{\text{max}}$  (in DMF), nm = 413.  $^1\text{H}$  NMR (DMSO- $d_6$ , 200 MHz),  $\delta_{\text{H}}$ (ppm): 2.45 (5H, s, CH<sub>3</sub>), 3.60 (1H, s, O-ArH), 6.62(2H, d, J = 8.10 Hz, ArH), 7.10 – 7.73 (4H, m, ArH), 8.10 (2H, d, J = 8.30 Hz, ArH), 8.65 (1H, s, ArH), 9.10 – 9.25 (2H, m, ArH),  $^{13}\text{C}$  NMR (DMSO -  $d_6$ , 50 MHz)  $\delta_c$ (ppm): 151.0, 145.3, 131.8, 130.8, 124.8, 124.0, 120.0, 119.3, 41.2, 40.4, 39.9, 39.8, 39.5, 39.1, 38.7. IR (KBr pellets,  $\text{cm}^{-1}$ ): 3476 (O-H str), 2932 (C-H str), 1668 (Ar C=C str), 12.55, 1095 (=C-O-C, symmetric and asymmetric stretch), 660 (C-H bend).

#### Synthesis of 2,4-dihydroxyphenyl azo-4-phenoxybenzene (3d)

Compound 3d was prepared and purified as described in 3c by using 1,3-dihydroxybenzene as the coupling material to give a brown solid in 63% yield, mp = 111 – 113 °C,  $\lambda_{\text{max}}$  (in DMF), nm = 425.  $^1\text{H}$  NMR (DMSO- $d_6$ , 200 MHz),  $\delta_{\text{H}}$ (ppm): 2.48 (5H, s, O-ArH), 6.60 (1H, m, ArH), 6.93 – 7.98 (5H, m, ArH),  $^{13}\text{C}$  NMR (DMSO -  $d_6$ , 50 MHz)  $\delta_c$ (ppm): 147.5, 147.2, 133.4, 137.1, 129.7, 128.0, 41.1, 40.7, 40.3, 39.9, 39.4, 39.0, 38.6. IR (KBr pellets,  $\text{cm}^{-1}$ ): 3515 (O-H str), 2931 (C-H str), 1675(Ar C=C str), 1493 (N=N), 1256, 1093 (=C-O-C, symmetric and asymmetric stretch) 660 (C-H bend).

#### Synthesis of 4-hydroxy-6-methoxyphenylazo-4-phenoxybenzene (3e)

Compound 3e was synthesized as explained in 3c by using 3-methoxyphenol as the coupling agent to afford a brown solid. The product yield was 87%, mp = 124 – 126°C,  $\lambda_{\text{max}}$  (in DMF), nm = 395.  $^1\text{H}$  NMR (DMSO- $d_6$ , 200 MHz),  $\delta_{\text{H}}$ (ppm): 2.47 (5H, s, OCH<sub>3</sub>), 3.60 (3H, s, OArH), 6.41(2H, d, J = 8.41 Hz, ArH), 6.66 – 6.85 (2H, s, ArH), 7.15 – 7.45 (3H, m, ArH), 10.45 (1H, s, OH),  $^{13}\text{C}$  NMR (DMSO -  $d_6$ , 50 MHz),  $\delta_c$ (ppm): 130.7, 120.0, 119.4, 41.1, 40.7, 40.3, 39., 39.5, 39.0, 38.6, 30.4. IR (in KBr pellets,  $\text{cm}^{-1}$ ): 3495 (O-H str), 2859 (C-H str), 1249, 1096 (=C-O-C, symmetric and asymmetric stretch) 659 (C-H bend).

#### Synthesis of 4-methoxy-2-nitrophenyl azo-2'-amino-4'-phenoxybenzene (3f)

4-methoxy-2-nitroaniline was dispersed in water (50 ml) and diazotized in sulphuric acid, (5 ml, 98%) by the dropwise addition of a solution of sodium nitrite (43.4 mmol). The diazonium solution obtained was added slowly to a solution of 4-phenoxyaniline in glacial acetic acid (8 ml). The resulting mixture was stirred for 3 h. The crude product was isolated and purified as described in 3a. The product yield was 85%, mp = 128 – 131°C.  $\lambda_{\text{max}}$  (in DMF), nm = 486.  $^1\text{H}$  NMR (DMSO- $d_6$ , 200 MHz),  $\delta_{\text{H}}$ (ppm): 1.27 (2H, s, NH<sub>2</sub>), 2.52 (3H, s, O-CH<sub>3</sub>), 3.98(5H, m, O-ArH), 6.89 – 7.82 (5H, m, ArH), 10.72 (1H, s, OH).  $^{13}\text{C}$  NMR (DMSO -  $d_6$ , 50 MHz)  $\delta_c$ (ppm): 147.6, 146.5, 132.5, 132.1, 131.3, 136.7, 130.1, 126.3, 41.2, 40.8, 46.3, 39.9, 39.5, 39.1, 38.7. IR (KBr pellets,  $\text{cm}^{-1}$ ): 3490 (N-H str), 2932 (C-H str), 1562, 1394 (NO<sub>2</sub>, symmetric and asymmetric stretch), 1255, 1094 (=C-O-C, symmetric and asymmetric stretch) 660 (C-H bend).

#### Dye applications

Each dye was dissolved in dimethylformamide (DMF) (5 ml) and made up to 100 ml mark with a dispersing agent (sodium ligno sulphonate) and stirred for 5 min during which the dye was turned into a fine and stable dispersion ready for use.

#### Nylon 6 fabric

The pH of the dye Liquor was adjusted to 4.5 with 2% of acetic acid. The nylon fabric was wetted and immersed into

the dyebath Liquor and the whole setup in a beaker (600 ml) was placed into a pressure dyeing vessel. The dyebath temperature was increased to 102°C and the pressure was raised to 40 kpa for 10 min. the dyebath was then allowed to cool to about 50°C and the dye fabric was removed from the bath and reduction-cleared with 10 det (2  $\text{gl}^{-1}$ ) in another bath containing water of material to liquor ratio 1:2 at 40°C for 15 min. the dyed fabric was rinsed thoroughly with water and air-dried. The effect of pressure on the Iodet nylon fabric was not investigated.

#### Polyester fabric

The polyester fabric was dyed in a high temperature, high pressure dyeing machine (Galvani-Marino VI) with liquor ratio of 1:20. 10 ml of the disperse dye was added to the dye bath and made up to 100 ml. The pH of the dye liquor was adjusted to 5.5 with acetic acid (2%). Dyeing was carried out at a rate of 3°C per minute and raised up to 135°C for 60 min. The dyebath temperature was cooled to 50 °C and the dyed fabric was removed and reduction- cleared in an aqueous solution of sodium hydroxide (2  $\text{gl}^{-1}$ ) sodium sulphite (2  $\text{gl}^{-1}$ ) at 50°C for 30 min. The dyed fabric was rinsed with water and air dried.

#### Rubbing fastness test

Fastness to rubbing was determined by using the Atlas rubbing fastness tester in accordance with ISO-105-X12 standard method (ISO, 1993). The dyed fabric was placed on the base of the crock meter and in close contact with an abrasive rubbing white cloth. The testing white cloth was slid on the dyed fabric about ten complete turns of the crank. For the wet rubbing tests, the white testing cloth was wetted with distilled water and the rest procedure was done as described for the dry rubbing test. The extent of staining of the white testing cloth was assessed by placing it side by side with the grey scale for staining.

#### Light fastness test

The reduction-cleared dyed fabrics were placed along side of a series of standard blue wool and the set up was exposed to xenon arc irradiation for 48 h following the procedure described in ISO BO2: 1994 standard protocol (ISO, 1994). The blue scale in this case is 1-8; where 1 signifies maximum fading and 8 no fading.

#### Wash fastness test

The wash fastness test was carried out by suing the ISO 105 – CO6 C25: 1994 standard method (ISO, 1994). The dyed samples were washed for 30 min at 60°C, rinsed with water and air-dried. The samples were then compared with grey scale for staining (1-5) where 1 indicates maximum fading and 5 no fading.

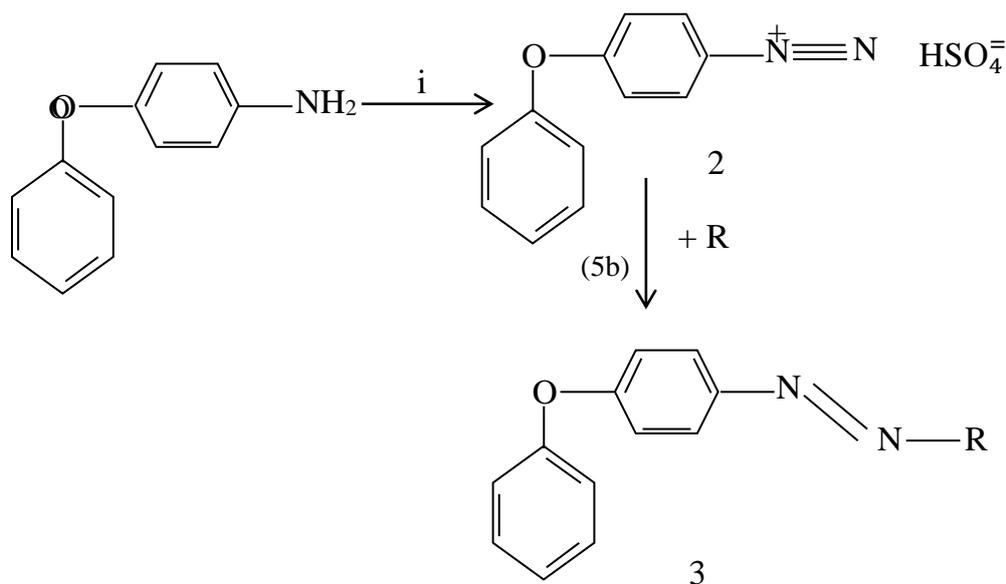
#### Sublimation fastness test

Colour fastness to heat was tested according to ISO 105 PO1: 1993 standard method. The dyed samples were paired and pressed between two plates for 30s at 102°C. The changes in colour of the adjacent samples were assessed for staining using the grey scale.

## Results and Discussion

### Synthesis

The synthesis route adopted to obtain the target compounds 3a to 3f are outlined in Scheme 1. The starting compound, 4-phenoxyaniline was subjected to diazotization and coupling reactions with some coupling agents according to published methods (Metwally *et al.*, 2013; Zhang and Hou, 2015; Salem *et al.*, 2015; El-Kolly *et al.*, 1998; Carey, 2003). The melting points of the monoazo compounds were found to be relatively moderate especially for compound 3d which has the lowest. The moderate melting points of the compounds could be ascribed to the ether group which formed the nucleus of the dye structure, the other factors such as the substituents on the coupling components not withstanding (Otutu *et al.*, 2011).



Scheme 1: The synthesis procedure of dye 3a – 3f

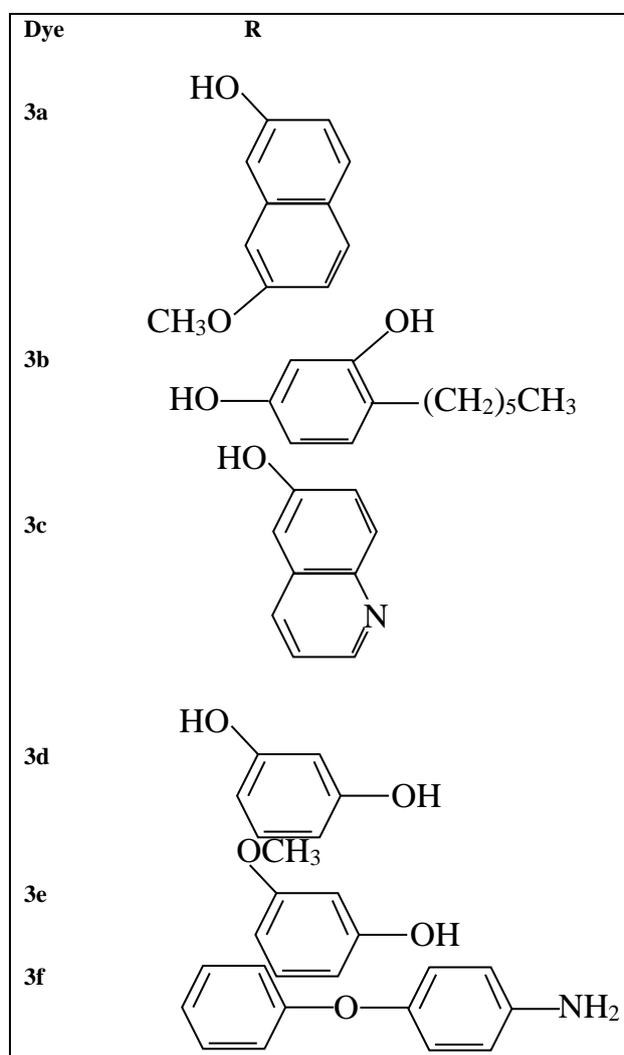


Fig. 1: Structures of dye 3a to 3f

**Physical and spectroscopic properties**

The physical and spectroscopic data of the purified monoazo disperse dyes were presented in the experimental section (Figs. 2 – 6). The results showed that  $\lambda_{\max}$  varied in the range 395 – 486 nm with dye 3f exhibiting the highest  $\lambda_{\max}$  and 3e the lowest in dimethylformamide. This indicated that all the monoazo compounds absorbed in the visible region. The results of the proton nuclear magnetic resonance and those of carbon-13 nuclear magnetic resonance actually gave values to confirm the structures of the monoazo compounds. This behaviour of 3f showed that it has the higher extended  $\pi$  conjugated system compared with others.

**Fastness properties****Fastness to washing**

The dyed nylon and polyester fabrics have good (grade 3) to very good (grade 4-5) fastness to washing according to international grey scale and this behaviour may be attributed to inadequate diffusion of dye molecules into the fabrics (Table 1).

**Table 1: Wash fastness and heat fastness properties of fabrics dyed with synthesised dyes 3a-f**

Dye	Fabric	Washing				
		CH	SN	SC	SP	heat (sublimation)
3a	Nylon	3	5	4-5		4
	Polyester	4-5	5	5		3-4
3b	Nylon	4-5	4-5	5		3-4
	Polyester	4-5	4-5	5		3-4
3c	Nylon	4-5	5	5		3-4
	Polyester	4-5	4-5	5		4
3d	Nylon	4-5	5	5		3-4
	Polyester	4-5	4-5	5		4
3e	Nylon	4-5	4-5	4-5		3-4
	Polyester	4-5	4-5	5		4
3f	Nylon	4-5	4-5	4-5		3-4
	Polyester	4-5	5	5		4

CH = Colour change; SN = staining on nylon; SC = staining on cotton

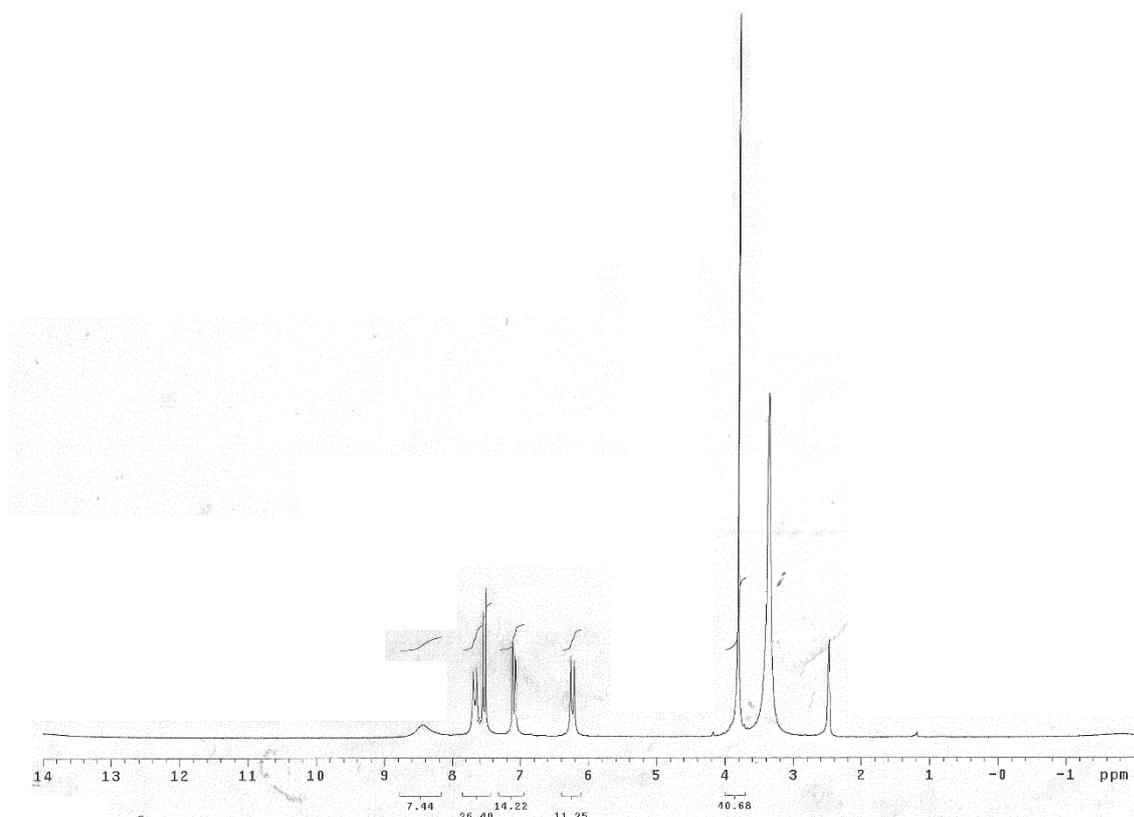


Fig. 2: <sup>1</sup>H NMR spectrum of dye 3a

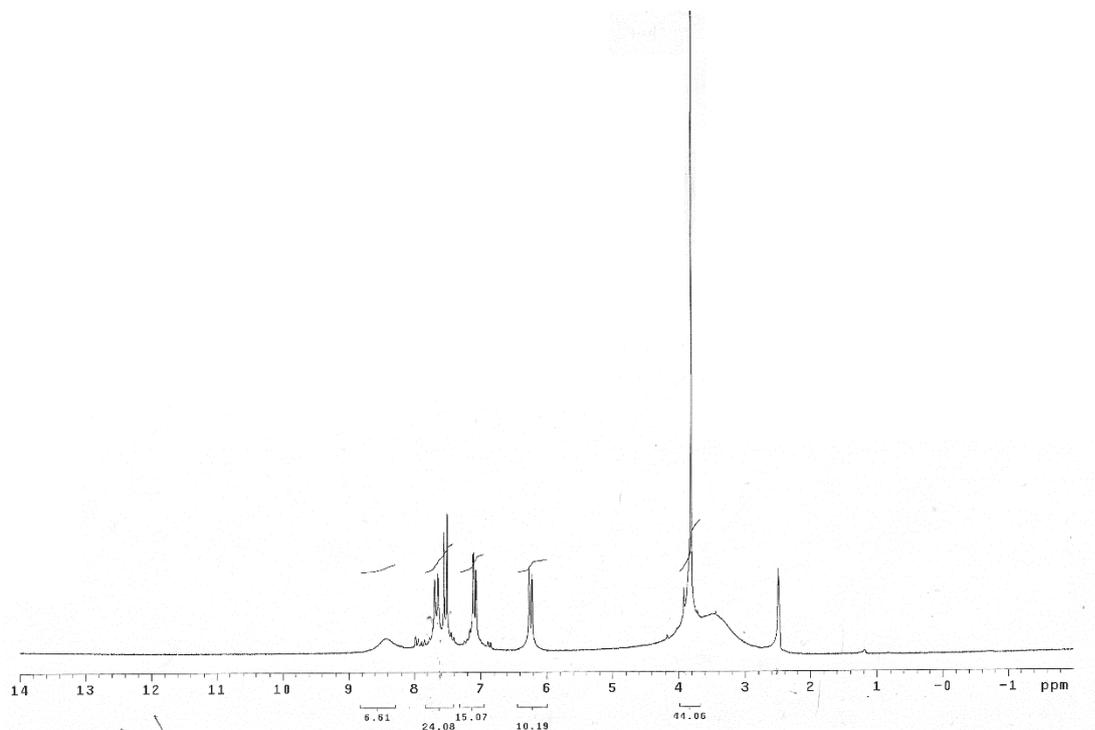


Fig. 3: <sup>1</sup>H NMR spectrum of dye 3b

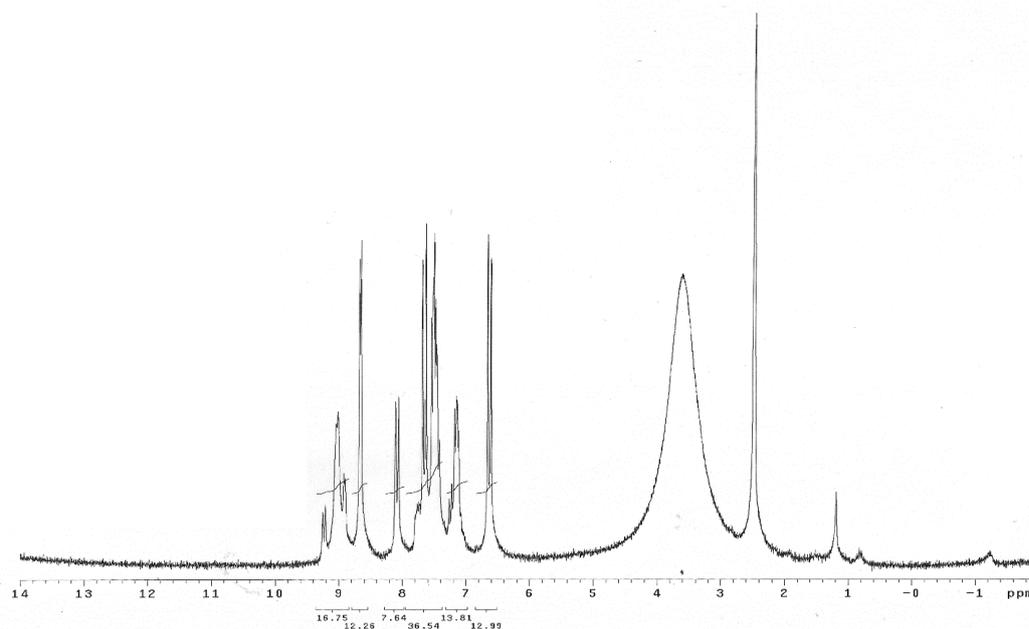


Fig. 4: <sup>1</sup>H NMR spectrum of dye 3c

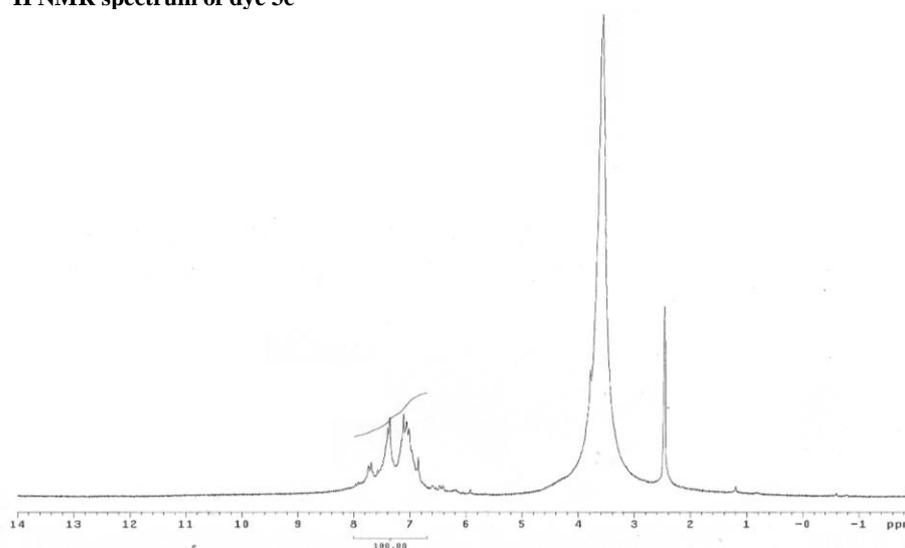


Fig. 5: <sup>1</sup>H NMR Spectrum of dye 3d

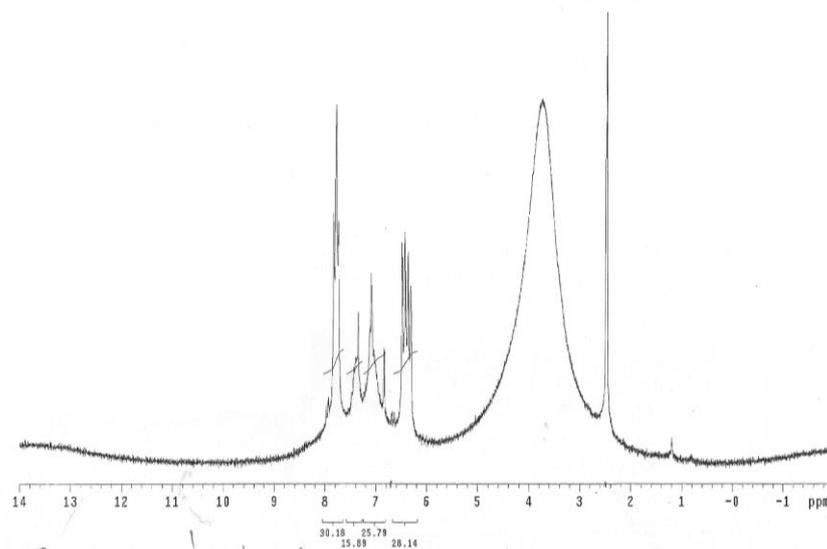


Fig. 6: <sup>1</sup>H NMR Spectrum of dye 3e

**Fastness to light**

The light fastness of colourants on any fabric is very significant and is dependent on the nature of the fabric and substituents. The substituents tend to change the density of electrons around the azo group. High fastness to light was observed for dyes 3a, 3e and 3f on dyed nylon fabric (Table 2) and this may be attributed to the increased electron density due to the OH group ortho to the azo group. These mentioned dyes exhibited lower fastness to light on the polyester fabric, compared to those of nylon fabric. This agrees with the notion that the azo compounds which have electron donating (OH) groups ortho to the azo group, generally show high light fastness on nylon fibres than to polyester fibres. In general, the dyes prepared in this study showed satisfactory photostability, ranging from grade 4 to 5 according to the international greyscale.

**Table 2: Light fastness and rub fastness properties of fabrics dyed with synthesised dyes 3a-f**

Dye	Fabric	Light	Rub	
			Dry	Wet
3a	Nylon	5/6	3-4	4
	Polyester	5	3-4	3-4
3b	Nylon	5	3-4	4
	Polyester	5	4	4
3c	Nylon	5	3-4	4
	Polyester	4-5	3-4	4
3d	Nylon	5	3-4	4
	Polyester	5	3-4	4
3e	Nylon	5/6	3-4	3-4
	Polyester	5	4	4
3f	Nylon	5/6	3-4	4
	Polyester	5	4	3-4

**Fasting to rubbing**

Rub fastness data are given in Table 2, and the results are expressed in terms of change in colour and staining of adjacent white fabric (cotton) in both wet and dry conditions. It was observed that dry rub fastness was less than wet rub fastness except for dyes 3a and 3e on polyester and nylon respectively. This observation is contrary to so many cases where wet rub fastness had lesser grade scale ratings due to the removal of dye molecules during rubbing, assisted by water molecules.

**Fastness to heat (sublimation)**

Textiles are frequently exposed to heat in either during the process of use or manufacturing. For example, they may come into contact with heat during ironing, heat setting and steaming. The change in colour of the dyed fabrics was investigated with adjacent nylon and polyester fabrics, and the staining of colour on the adjacent fabrics was assessed using the grey scale Good (grade 3-4) to very good (grade 4) for dyed polyester and nylon fabrics was found. These results are not surprising as all the dye molecules contain the ether group in their structures. The results of the heat fastness (Table 2) showed that dyes 3a, 3b, and 3c gave lower grades (3-4), especially on nylon fabric than dyes 3d, 3e and 3f. This indicates that they have special ease of sublimation.

**Conclusion**

In this paper, a new series of monoazo disperse dyes, 3a – 3f were synthesised from 4-phenoxyaniline and various carbocyclic crude heterocyclic coupling compounds. They were applied onto nylon and polyester fabrics via pressure dyeing method. It was observed that the pressure dyeing method enhanced the concentration of dye molecules in the

polymer matrix particularly in the case of nylon fabric. The colourants 3a, 3e and 3f showed good photostability of grade 5/6 compared to those on polyester with grade 5. The wash fastness of the dyes was found to be lower on the nylon fabric than on the polyester fabric. The heat fastness thermal stability of the colourants 3b, 3c, 3d and 3e were found to be good (grade 3-4) but the heat fastness of the rest of the colourants on polyester and nylon were very good. In general, the dyes showed better light and heat fastness on nylon fabric than on polyester fabric which may be attributed to the increase in the concentration of dye molecules due to pressure dyeing. The results of this study, especially on the basis of sublimation fastness indicate that the dyes are potential candidates for transfer printing.

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