



Journal of Applied Sciences

ISSN 1812-5654

science
alert

ANSI*net*
an open access publisher
<http://ansinet.com>



Research Article

Disazo Disperse Dyes Derived from 2-chloro-4-methylaniline and 3-aminophenol and Their Application on Nylon 6 Fabric Using Pressure Dyeing Method

¹J.O. Otutu, ¹E.K. Ossai and ²S.U. Ameuru

¹Department of Chemistry, Faculty of Science, Delta State University, P.M.B. 1, Abraka, Nigeria

²Department of Textile Science and Technology, Ahmadu Bello University, Zaria, Nigeria

Abstract

In this study, the synthesis and application of some new disazo disperse dyes derived from 2-chloro-4-methylaniline and 3-aminophenol on nylon 6 fabric are described. The dyes were synthesized via diazotisation of 2-chloro-4-methylaniline using sodium nitrite in an acid (H_2SO_4) medium and coupling the diazonium salt solution produced with 3-aminophenol to give an intermediate azo compound. The amino azo intermediate was subjected to further diazotisation reaction and coupling with 1-naphthylamine, 3-chloroaniline, 4-chloroaniline, 4-hydroxybenzoic acid, 1,3-dihydroxybenzene and 2-chloroaniline to afford the six disazo disperse dyes. The dyes were characterized by spectral methods (IR, NMR and UV-visible). The results of the fastness tests showed that the light, rubbing and washing fastness of the nylon dyeings using the pressure dyeing method were higher compared with those obtained through normal dyeing method. The results of the study, thus showed that the fastness performance of disperse dyes on nylon fibre could be enhanced by using the pressure dyeing method.

Key words: Disperse dyes, polyamide, pressurized dyeing, fastness properties, spectral methods

Received: September 21, 2015

Accepted: January 29, 2016

Published: March 15, 2016

Citation: J.O. Otutu, E.K. Ossai and S.U. Ameuru, 2016. Disazo disperse dyes derived from 2-chloro-4-methylaniline and 3-aminophenol and their application on nylon 6 fabric using pressure dyeing method. *J. Applied Sci.*, 16: 146-153.

Corresponding Author: J.O. Otutu, Department of Chemistry, Faculty of Science, Delta State University, P.M.B. 1, Abraka, Nigeria

Copyright: © 2016 J.O. Otutu *et al.* This is an open access article distributed under the terms of the creative commons attribution License, which permits unrestricted use, distribution and reproduction in any medium, provided the original author and source are credited.

Competing Interest: The authors have declared that no competing interest exists.

Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

Polyethylene terephthalate (PET) fibres are characterized by the crystalline structure and hydrophobic nature. At present, disperse dyes are the only class of dyes that is best suitable for dyeing these mordant fibres (Hou *et al.*, 2013; Metwally *et al.*, 2013; Shuttleworth and Weaver, 1990; Oner *et al.*, 2013; El-Zawahry and Abdelghaffar, 2013). The dyes are usually applied from fine aqueous suspension in water. This is brought about by the addition of dispersants since disperse dyes are by nature non ionic aromatic compounds with sparing solubility in water (Daruwalla, 1974; Ortega-Luoni *et al.*, 2007). The main functions of the dispersing agent are to assist the process of reducing the particle size of the dye during manufacture, encourage the change from powder to dispersion during the preparation of the dyebath and also help to maintain a stable dispersion during dyeing (Koh *et al.*, 2004).

In dilute conditions, the dispersants can assist to increase the amount of dissolved dye molecules in the dyebath and hence the rate at which the absorption of dye is taking place (Lee *et al.*, 2005; Ukponmwan *et al.*, 1999; Otutu, 2012). However, most disperse dyes are now being applied in the dyeing of nylon, acrylic, polypropylene and poly (butylene terephthalate) fabrics (Dadras *et al.*, 2014). Chemically, there are many chromophoric systems involved in the disperse dye chemistry. These are the azo dyes (60%), anthraquinones (25%) and the rest is distributed among the naphthalimide, quinophthalone, nitro and methane dyes (Otutu *et al.*, 2008; Towns, 1999; Huang *et al.*, 2013; Acar *et al.*, 2013).

The 2-chloro-4-methylaniline, which is the main intermediate for this study is yet to be used in the preparation of azo disperse dyes. Thus, this study is aimed at synthesising disazo disperse dyes from 2-chloro-4-methylaniline and 3-aminophenol and their subsequent application on nylon. Also, in this study, the traditional method of dyeing this type of fibre was compared with the pressure method in terms of fastness properties (such as wash, light, sublimation and rubbing).

MATERIALS AND METHODS

The 2-Chloro-4-methylaniline and 3-aminophenol were purchased from sigma-Aldrich chemical company. All other chemical reagents were of analytical grade and were used as procured from different chemical companies (Merck, Loba chem.-limited and S.D fine).

Analytical methods: The infrared spectra were recorded on KBr discs, using a shimadzu 8500s FT-IR spectrometer. Melting

points were measured with an electrothermal 9100 apparatus in open capillaries and are not corrected. The proton nuclear magnetic resonance spectra ($^1\text{H-NMR}$) and carbon-13 nuclear magnetic resonance ($^{13}\text{C-NMR}$) were recorded on mercury 200 BB series spectrometer. Deuterated chloroform (CDCl_3) and dimethyl sulphoxide (DMSO-d_6) were used as NMR solvents. Chemical shifts were reported in parts per million (ppm) down field from internal tetramethylsilane (TMS) reference standard. The UV-visible absorption spectra were recorded on Genesys 10S V 1.200 spectrometer. The progress of the reaction was monitored in silica gel, 60 coated plate, F_{254} , 0.2 mm (Merck) using chloroform-methanol (95:5) and toluene-acetic acid (90:10) as the solvent systems.

Synthesis of dye 3: The amino compound, 2-chloro-4-methylaniline (12 mL) was dissolved in sulphuric acid (98%, 10 mL) and diazotized by dropwise addition of sodium nitrite (8.55, 124 mmol) in 20 mL water at 0-5°C. Excess nitrous acid was detected using urea (0.75 g). The coupling reagent, 3-aminophenol was dissolved in 25 mL of sodium hydroxide solution. The diazotized amine was added slowly to the diazonium salt solution and stirred at 0-5°C for 1 h. The precipitate obtained was isolated by filtration, washed with water, dried and recrystallized from hot ethanol-methanol mixture to afford compound 3 in 91% yield.

Synthesis of 4-aminonaphthylazo-2-hydroxyphenylazo 2-chloro-3-methylbenzene (5a): Compound 3 (7.0 mmol) was mixed with water (15 mL) and H_2SO_4 (5 mL). Sodium nitrite (7.0 mmol) in 10 mL of water was added to the mixture and stirred for 20 min at 0-5°C. The diazonium salt solution prepared was added dropwise to the solution of 1-naphthylamine (2.0 g, 14 mmol) in acetic acid (6 mL). The reaction mixture was stirred at room temperature for 2 h. The product formed was isolated by filtration washed with water and recrystallized from ethanol-methanol mixture. The solvent-system used for the Thin Layer Chromatography (TLC) was chloroform-methanol (95:5).

The 2-amino-4-chlorophenylazo-2'-hydroxyphenylazo-2''-chloro-3-methylbenzene (5b): Compound 3 (7.0 mmol) was diazotized in concentrated sulphuric acid (5 mL) by the addition of sodium nitrite (7.0 mmol) in 10 mL of water and stirred for 20 min at 0-5°C. The 3-chloroaniline (3.5 mL) was dissolved in acetic acid (5 mL). The diazotized amine was added slowly to this solution and stirred for 1 h and left to stand over night. The precipitate obtained was isolated by filtration, washed with water and purified by column

chromatography on silica with increasing amounts of methanol as eluent. The solvent system used for the TLC was chloroform-methanol (95:5).

The 2-amino-4-chlorophenylazo-2'-hydroxyphenylazo-2''-chloro-3-methyl benzene (5c): Compound 3 (7.0 mmol) was mixed with water (15 mL), sulphuric acid (6 mL) and the mixture was cooled to 0-5°C. Sodium nitrite (7.0 mmol) in 10 mL of water at 0-5°C was added drop wise to the above mixture and stirred for 20 min. The diazonium salt solution obtained was added slowly to a solution of 4-chloroaniline (7.0 mmol) in acetic acid (6 mL) with stirring for 1 h. The precipitate formed was isolated by filtration, washed with water and purified by recrystallization from N,N-dimethylformamide-water mixture (1:2). The solvent-system for the TLC was toluene-acetic acid (90:10).

The 2,4-dihydroxyphenylazo-2'-hydroxyphenylazo-chloro-3-methyl benzene (5d): Compound 3 (7.0 mmol) was diazotised in concentrated sulphuric acid (5 mL) by the dropwise addition of sodium nitrite (7.0 mmol) in 10 mL of water and stirred at 0-5°C for 20 min 4-hydroxybenzoic acid (0.932 g, 7.0 mmol), dissolved in acetic acid (10 mL). The diazonium salt solution prepared was slowly added to the 4-hydroxybenzoic acid solution and stirred at room temperature for 2 h and left to stand overnight. The product formed was isolated by filtration, washed with water and purified by recrystallization from N,N-dimethylformamide-water (1:2) mixture. The solvent-system used for the TLC was chloroform-methanol (95:5).

The 4-amino-3-chlorophenylazo-2-hydroxyphenylazo-2''-chloro-3'-methylbenzene (5e): Compound 3 (7.0 mmol) was diazotised in concentrated sulphuric (5 mL) water (20 mL) by the dropwise addition of sodium nitrite (7.0 mmol) in 10 mL of water and stirred at 0-5°C for 20 min. The 1,3-Dihydroxybenzene (1.0 mmol) was dissolved in (1.0 g, 25 mmol) of sodium hydroxide (20 mL). The diazonium salt solution prepared was slowly added to the solution of 1,3-dihydroxybenzene and stirred for 1 h. The precipitate formed was isolated by filtration, washed with water and purified by recrystallization from N,N-dimethylformamide-water (1:2) mixture. The solvent-system used for the TLC was chloroform-methanol (95:5).

The 4-amino-3-chlorophenylazo-2-hydroxyphenylazo-2''-chloro-3-methylbenzene (5f): Compound 3 (7.0 mmol) was mixed with water (20 mL) and concentrated sulphuric acid

(5 mL) and diazotized by the dropwise addition of sodium nitrite (7.0 mmol) with stirring at 0-5°C for 20 min. The diazonium salt solution prepared was slowly added to 2-chloroaniline (3.5 mL) in acetic acid (5 mL) with stirring for 1.5 h. The crude product formed was isolated by filtration, washed with water and purified by column chromatography on silica with increasing amount of methanol. The solvent system used for the TLC was toluene-acetic acid (90:10).

Dye application

Preparation of dye dispersions: A disperse solution of a sample containing a single dye was prepared by dissolving the dye (1.0 g) and adding a dispersing agent, sodium lignin sulfonate (1.0 g, Kao Corporation, Japan) and the appropriate volume of the disperse solution was adjusted to 100 mL mark.

Nylon 6 fabric: The dyebath was prepared by mixing the dye dispersion with 2% acetic acid to bring the pH to 5. The nylon fabric was wetted and immersed in the dyebath at 45°C. The bath temperature was then raised to 102°C over 15 min and this was maintained for 30 min. At this temperature, formic acid (1.5 mL of 10%) was added to the dyebath to achieve good exhaustion. After cooling, the dyed nylon 6 fabric was rinsed with water and reduction-cleared with iodet T (2.0 g L⁻¹) in a bath containing water (material to liquor ratio 1:2) at 46°C for 15 min followed by rinsing thoroughly with cold water and air dried (Giles, 1974; Burkinshaw, 1994).

In the pressure dyeing, the dyebath was prepared as described in the normal dyeing method and the bath was then placed in a vessel whose pressure was set at (40 kPa) and heated to 100°C for 10 min. The bath was then cooled and the dyed fabric was reduction-cleared as described for the normal dyeing method.

Fastness tests

Light fastness: In this test, the dyed fabric samples were exposed to xenon arc fading lamp of an Atlas ES 25 weather-O-meter, according to the conditions of AATCC test method (AATCC., 2007). The tested samples were then placed alongside with a scale of light sensitive blue dyed wool standards of ratings 1-8 designed to fade after different time periods.

Washing fastness: Colour fastness to laundering was determined by the ISO 105 CO₂ (ISO., 1989) standard test method, according to which the test specimen was stitched with two pieces of test fabrics nylon) and then put into

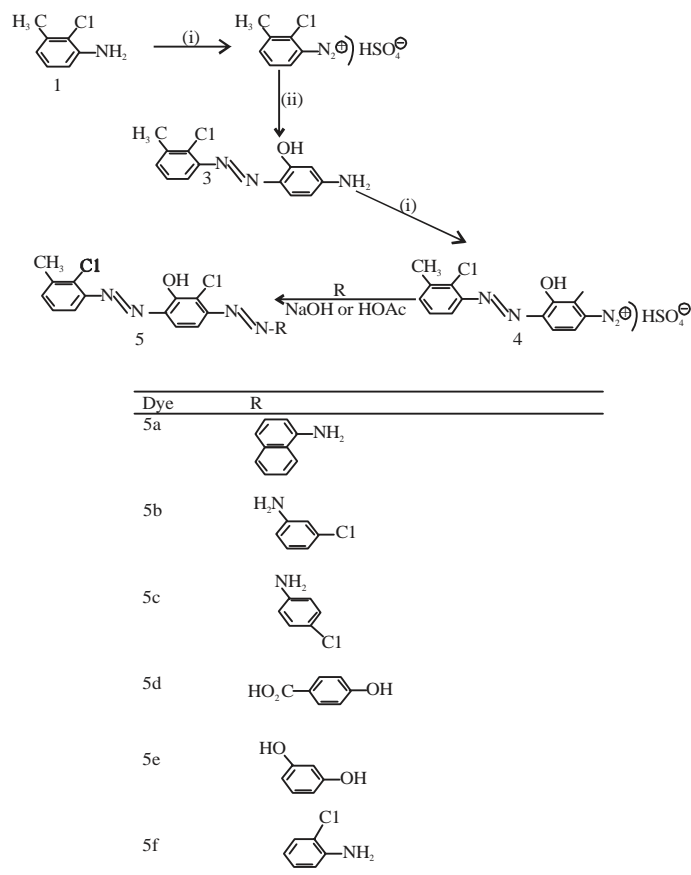


Fig. 1: Reaction scheme for the synthesis of disazo dye compounds, (i) NaNO_2 , H_2SO_4 , H_2O , $0-5^\circ\text{C}$ and (ii) $\text{C}_6\text{H}_7\text{NO}$

Table 1: Physical characteristics of prepared disazo disperse dyes

Dyes	Molecular formula	Molecular weight (g mol^{-1})	Yield (%)	MP ($^\circ\text{C}$)
5a	$\text{C}_{23}\text{H}_{18}\text{N}_5\text{OCl}$	415.5	55	155
5b	$\text{C}_{19}\text{H}_{15}\text{N}_5\text{OCl}_2$	400	64	112
5c	$\text{C}_{19}\text{H}_{15}\text{N}_5\text{OCl}_2$	400	62	180-182
5d	$\text{C}_{19}\text{H}_{15}\text{N}_4\text{O}_3\text{Cl}$	366.5	72	165-167
5e	$\text{C}_{19}\text{H}_{15}\text{N}_4\text{O}_3\text{Cl}$	382.5	68	120
5f	$\text{C}_{19}\text{H}_{15}\text{N}_5\text{OCl}_2$	400	58	135

Table 2: Wash fastness and light fastness properties of dyes 5a-5e (2% owf) on nylon fabric (normal dyeing method)

Dyes	Change in color	Staining			Light fastness
		Cotton	Nylon	Acrylic	
5a	4-5	5	5	4/5	5
5b	4-5	5	5	4/5	5
5c	4-5	4/5	5	4/5	5
5d	4-5	4/5	5	4/5	5
5e	4-5	5	5	4/5	5
5f	4-5	5	5	4/5	5

washing liquid containing 5 g L^{-1} soap and 2 g L^{-1} sodium carbonate and liquor ratio 50:1 for 30 min at 60°C . The combination of specimen and test fabrics was then washed with distilled water and dried.

Rubbing fastness: Colour fastness to rubbing was evaluated according to ISO 105-X12 (ISO., 2001). This test was undertaken using a crockmeter (Taber@crockmeter, USA) on which the fabric specimen was subjected to rubbing with a sample of standard undyed cotton fabric in order to evaluate the colour transfer. Two tests were involved, one with dry rubbing fabric, the other with wetted fabric.

RESULTS

The preparation of 2-chloro-3-methylaniline disazo disperse dye derivatives (5a-5e) were carried out according to Fig. 1. The characterization data of the synthesized disazo compounds are given below and their physical properties are shown in Table 1. Also, Fig. 2-4 shows the NMR spectra of dyes 5a, 5c and 5e.

Table 2 shows the wash fastness properties of the disazo disperse dyes. As shown in the table, the colour change values were found to be 5 (excellent) and the staining test results of adjacent multifibres were generally found to be 4-5

and 5 greyscale ratings. The colour fastness to light were found to be 5 greyscale ratings (in the normal dyeing method)

but there was a slight increase on light fastness between 5-6 and 6 (pressure dyeing method) (Table 3).

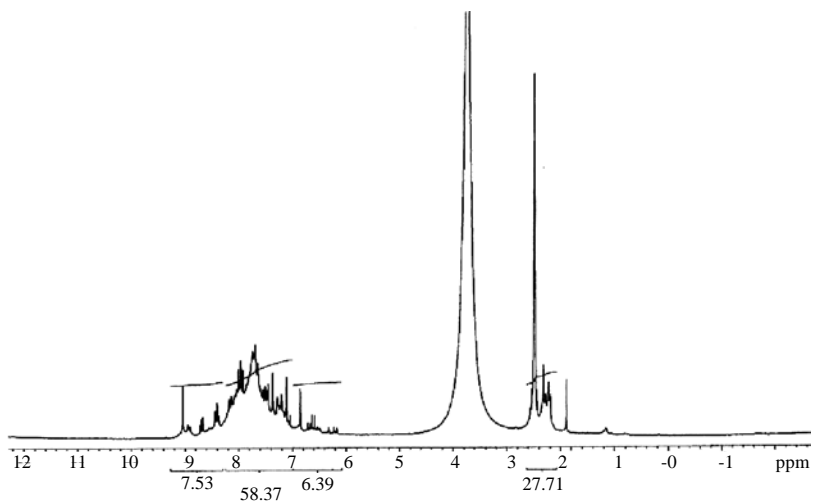


Fig. 2: ¹H-NMR of dye 5a

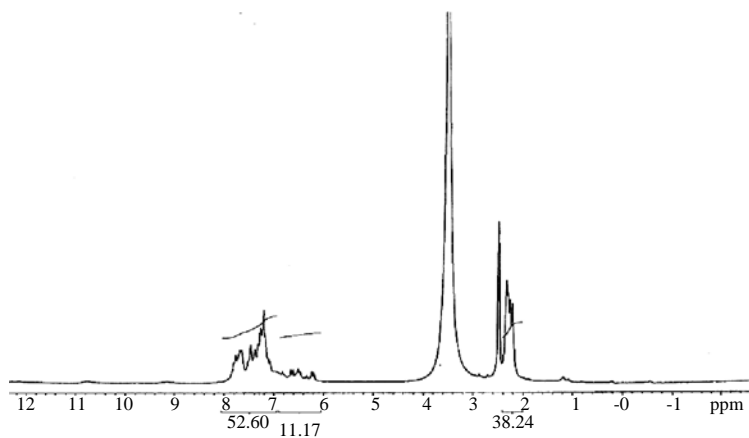


Fig. 3: ¹H-NMR of dye 5b

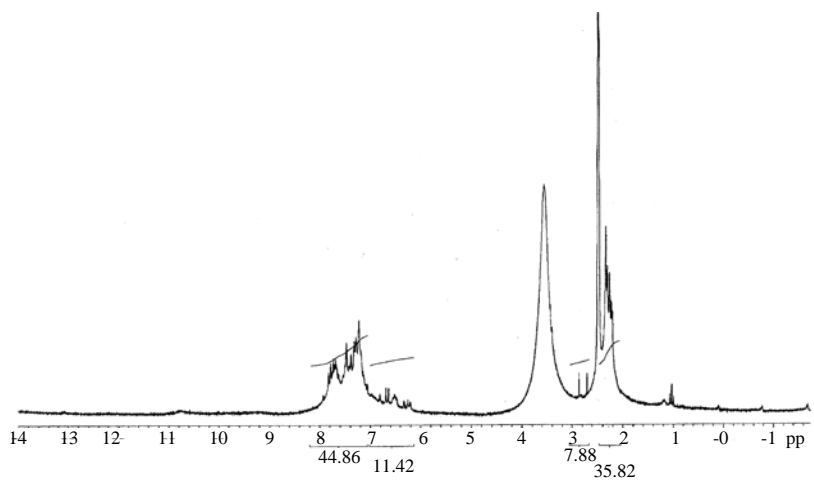


Fig. 4: ¹H-NMR of dye 5c

Table 3: Wash fastness and light fastness of dyes 5a-5e (2% owf) on nylon fabric (pressurized dyeing method)

Dyes	Change in color	Staining			Light fastness
		Cotton	Nylon	Acrylic	
5a	5	5	5	5	5-6
5b	5	5	5	5	5-6
5c	5	5	5	5	5-6
5d	5	5	5	5	5-6
5e	5	5	5	5	6
5f	5	5	5	5	5-6

Table 4: Rubbing fastness properties of dyes 5a-5e on nylon fabric

Dyes	Normal dyeing method		Pressure dyeing method	
	Dry	Wet	Dry	Wet
5a	3/4	3/4	4/5	4/5
5b	3/4	3	4/5	4/5
5c	3/4	3/4	4/5	4/5
5d	3/4	3	4/5	4/5
5e	3/4	3/4	4/5	4/5
5f	3/4	3	4/5	4/5

Table 4 shows the rubbing fastness values. In the normal dyeing method, but the dry and wet rubbing test results showed grayscale ratings of between 3 and 3-4. However, in the pressure dyeing method, the result were found to be 4.

Preparation of 4-aminonaphthylazo-2-hydroxyphenylazo-2-chloro-3-methylbenzene (5a): Compound 5a was prepared by diazotizing dye 3 and adding 1-naphthyliniline under acidic conditions (Karci, 2005; David and Geoffery, 1994). The yield of product was 55%, in p 155 °C.

The IR (KBr); V (cm⁻¹) 3396 (OH, NH_{str}), 2862-3090 (C-H_{str}), 1625-1585 (Ar-ring) 1467 cm⁻¹ (naphthyl), 1315-1296 (O-H bending) 1051 (C-O_{str}), 848- 920 (Ar-C-H out of place) ¹HNMR (DMSO-d₁ 199.97 MHz), dH (ppm): 2.22 (2H, s, NH₂), 2.81 (3H, s, CH₃) 6.24 (2H, d, J = 8.20 Hz, ArH). 6.68- 7.68 (6H, naphthyl H) 8.90 (3H, M, ArH), 9.15 (1H, s, OH).

The ¹³C-NMR (DMSO-d₆), 199.97MHz and δc (ppm): 133.8, 132.6, 131.8, 130.2, 129.8, 128.8, 128.7, 127.9, 127.5, 127.0, 126.3, 125.1, 124.1, 123.5, 122.5, 120.6, 41.4, 41.0, 40.6, 40.1, 39.7 and 38.9.

$$\lambda_{\max} \text{ (nm) (in DMF) = 400}$$

Preparation of 2-amino-4-chlorophenylazo-2-hydroxyphenylazo-2-chloro-3-methyl benzene (5b): Compound 5b was prepared via diazotization of dye 3 and coupling with 3-chloroaniline in acidic conditions. The yield of the pure product was 64%, m.p. 112 °C.

The IR(KBr), V (cm⁻¹): 3416 (OH, NH_{str}), 1600 (Ar str) 1410 (-N-N), 1205-1103 (C-O_{str}) 883- 819 (Ar C-H out of plane bending) and 684 (C-Cl_{str}).

The ¹H-NMR (DMSO-d₆ 199.9 MHz dH (ppm): 2.25 (2H, S, NH₂), 3.45 (3h, S CH₃), 6.25 (2H, d, J = 8.25 Hz, ArH), 7.20- 7.86 (3H, m, ArH), 8.40-8.92 (3H, m, ArH) and 9.10 (1H, s, OH).

The ¹³C-NMR (CDCl₃, 50.28 MHz), δc (ppm): 143.8, 140.7, 138.2 131.2, 130.7, 128.8, 127.5 126.4, 115.4, 112.8, 41.4, 41.0, 40.6, 40.2, 39.6, 39.5, 38.8 and 21.7.

$$\lambda_{\max} \text{ (nm) DMF = 437}$$

Preparation of 2-amino-5-chlorophenolazo-2-hydroxyphenylazo-2-chloro-3-methylbenzene (5c):

Compound 5c was prepared by the diazotization of dye 3 and coupling with 4-chloroaniline under acid conditions. The yield of the pure product was 62%, m.p 180-182 °C.

The IR (KBr), V (cm⁻¹) 3404- 3520 (OH_{str} NH_{str}) 3063-3167 (C-H_{str}), 1600 (Ar str) 1471 (N = N), 1315 (OH bending) 1051-1178 (C-O_{str}) and 821- 960 (Ar-CH out of plane bending) 702 (C-Cl_{str}).

The ¹H-NMR (DMSO-d₆, 199.97 MHz) d_H (ppm): 2.24 (2H, s, NH₂), 3.45 (3H, s, CH₃), 6.42 (2H, d, J = 8.10 Hz, ArH), 6. 46-7.25 (3H, m, ArH), 7.45-7.66 (3H, m, ArH), 10.68 (1H, s, OH).

The ¹³C-NMR (DMSO-d₆, 50.28 MHz), δ_c (ppm): 142.3, 137.4, 132.3, 131.2, 130.6, 129.9, 129.4, 117.7, 116.2, 111.8, 41.4, 41.0, 40.6, 40.2, 39.7, 39.3, 38.9, 21.2 and 21.0.

$$\lambda_{\max} \text{ (nm) in DMF = 437}$$

Preparation of 4-hydroxyphenylazo-2-hydroxyphenylazo-2-chloro-3-methylbenzene (5d):

Compound 5d was synthesized via the diazotization of dye 3 and subsequent coupling with 4- hydroxybenzoic acid. The yield of the pure product was 72% mp: 165-167 °C.

The IR (KBr), Vcm⁻¹: 3404-3518 (OH_{str}) 3.061-3140 (C-H_{str}), 1600 (Ar-Str), 1498 (N = N), 1031-1134 (C-O_{str}), 817-889 (Ar C-H out of plane bending) and 702 (C-Cl_{str}).

The ¹H-NMR (DMSO-d₆, 199.97 MHz) dH (ppm): 3.46 (3H, s, CH₃), 6.12 (2H, d, J = 8.2 Hz, ArH), 6.26-7.26 (3H, m, ArH), 7.41-7.80 (3H, m, ArH) and 10.67 (1H, s, OH).

The ¹³C-NMR (CDCl₃, 50.28 MHz), δ_c (ppm): 132.35, 131.2, 130.6, 130.0, 117.7, 94.6, 41.4, 41.0, 40.6, 40.2, 39.7, 39.3, 38.9 and 21.3.

$$\lambda_{\max} \text{ (nm) DMF = 477}$$

Preparation of 2, 4-dihydroxyphenylazo-2-hydroxyphenylazo-2-chloro-3-methylbenzene (5e):

Compound 5e was prepared by the diazotization of dye 3 and coupling the diazonium salt solution with 1, 3-dihydroxybenzene under alkaline. The yield of the product was 08%, m.p. 120 °C.

The IR (KBr), $V(\text{cm}^{-1})$: 3464 (OH_{str}), 3060 (C-H_{str}), 1535 (Ar str), 1476 (N=N) 1050 (C-O_{str}), 820- 877 (Ar C-H out of plane bending) and 702 (C-Cl_{str}).

The $^1\text{H-NMR}$ (DMSO-d_6 , 199.97 MHz), d_{H} (ppm): 3.63 (3H, s, CH_3) 6.15 (2H, d, $J = 8.10$ Hz, ArH), 6.45-7.25 (3H, m ArH), 7.81 (3H, m, ArH) and 10.80 (1H, s, OH).

The $^{13}\text{C-NMR}$ (DMSO-d_6 , 50.28 MHz), δ_{C} (ppm): 128.7, 128.0, 117.6, 105.0, 96.8, 57.9, 41.2, 40.8 40.4, 40.0. 39.5, 39.1, 38.7 and 21.1.

$$\lambda_{\text{max}} (\text{nm, in DMF}) = 404$$

Preparation of 4-amino-2-chlorophenylazo-2'-hydroxyphenylazo-2-chloro-3-methyl benzene (5f):

Compound 5f was synthesized via the diazotization of dye 3 and subsequent coupling with 2-chloroaniline under acidic conditions. The yield of the pure product was 58% m.p: 135 °C.

The IR (KBr), $V(\text{cm}^{-1})$: 3456 (O-H_{str}), 2974 (C-H_{str}), 1524-1652 (Ar str), 1467 (C = N) 1380 (OH bending), 1061. (C-O_{str}) 817-886 (Ar C-H out of plane bending) 719 (C-Cl_{str}).

The $^1\text{H-NMR}$ (DMSO-d_6 , 199.97 MHz), d_{H} (ppm): 2.23 (2H, s, NH_2), 3.42 (3H, s, CH_3), 6.25 (2H, d, $J = 8.20$ Hz, ArH), 6.76-7.45 (3H, m, ArH) 7.75-8.85 (3H, m-ArH) and 9.12 (1H, s, OH).

The $^{13}\text{C-NMR}$ (DMSO-d_6 , 50.28 MHz), δ_{C} (ppm): 128.6, 127.9, 117.6, 105.6, 96.7, 57.8, 41.3, 40.9 and 21.7.

$$\lambda_{\text{max}} (\text{nm, DMF}) = 425$$

DISCUSSION

Synthesis and spectroscopic characterization: The 4-amino-2-hydroxyphenylazo-2-chloro-3-methylbenzene disazo disperse dye derivatives were obtained in 55-72% yields as brown crystals from the diazotization and coupling reactions of the aforementioned intermediate with various aryl coupling agents. The main reason for the variation in the yields of the disazo disperse dyes is connected with the different electron-donating and electron withdrawing substituent groups attached to the aryl azo compounds. This assertion corroborates with those in the literature. The relatively lower yield of compound 5a is not completely understood, however, the acidic condition of the coupling reaction is believed to be responsible for this (Francis, 2003; Paula, 1995; Otutu and Osabohien, 2013). It was also observed that the melting points of some of the dyes were relatively low, suggesting that they could be potential candidates for transfer printing of textiles. This assertion corroborates with those in the literature (Nkeonye, 1987; Dong and Jang, 2014).

The assumed structures of the disazo disperse dyes were established by FTIR, $^1\text{H-NMR}$, $^{13}\text{C-NMR}$ and UV-visible spectra as presented in the results section.

Colourfastness properties: The wash fastness of dyed textiles is of great importance to the consumer. The wash fastness against 60 °C for 30 min washings were carried out and the colour change values were found to be good ratings for the nylon dyeings obtained using the normal dyeing method compared with the excellent ratings found for the pressure dyeing method.

It was also observed that the photostability of the nylon dyeings obtained from the normal dyeing method was lower compared to those obtained from the pressure method. The main reason for this increase is attributed to the increase in dye concentration in the dyed nylon fabric (Evans and Stapleton, 1978; Otutu *et al.*, 2014; Huang *et al.*, 2013).

Also there was a slight increase in both dry and wet rubbing fastness of the grayscale ratings of nylon dyeings obtained from pressure dyeing method compared with the normal dyeing method. The reason for this difference is not immediately understood, but it is believed to be connected with the high dye concentration in the nylon fabric.

CONCLUSION

The results of the fastness properties showed that wash fastness values of the dyed nylon fabrics were higher in the pressure dyeing method compared to the values obtained in normal dyeing method. It was also found that the photostability of the dyes on nylon dyed using pressure method was higher compared to those of the normal dyeing method due to the high dye concentration in the dyed nylon fabric. The results of the study, therefore showed that the performance of the disazo disperse dyes was enhanced by using pressure dyeing method. However, the effect of moderate pressure on the polyamide fibre was not investigated.

REFERENCES

- AATCC., 2007. Colorfastness to light testing method AATCC 16. American Association of Textile Chemistry and Colourists, Research Triangle Park, North Carolina.
- Acar, I., S. Topcu, Z. Biyiklioglu and H. Kantekin, 2013. Synthesis, characterisation and electrochemical investigation of phthalocyanines with pendant 4-{2-[2-(4-*tert*-butylphenoxy) ethoxy]ethoxy} substituents. *Coloration Technol.*, 129: 259-266.

- Burkinshaw, S.M., 1994. Application of Dyes. In: The Chemistry and Application of Dyes, David, R.W. and H. Geoffery (Eds.). Plenum Press, New York, London, pp: 326-332.
- Dadras, F.S., K. Gharanjig and S. Raissi, 2014. Optimising by response surface methodology the dyeing of polyester with a liposome-encapsulated disperse dye. *Coloration Technol.*, 130: 86-92.
- Daruwalla, E.H., 1974. Physical Chemistry of Dyeing: State of Dye in Dyebath and in Substrate. In: The Chemistry of Synthetic Dyes, Volume VII, Venkataraman, K. (Ed.). Academic Press, New York, pp: 69-114.
- David, R.W. and H. Geoffery, 1994. The Chemistry and Application of Dyes. 1st Edn., Plenum Press, New York and London, pp: 115-231.
- Dong, Y. and J. Jang, 2014. Environmentally friendly coloration of cotton by the UV-induced photografting of α -bromoacrylamido dyes. *Coloration Technol.*, 130: 296-304.
- El-Zawahry, M.M. and F. Abdelghaffar, 2013. Preparation and characterisation of novel phospholipid cationic liposomes to improve the alkaline hydrolysis and dyeability of polyester fabric. *Coloration Technol.*, 129: 193-202.
- Evans, N.A. and I.W. Stapleton, 1978. Structural Factor Affecting the Light Fastness of Dyed Fibre. In: The Chemistry of Synthetic Dyes, Volume VIII, Venkataraman, K. (Ed.). Academic Press, New York, London, pp: 221-227.
- Francis, A.C., 2003. Organic Chemistry. 5th Edn., McGraw Hill, New York, pp: 944-945.
- Giles, C.H., 1974. A Laboratory Course in Dyeing. Soc. Dyers and Colourists (SDC), Bradford, UK., pp: 93-101.
- Hou, A., M. Li, F. Gao, K. Xie and X. Yu, 2013. One-step dyeing of polyethylene terephthalate fabric, combining pretreatment and dyeing using alkali-stable disperse dyes. *Coloration Technol.*, 129: 438-442.
- Huang, G., J. Dai, F. Dong, J. Wang and Y. Jia, 2013. Compatibility of a disperse dye mixture in supercritical carbon dioxide dyeing. *Coloration Technol.*, 129: 305-311.
- ISO., 1989. Textings-tests for colour fastness-part CO2: Colour fastness to washing test 2. ISO 105-CO2, International Organization for Standardization, Geneva, Switzerland. http://www.iso.org/iso/iso_catalogue/catalogue_ics/catalogue_detail_ics.htm?csnumber=3803.
- ISO., 2001. Textiles-tests for colour fastness-part X12: Colour fastness to rubbing. ISO 105-X12, International Organization for Standardization, Geneva, Switzerland. http://www.iso.org/iso/iso_catalogue/catalogue_tc/catalogue_detail.htm?csnumber=32339.
- Karci, F., 2005. Synthesis of disazo dyes derived from heterocyclic components. *Coloration Technol.*, 121: 275-280.
- Koh, J., S. Kim and J.P. Kim, 2004. Synthesis and spectral properties of azohydroxypyridone disperse dyes containing a fluorosulphonyl group. *Color. Technol.*, 120: 241-246.
- Lee, J.J., W.J. Lee, J.H. Choi and J.P. Kim, 2005. Synthesis and application of temporarily solubilised azo disperse dyes containing β -sulphatoethylsulphonyl group. *Dyes Pigments*, 65: 75-81.
- Metwally, M.A., S. Bondock, E.S.I. El-Desouky and M.M. Abdou, 2013. A facile synthesis and tautomeric structure of novel 4-arylhydrazono-3-(2-hydroxyphenyl)-2-pyrazolin-5-ones and their application as disperse dyes. *Coloration Technol.*, 129: 418-424.
- Nkeonye, P.O., 1987. Fundamental Principles of Textile Dyeing, Printing and Finishing. Ahmadu Bello University Press, Zaria, pp: 197-265.
- Oner, E., Y. Buyukakinci and N. Sokmen, 2013. Microwave-assisted dyeing of poly(butylene terephthalate) fabrics with disperse dyes. *Coloration Technol.*, 129: 125-130.
- Ortega-Luoni, P., L. Vera, C. Astudillo, M. Guzman and P. Ortega-Lopez, 2007. Synthesis of metallic azoderivatives of 2-amino-5-mercapto-1,3,4-thiadiazole. *J. Chilean Chem. Soc.*, 52: 1120-1122.
- Otutu, J.O., D. Okoro and E.K. Ossai, 2008. Preparation of dis-azo dyes derived from p-aminophenol and their fastness properties for synthetic polymer-fibres. *J. Applied Sci.*, 8: 334-339.
- Otutu, J.O., 2012. Synthesis of halogenated disazo disperse dyes derived from 2,4-dichloroaniline and 3-aminophenol. *Curr. Res. Chem.*, 4: 119-127.
- Otutu, J.O. and E. Osabohien, 2013. Synthesis and absorption spectra of monoazo dyes derived from 2-methoxy-5-nitroaniline. *Asian J. Mater. Sci.*, 5: 1-8.
- Otutu, J.O., E.M. Efurhievwe and S.U. Ameru, 2014. Synthesis and application of disazo dyes derived from 2-amino-5-mercapto-1,3,4-thiadiazole and 2-chloroaniline on acrylic fabric and nylon 66 fabric. *Curr. Res. Chem.*, 6: 1-9.
- Paula, Y.B., 1995. Organic Chemistry. 1st Edn., Prentice Hall Inc., New York, pp: 658-659.
- Shuttleworth, L. and M.A. Weaver, 1990. Dyes for Polyester Fibres. In: The Chemistry and Application of Dyes, David, R.W. and H. Geoffery (Eds.). Chapter 4, Plenum Press, New York, London, pp: 107-163.
- Towns, A.D., 1999. Developments in azo disperse dyes derived from heterocyclic diazo components. *Dyes Pigm.*, 42: 3-28.
- Ukponmwan, D.O., A. Osaze-Amayaevbo and A.P. Oviave, 1999. Mono-azo disperse dyes derived from 4-amino-3-nitrotoluene and 4-amino phenol. *J. Chem. Soc. Pak.*, 21: 179-183.