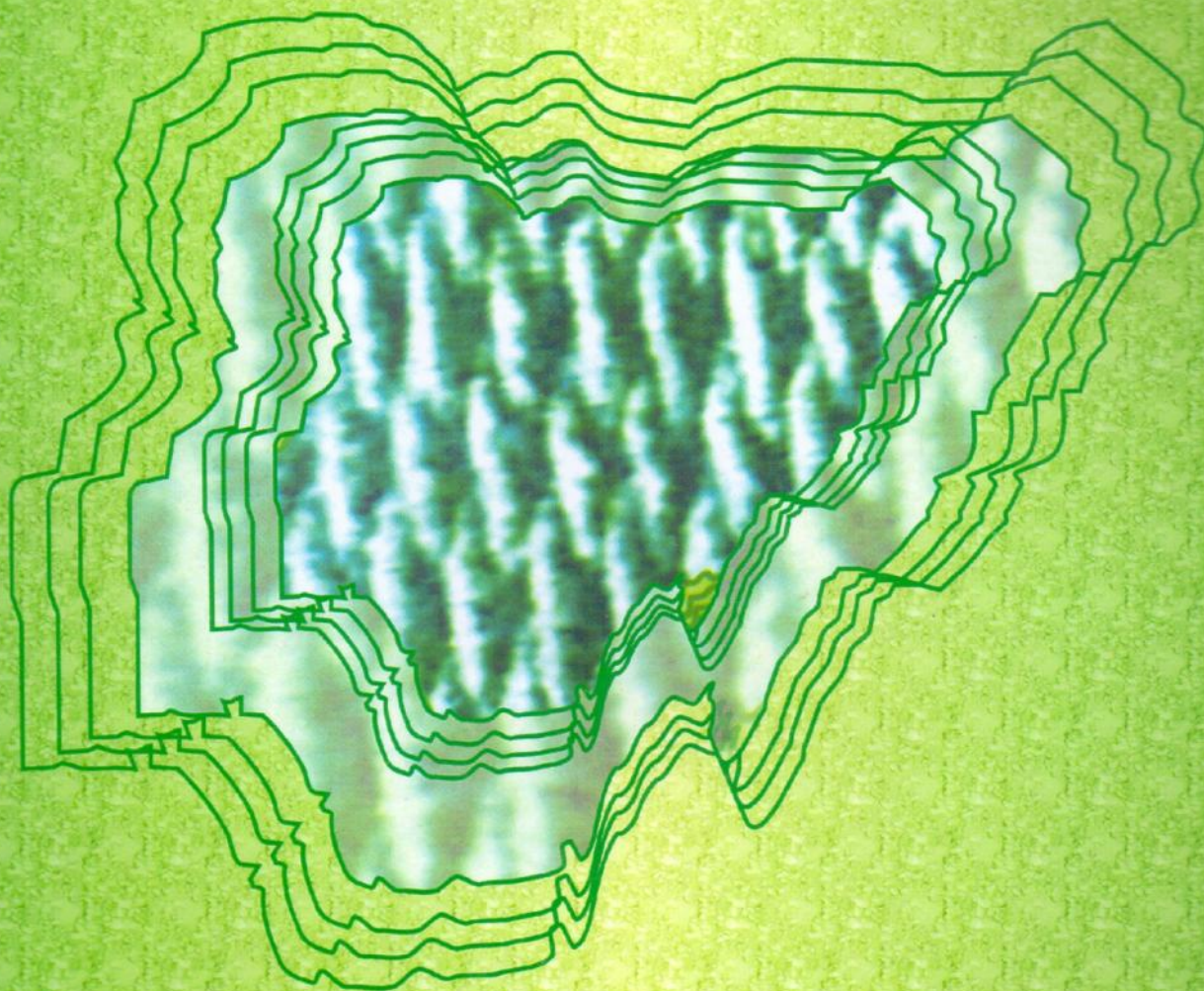


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## Table of Contents

	<b>Cover Page</b>	i
	<b>Editorial Board</b>	ii
	<b>Table of Contents</b>	iii
1	Synthesis and Application of Monoazo Disperse Dyes Derived from 4-amino- <i>N</i> -hexadecyl-1, 8-naphthalimide and their Dyeing Performance on Polyester Fabrics <b>U. S. Ameuru, M. K. Yakubu, K. A. Bello, P. O. Nkeonye &amp; A. Z. Halimehjani</b>	1 – 10
2	Effect of Coupling Agent on the Mechanical Properties of High-Density Polyethylene-Filled Calabash Particles Composites <b>A. B. Musa, J. BabaAli, A. Danladi, A. S. Lawal &amp; P. A. P. Mamza</b>	11 – 16
3	Industrial Design in Nigeria: A Mechanism for Expanding the Roles of Industrial Sector as Empowerment for Youth and National Development <b>Alesagba Ebenezer Igbekele, Yusuf Onipe John &amp; Olorunrayetan Titilayo Lizzy</b>	17 – 22
4	Service Properties of Selected Locally Manufactured Carpets <b>Ukanah P.S., Fashola K.O., Ameuru S.U., Gadimoh S. &amp; Salisu Z.M.</b>	23 – 27
5	Synthesis and Application of Monoazo Acid Dyes Derived from 5-amino-4-cyano-2-methyl- <i>N</i> -phenylthiophene-3-carboxamide on Chrome Tanned Leather and Nylon 6,6 Substrate <b>Magaji I.Y., Giwa A., &amp; Bello K. A</b>	28 – 36
6	Information Communication Technology and Dress Culture among Senior Secondary School Students in Osun State, Nigeria <b>Diyaolu I. J., Obayomi E. O. &amp; Bamidele T. A.</b>	37 – 41
7	Study of the Adsorption of Anionic Dye onto Activated Bone Char <b>Jibril A., Bukhari M. M., Giwa A. &amp; Ibrahim H. D.</b>	42 – 46
8	Evaluation of the Mechanical Properties of Chemically Modified Cow Hair Fibres Filled Recycled Low-Density Polyethylene Composites <b>Muktari S., Ishiaku U.S. &amp; Lawal A.S.</b>	47 – 53
9	Dyeing Performance of Dyes Derived from 2-Amino Heterocycles on Leather <b>O.R.A. Iyun, K.A. Bello, J.D. Putshaka, S. F. Tanko &amp; S. Abdullahi</b>	54 – 57
10	Production and Investigation of the Properties of Non-Disposable Diaper in Comparison with Disposable Diapers <b>Alebiosu S.O., Akindiya I.O., Adekeye T.O., Muhammed-Raji A. &amp; Oseghale H.E.</b>	58 – 61
11	Dyeing of Polyester with Pristine Multiwalled Carbon Nanotubes <b>E.B. Iliya &amp; R. Kotek</b>	62 – 66
12	Effect of Filler Particle Size on the Mechanical Properties of Waste Polypropylene/ Date Seed Particulate Composites <b>N. Lawal, A. Danladi, B. M. Dauda &amp; A. A. Kogo</b>	67 – 72
	Author's Guide	73 – 74







## Synthesis and Application of Monoazo Disperse Dyes Derived from 4-amino-*N*-hexadecyl-1, 8-naphthalimide and their Dyeing Performance on Polyester Fabrics

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### ABSTRACT

*A series of monoazo disperse dyes were synthesized using 4-amino-*N*-hexadecyl-1, 8-naphthalimide by coupling with different substituted electron rich arenes in appropriate reaction conditions. The synthesized diazo component and the dyes were purified by recrystallization method. The structures of the diazo component and the dyes were confirmed using UV-visible, FT-IR, NMR and MS spectroscopic techniques. Spectrophotometric investigations of the dyes in solvents of different polarities were carried out in order to obtain their absorption maxima, molar extinction coefficient and solvatochromic effects. These dyes were applied on polyester fabrics using High Temperature and High Pressure (HTHP) exhaust dyeing method. The dyeing characteristics such as build up on fabrics, wash, perspiration and light fastness were examined. The dyed fabric generally gave deep and bright hue ranging from light yellow, orange, maroon and neon red. The colour fastness of the dyed fabrics to washing and perspiration were examined to be very good, however, the light fastness was found to be fair to good.*

**Keywords:** Disperse dyes, 4-amino-*N*-hexadecyl-1, 8-naphthalimide, Solvatochromism, Dyeing, Polyester fabrics, Fastness properties

### INTRODUCTION

Azo dyes account for more than half of the total number of disperse dyes and cover nearly the full colour spectrum from greenish yellow to bluish green [1, 2]. They are relatively inexpensive and generally do not require numerous intermediate stages in their manufacture [2]. Disperse dyes are substantially water-insoluble dye having substantivity for hydrophobic fibres and usually applied from aqueous dispersion [3]. Disperse dyes are used mainly for dyeing polyester, secondary cellulose acetate and cellulose triacetate but they are also used in limited application for dyeing nylon and acrylic fibre [4]. The majority of disperse dyes are monoazo (50%) or anthraquinone dyes (25%) [5]. The remaining dyes are based on other dye chromophores such as benzodifuranone, coumarin, methane, naphthalimide, nitrophenylamine and quinophthalone.

Heterocyclic derivatives of naphthalimides, phenylazophthalimides and 1, 8-naphthalic anhydrides have been studied to assess their role as intermediates for dye synthesis [6]. These compounds have been investigated for the synthesis of disperse dyes, dyes for other polymer fibres, acid

dyes, dyes for polymeric materials and dyes capable of copolymerization. It has been shown in the last decade that the use of diazo components such as 4-aminonaphthalimides have advantageous effects on the dyeing properties of the derived disperse azo dyes. The presence of the naphthalimide system also leads to a deep and intense colour [7]. 4-Amino-*N*-substituted-naphthalimide is a useful diazo component for the synthesis of azo dyes [8]. Naphthalimide derivatives have obtained a great industrial importance because of their excellent fastness of some of these dyes [9]. When substituted with electron donating groups, naphthalimides are useful for colour chemists, preferably in the 4-position. The substitution of an amino group into the 4-position yields brilliant greenish yellow compounds [10]. 4-Amino-*N*-(substituted)-1, 8-naphthalimides were used for synthesis of azo and non-azonaphthalimide dyes [11]. The range of applications for these dyes are very wide; alongside classical fields such as dyeing natural and synthetic textile fibres, plastics, paints and coatings, increasingly novel applications are coming to light, in which aesthetic considerations are no longer of primary importance. These include applications in

solar energy collectors, light emitting diodes, liquid crystal displays, and laser active media, potential photosensitive biologically active units, as fluorescent markers in biology and in medicine as antiviral compounds or as analgesics [12]. Aminonaphthalimides are known to produce fluorescent yellow dyes for synthetic polymer fibres. In these compounds, when the *N*-substituent of naphthalimide is an aryl, most of the dyes exhibit properties similar to typical organic colorants, due to strong aggregation. The dyes with alkyl group in *N*-substituted naphthalimides can be converted into disperse dyes which are used in the colouration of synthetic fibres [13]. Generally, monoazo disperse dyes derived from naphthalimides show exceptionally good thermal stability and good dyeing fastness properties [14]. However, the 4-amino-*N*-alkyl (ester)-1, 8-naphthalimide derivatives of monoazo disperse dyes have relatively low heat fastness properties. Moreover, lack of polar groups in their structures made them hardly soluble in water which results to the milling of the dye into fine dispersion during application. This work aims to introduce amphiphiles into 4-aminonaphthalimide compound which serve as a diazo component to synthesize dyes with improved solubility, penetration, levelness and also, save time of milling which is associated to the conventional method of disperse dyes application.

## EXPERIMENTAL

### MATERIALS AND METHODS

All the chemicals used in the synthesis were of commercial grade and were used as received. The synthesized compounds were characterized by melting point, TLC, FT-IR, <sup>1</sup>H and <sup>13</sup>C NMR, MS and elemental analysis (CHN). Melting points were recorded on Barnstead electrothermal 9200 apparatus and are uncorrected. Infra-red were recorded using Perkin-Elmer Spectrum RX1 FT-IR Spectrometer, <sup>1</sup>H and <sup>13</sup>C NMR were carried out on 300 MHz Bruker instrument using deuterated chloroform (CDCl<sub>3</sub>) and dimethylsulphoxide (DMSO-*d*<sub>6</sub>) as solvent. Chemical shifts were reported in parts per million (ppm) downfield from internal tetramethylsilane (TMS). Elemental analyses (CHN) were recorded on Perkin-Elmer PE 2400 series II and mass spectra were reported using Agilent Technologies 5975C VL MSD mass spectrometer.

#### Synthesis of 5-Nitroacenaphthene

Acenaphthene **1** (15.4 g, 0.1 mol) was dissolved in hot acetic acid (57.2 cm<sup>3</sup>) and was cooled with vigorous stirring to separate crystalline acenaphthene as fine as possible. Concentrated nitric acid (65%, *d* = 1.40 g/cm<sup>3</sup>) (9.84 g) was added

drop wise for 1 h at 22-27 °C, then further stirred for 1 h at 25 °C. The solution was warmed gradually for 30 min to 68 °C then cooled to form the crystals which were filtered, washed with 1:1 acetic acid – water and water to give yellow needles 5-nitroacenaphthene (**2**) [15], yield 85 %, Mp: 101-102 °C.

#### Synthesis of 4-nitro-1, 8-naphthalic anhydride

5-Nitroacenaphthene (24.87 g, 0.125 mol) was dissolved in hot acetic acid (248.75 cm<sup>3</sup>), sodium dichromate (158.5 g) was added for 3 hours at 65 – 70 °C. The solution was warmed gradually to 98 – 100 °C for 30 min and further refluxed for 5 h. The reaction mixture was washed out from the flask with hot water (0.6 L), cooled, filtered and the solid was washed with dilute HCl. In addition, the solid was boiled with 200 cm<sup>3</sup> of 5 % Na<sub>2</sub>CO<sub>3</sub> solution for 30 min and filtered. The filtrate was acidified and the separated crystals dried at 120 °C for 4 hours to obtain 4-nitro-1, 8-naphthalic anhydride (**3**), which was recrystallized from concentrated HNO<sub>3</sub> (65%, *d* = 1.40 g/cm<sup>3</sup>) to afford colourless needles [16], yield 74%, Mp: 231-232 °C.

#### Synthesis of 4-nitro-*N*-hexadecyl-1, 8-naphthalimide

A suspension of 4-nitro-1, 8-naphthalic anhydride (0.02 mol, 4.86 g) and hexadecylamine (0.03 mol, 7.17 g) was stirred under reflux for 7 h in ethanol (102 cm<sup>3</sup>). The cooled suspension was filtered out and recrystallized with ethanol to give off-white crystals of 4-nitro-*N*-hexadecyl-1, 8-naphthalimide (**4**) [17], yield 64.6 %; Mp: 99-101 °C.

#### Synthesis of 4-amino *N*-hexadecyl -1, 8-naphthalimide

A mixture of 4-nitro-*N*-hexadecyl-1, 8-naphthalimide (18 mmol, 8.39 g) and stannous chloride (90 mmol, 20.25 g) in ethanol (81 cm<sup>3</sup>) was refluxed for 1hr with hydrochloric acid (10.1 cm<sup>3</sup>) till the reaction was complete. The mixture was transferred into 100 cm<sup>3</sup> of water and the observed precipitate was filtered. The crude product (4-amino *N*-hexadecyl -1, 8-naphthalimide (**5**)) was purified by recrystallization from ethanol as orange crystals [17], yield 71 %; Mp: 138-139 °C;

#### Representative Procedure for the Synthesis of Dyes (7a-e)

#### Synthesis of (Z)-2-hexadecyl-6-((2-hydroxynaphthalen-1-yl)diazonyl)-benzo[de]isoquinoline-1,3(2*H*)-dione (7a)

Sodium nitrite (1.5 mmol, 0.104 g) was slowly added with constant stirring to cold concentrated sulphuric acid (98 %, 1.1 cm<sup>3</sup>) at below 10 °C. The

temperature of the reaction mixture was gradually increased to 65 °C using water bath until all the sodium nitrite was dissolved. The solution was then cooled externally to 5 °C and diluted with a mixture of propionic acid and acetic acid (10 cm<sup>3</sup> 1.5:8.5 v/v). The finely ground powder of 4-amino-*N*-hexadecyl-1, 8-naphthalimide (1.5 mmol) was added portion wise at less than 10 °C, and then the liquor was stirred for 3 h. The obtained clear diazonium salt solution (**6**) was used immediately in coupling reactions with 2-naphthol (1.5 mmol) dissolved in ethanol by adding over 30-40 min with vigorous stirring. The mixture was stirred further for 2 h, maintaining a temperature of 5 °C. The pH of the solution was adjusted to 4-5 using 10 % sodium acetate and stirred for 1 h. The resulting product was then collected by filtration, washed with warm water and then cold water and dried. The crude product was purified by recrystallization method using DMSO several times to obtained dye **7a**, Yield 71.1 %; Mp: 154–156 °C. The same procedure were used to produce dyes **7b – 7e**

#### Dyeing Procedure

The dyeing was carried out using KMS-IRE Laboratory Dyeing Machine – ELITE. The material to liquor ratio of 1:50 and 2 % shade was used for dyeing On Weight of the Fabric (OWF). The dye was dissolved in DMF (5 cm<sup>3</sup>) and water (45 cm<sup>3</sup>) was added with constant stirring, the pH was adjusted to 5 using glacial acetic acid. The dye suspension (50 cm<sup>3</sup>) was transferred into a beaker provided with a lid and a screw cap. Before closing the lid and tightening the metal cap over the beaker, a wetted pattern of polyester fabric was rolled into the beaker. The beaker was then placed vertically on the rotatory carrier inside the tank and the clamp plate was firmly tightened. The rotatory carrier was then allowed to rotate in the glycerin-bath and the temperature was raised to 130 °C at the rate of 2 °C per min. The dyeing was continued for 1h, under pressure. After cooling to 70 °C the beaker was removed from the bath, the dyed pattern was removed, thoroughly washed with water and dried at room temperature [17].

**Reduction Clearing:** The dyed fabric was treated with an aqueous solution containing sodium dithionite (2 g/L) and sodium hydroxide (2 g/L) for 45 min at 70 °C. The fabrics was then washed with water and dried.

**Fastness Properties:** The dyed fabrics were evaluated using a standard test methods, wash fastness (ISO 3), perspiration (ISO 105 E04:2013) and light fastness (ISO 105 B02:2014).

#### Synthesis of Intermediates and the Dyes

Acenaphthene **1** was reacted with concentrated nitric acid in an acetic acid media at room temperature to give yellow crystals of 5-nitroacenaphthene **2** in 85 % isolated yield. 5-Nitroacenaphthene **2** was oxidized using sodium dichromate and hot glacial acetic acid as the solvent to give colourless crystals of 4-nitro-1, 8-naphthalic anhydride **3** in 74 % isolated yield after recrystallization from nitric acid. 4-Nitro-1, 8-naphthalic anhydride **3** was reacted with hexadecylamine in absolute ethanol media under reflux condition to produce off-white crystals of 4-nitro-*N*-hexadecyl-1, 8-naphthalimide **4** in 64.6 % isolated yield. 4-Amino-*N*-hexadecyl-1, 8-naphthalimide **5** was synthesized by the reaction of 4-nitro-*N*-hexadecyl-1, 8-naphthalimide **4** with stannous chloride (II) in hydrochloric acid as a reducing agent and ethanol as solvent to give the corresponding orange crystals of **5** in 71 % isolated yield (Scheme 1). The structure of the intermediates were confirmed by various techniques which include FT-IR, NMR, elemental analysis (CHN) as shown in Tables 1 and 2, and mass spectroscopy. The IR spectra of the intermediate 4-Amino-*N*-hexadecyl-1, 8-naphthalimide **5** showed absorptions at 3352 and 3254 cm<sup>-1</sup> due to NH<sub>2</sub> stretch, the signal at 2998 cm<sup>-1</sup> is attributed to aromatic C-H stretch while the signal at 2917 indicates C-H stretch of aliphatic. The absorption at 1675 cm<sup>-1</sup> and 1653 cm<sup>-1</sup> are due to –N-C=O and C=O stretch respectively and the absorption band at 1636 cm<sup>-1</sup> corresponding to C=C groups. The <sup>1</sup>H NMR spectra displayed two signals at 0.86 and 1.24-1.77 ppm corresponds to the terminal methyl (-CH<sub>3</sub>) and the methylene (-CH<sub>2</sub>-) groups of the hexadecylamine chain respectively. The peaks at 4.11 ppm showed a downfield correspond to (-*N*-CH<sub>2</sub>-), the peak at 5.06 ppm was observed which account for the hydrogens associated with NH<sub>2</sub> substituent. The aromatic protons were showed signals at 6.86-8.57 ppm as multiplet. <sup>13</sup>C NMR spectrum showed 16 peaks at 14.1 and 22.6-40.2 ppm related to the terminal –CH<sub>3</sub> and –CH<sub>2</sub> groups of hexadecylamine carbons respectively. The peaks at 109.5-149.1 ppm correspond to the carbons at the aromatic region and the carbonyl groups appeared at 164.0 and 164.5 ppm. The elemental analysis results showed good agreement with the theory. The mass spectrometry analysis showed a molecular ion [M<sup>+</sup>] at m/z 436.4 which is in agreement with the formula mass of **5** as shown in Table 3.

The diazotisation of 4-amino-*N*-hexadecyl-1, 8-naphthalimide was carried out using nitrosylsulphuric acid method and coupling with various electron-rich arenes produce the dyes (Scheme 1). The structures of the dyes were

characterized using FT-IR, <sup>1</sup>H NMR, MS and UV spectroscopic methods. The FT-IR, <sup>1</sup>H NMR and UV- visible results were shown in the Table 3 and 4 respectively. Figures 1–4 showed the FT-IR and Mass spectra of the selected compounds **7a** and **7b**. The dye **7a** showed absorption band at 3351 cm<sup>-1</sup> due to the presence of OH group. All the dyes showed absorption bands at 3060-2953 cm<sup>-1</sup> and 2921-2917 cm<sup>-1</sup> due to the presence of C-H stretching vibration of aromatic C-H, methyl and methylene groups. The absorption band at 1657-1647 cm<sup>-1</sup> corresponding to C=O groups. The absorption band at 1646-1604 cm<sup>-1</sup> confirmed the presence of C=C groups. The signal at 1589-1575 cm<sup>-1</sup> is attributed to –N=N- stretch while signal at 1386-1351 cm<sup>-1</sup> is due to C-N. The <sup>1</sup>H NMR spectra of the dyes displayed signals at 0.84-0.86 and 1.23-1.76 ppm for the methyl and methylene protons of the aliphatic regions. The signal at 4.09-4.18 ppm

shows a downfield due to *N*-CH<sub>2</sub> protons of the aliphatic group. The diazo group was substituted in the alpha position of 2-naphthol in the dye **7a**. For this purpose, the dye **7a** showed signals at 17.07 ppm for OH proton in <sup>1</sup>H NMR spectra. This amount of deshielding can be attributed to the intermolecular hydrogen bonding of the diazo group and the –OH group. The aromatic protons were showed signals at 6.66 - 9.21 ppm and the number of protons is in agreement with the aromatic protons in the structure of dyes. The presence of two signals at 6.7 and 7.9 ppm as doublet in the <sup>1</sup>H NMR spectra of **7b** and **7c** exactly confirm that the diazo group substituted at para position on the *N*, *N*-(dialkyl)aniline ring. In addition mass analysis exactly confirms the molar mass of the dyes.

## RESULTS AND DISCUSSION

Table 1: The FT-IR, <sup>1</sup>H NMR and <sup>13</sup>C NMR Spectra of the Synthesized Intermediates

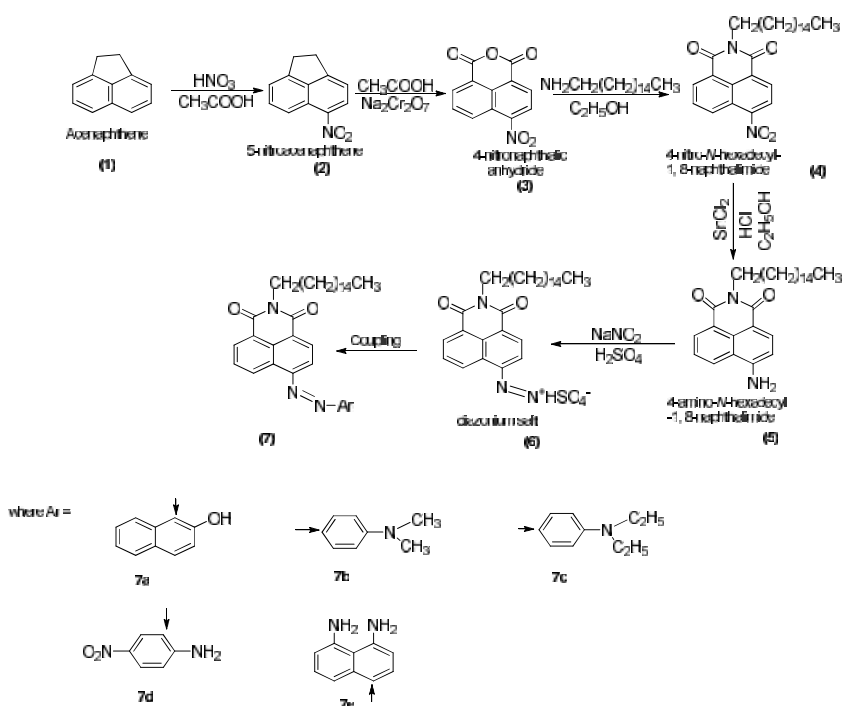
Compound	FT-IR (cm <sup>-1</sup> )	<sup>1</sup> H NMR and <sup>13</sup> C NMR ( ppm, CDCl <sub>3</sub> )
5-nitroacenaphthene (2)	3085 (C-H str. Ar.), 2946 (C-H str. Aliphatic), 1620, 1596 (NO <sub>2</sub> str.), 1508 (C=C str.)	3.43 (4H, s, 2CH <sub>2</sub> ), 7.31(1H, d, <i>J</i> = 7.8 Hz, 1-H), 7.45 (1H, t, <i>J</i> = 7.0 Hz, 9-H), 7.66 (1H, dd, <i>J</i> = 6.7 and 7.1 Hz, 2-H), 8.47 (1H, d, <i>J</i> = 7.7 Hz, 8-H), 8.54 (1H, d, <i>J</i> = 8.6 Hz, 3-H); 30.6, 33.1, 117.9, 120.1, 121.2, 122.7, 124.3, 127.7, 131.7, 140.1, 146.6, 155.7
4-nitro-1, 8-naphthalic anhydride (3)	3078 (C-H str. Ar.), 2914, 2849 (C-H str. Aliphatic) 1789.1756 (C=O str.), 1624 (NO <sub>2</sub> str.), 1526 C=C str.	( ppm, DMSO- <i>d</i> <sub>6</sub> ) 8.09 (1H, t, <i>J</i> = 7.8 Hz, 3-H), 8.54 (1H, d, <i>J</i> = 8.0 Hz, 8-H), 8.61 (1H, s, 2-H), 8.64 (1H, d, <i>J</i> = 7.5 Hz, 9-H), 8.73 (1H, dd, <i>J</i> = 8.2 and 8.7 Hz, 4-H); 120.1, 122.6, 124.0, 124.3, 129.8, 130.3, 130.6, 131.1, 133.2, 149.5, 159.4, 160.0.
4-nitro- <i>N</i> -hexadecyl-1, 8-naphthalimide (4)	3081 (C-H str.), 2947 2916 (C-H str. Aliphatic), 1699 (-N-C=O str.), 1656 (C=O str.), 1622 (C=C str.), 1593 (NO <sub>2</sub> str.)	0.86 (3H, t, <i>J</i> = 6.9 Hz, CH <sub>3</sub> ), 1.26-1.79 (m, 28H, Aliphatic), 4.16 (2H, t, <i>J</i> = 7.6 Hz, <i>N</i> -CH <sub>2</sub> ), 7.96 (1H, t, <i>J</i> = 7.3 Hz, 3-H), 8.40 (1H, d, <i>J</i> = 8.0 Hz, 8-H), 8.68 (1H, d, <i>J</i> = 5.7 Hz, 9-H), 8.75 (1H, dd, <i>J</i> = 8.0, 8.3 Hz, 2-H), 8.85 (1H dd, <i>J</i> = 8.7 and 8.8 Hz, 4-H); 14.1, 22.7, 27.1, 28.0, 29.3, 29.4, 29.4, 29.4, 29.5, 29.5, 29.6, 29.7, 29.7, 31.9, 40.9, 123.1, 123.7, 123.9, 127.1, 129.1, 129.2, 129.7, 129.9, 132.4, 149.5, 162.4, 163.3.
4-amino <i>N</i> -hexadecyl -1, 8- naphthalimide (5)	3254 (N-H str.), 2998 (C-H str. Ar.), 2917, 2851 (C-H str. Aliphatic), 1675 (-N-C=O str.), 1653 (C=O str.), 1636 (C=C str.)	0.86 (3H, t, <i>J</i> = 6.9 Hz, CH <sub>3</sub> ), 1.24-1.77 (m, 28H, Aliphatic), 4.13 (2H, t, <i>J</i> = 7.6 Hz, <i>N</i> -CH <sub>2</sub> ), 5.06 (2H, s, NH <sub>2</sub> ), 6.86 (1H, d, <i>J</i> = 8.2 Hz, 8-H), 7.60 (1H, t, <i>J</i> = 7.8 Hz, 2-H), 8.11 (1H, d, <i>J</i> = 8.2 Hz, 9-H), 8.38 (1H, d, <i>J</i> = 8.2 Hz, 3-H), 8.57 (1H, d, <i>J</i> = 7.2 1-H); 14.1, 22.7, 27.2, 28.2, 29.3, 29.4, 29.4, 29.4, 29.4, 29.5, 29.5, 29.6, 29.7, 31.9, 40.3, 109.5, 112.2, 120.1, 123.2, 124.9, 126.7, 129.8, 131.4, 133.7, 149.1, 164.0, 164.5

Table 2: The Yield, Melting Point and Elemental Analysis of the Synthesized Intermediates

Compound	Yield (%)	Melting point (°C)	Found (%)			Required (%)		
			C	H	N	C	H	N
5-nitroacenaphthene (2)	85	101-102	72.18	4.57	7.04	72.3	5.45	7.03
4-nitro-1, 8-naphthalic anhydride (3)	74	231-232	59.17	2.02	5.75	59.27	2.07	5.76
4-nitro-N-hexadecyl-1, 8-naphthalimide (4)	64.6	99-101	72.39	8.79	5.91	72.07	8.21	6.00
4-amino N-hexadecyl -1, 8-naphthalimide (5)	71	138-139	76.91	9.33	6.23	77.02	9.23	6.42

Table 3: The FT-IR and <sup>1</sup>H NMR Spectra of the Synthesized Dyes

Dyes	FT-IR (cm <sup>-1</sup> )	<sup>1</sup> H NMR (ppm, CDCl <sub>3</sub> )
7a	3351 (OH str.), 3060, 2955 (C-H str.), 2920, 2849 (C-H str. Aliphatic), 1701 (-N-C=O str.), 1655 (C=O str.), 1623 (C=C str.), 1587 (N=N str.), 1382 (C-N str.)	0.85 (3H, t, J = 6.8 Hz, CH <sub>3</sub> ), 1.25–1.72 (28H, m, Aliphatic), 4.09 (2H, t, J = 7.7 Hz, N-CH <sub>2</sub> ), 6.66–8.59 (11H, m, Aromatic), 17.07 (1H, s, OH)
7b	2955 (C-H str.), 2917, 2849 (C-H str. Aliphatic), 1693 (-N-C=O str.), 1657 (C=O str.), 1604 (C=C str.), 1586 (N=N str.), 1351 (C-N str.)	0.85 (3H, t, J = 6.9 Hz, CH <sub>3</sub> ), 1.25–1.74 (28H, m, Aliphatic), 3.15 (3H, s, CH <sub>3</sub> ), 4.15 (2H, t, J = 7.6 Hz, N-CH <sub>2</sub> ), 6.76–9.21 (9H, m, Aromatic)
7c	2954 (C-H str.), 2917, 2850 (C-H str. Aliphatic), 1694 (-N-C=O str.), 1675 (C=O str.), 1636 (C=C str.), 1575 (N=N str.), 1379 (C-N str.)	0.85 (3H, t, J = 6.9 Hz, CH <sub>3</sub> ), 1.25–1.74 (28H, m, Aliphatic), 3.48 (4H q, J = 7.1 Hz, 2CH <sub>2</sub> ), 4.15 (2H, t, J = 7.5 Hz, N-CH <sub>2</sub> ), 6.75–9.21 (9H, m, Aromatic)
7d	3208 (NH str.), 2953 (C-H str.), 2921, 2850 (C-H str. Aliphatic), 1699 (-N-C=O str.), 1647 (C=O str.), 1589 (N=N str.), 1382 (C-N str.)	0.84 (3H, t, J = 7.0 Hz, CH <sub>3</sub> ), 1.23–1.73 (28H, m, Aliphatic), 4.13 (2H, t, J = 7.4 Hz, N-CH <sub>2</sub> ), 7.54–8.74 (9H, m, Aromatic)
7e	3353 (NH str.), 2953 (C-H str.), 2919, 2850 (C-H str. Aliphatic), 1696 (-N-C=O str.), 1614 (C=O str.), 1575 (N=N str.), 1379 (C-N str.)	0.85 (3H, t, J = 6.9 Hz, CH <sub>3</sub> ), 1.24–1.72 (28H, m, Aliphatic), 4.14 (2H, t, J = 7.6 Hz, N-CH <sub>2</sub> ), 5.02 (2H, s, NH <sub>2</sub> ), 6.85–9.08 (11H, m, Aromatic)



Scheme 1: Synthesis of the Intermediates and the Dyes

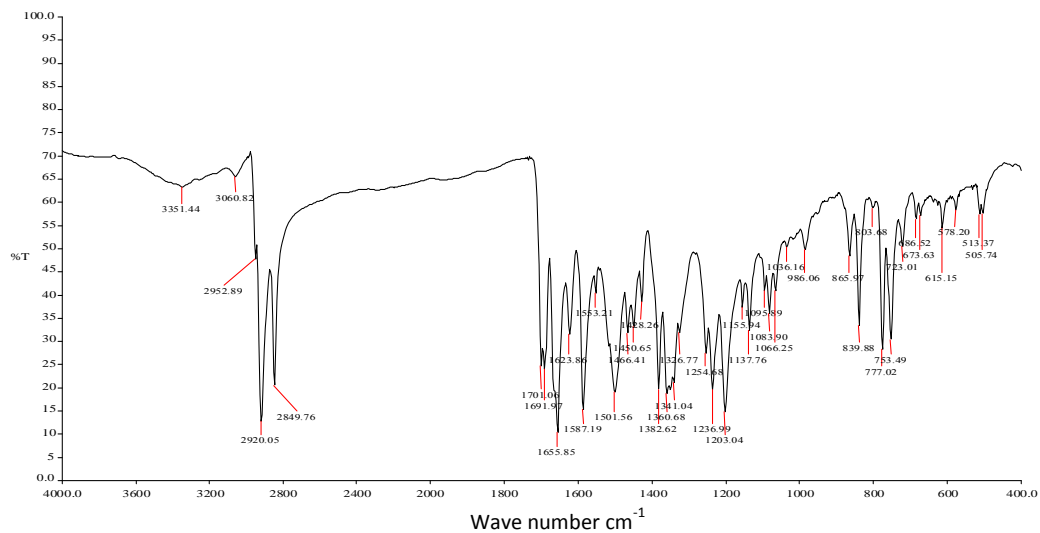


Figure 1: FT-IR for the dye 7a

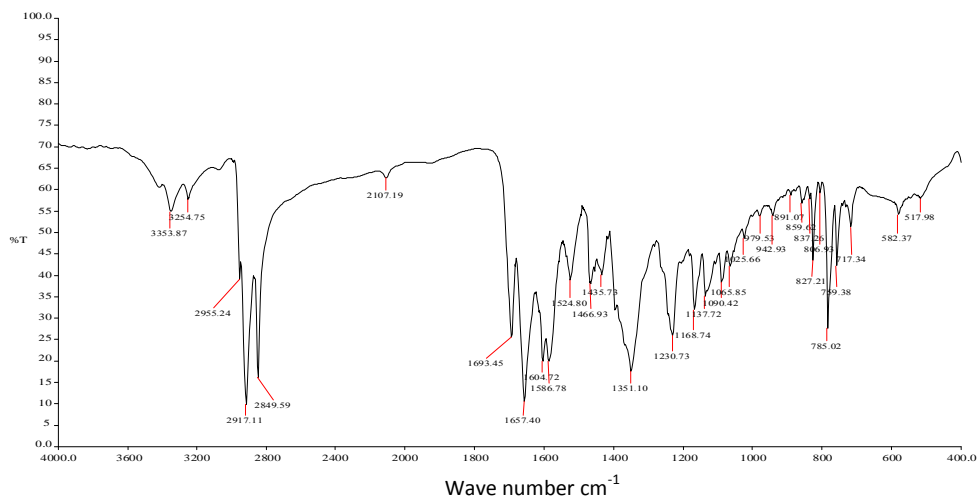


Figure 2: FT-IR for dye 7b

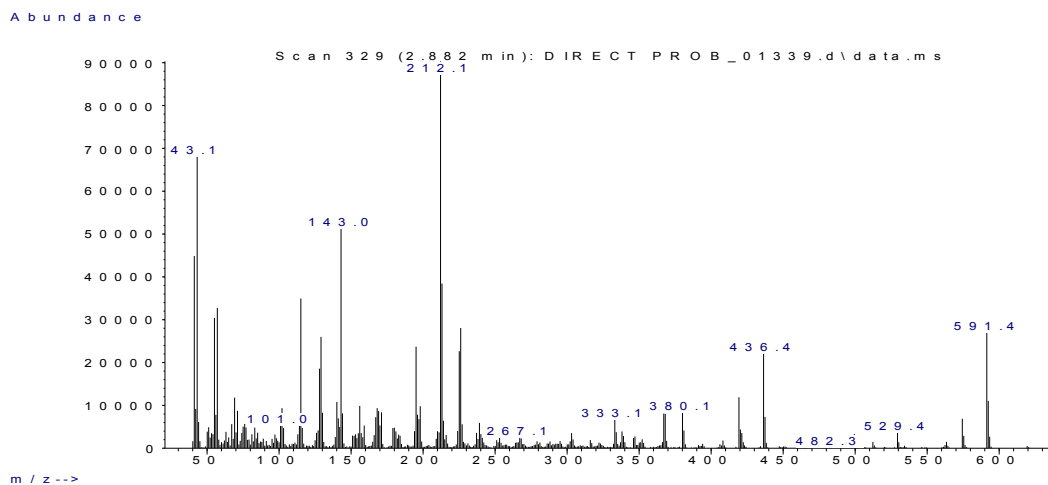


Figure 3: Mass Spectra of the dye 7a

Abundance

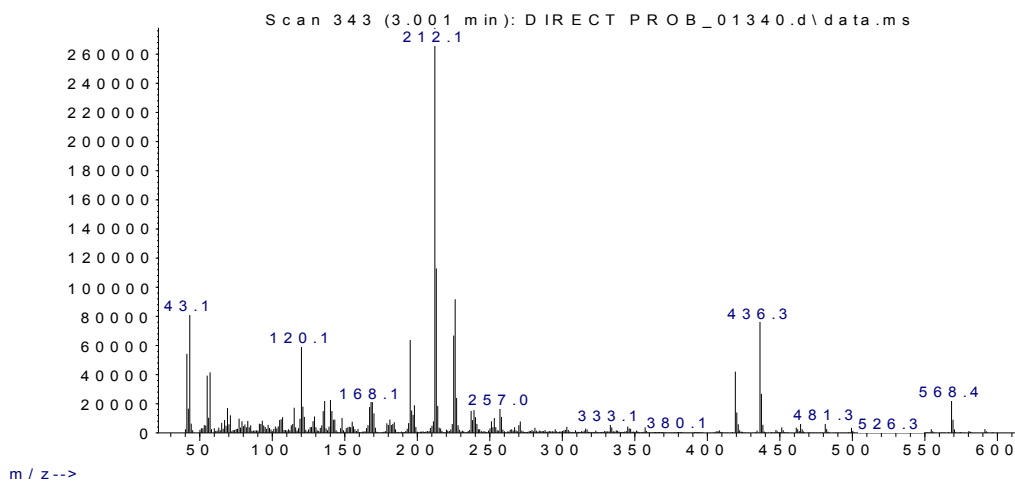


Figure 4: Mass Spectra of the dye 7b

### Spectral Characteristics of the Synthesized Dyes

The absorption spectra of the synthesized dyes were measured in various solvents. Absorption maxima, molar extinction coefficients of each dye in various solvents are listed in Table 4. Wavelengths of maximum absorption ( $\lambda_{max}$ ) and molar extinction coefficient ranged from 440-610 nm and  $1.06 \times 10^4$ - $3.18 \times 10^4$  l/mol cm for the synthesized dyes in DMF respectively. The wavelength ( $\lambda_{max}$ ) shifts in different solvents are brought about by the solvatochromic effect resulting from changes in the dielectric constant of the solvent. In many dyes, however, the ground state is less polar than the excited state, thus a polar solvent will tend to stabilize the excited state more than the ground state, giving rise to bathochromic shift [18]. It was observed that in chloroform, ethanol and ethanol plus a drop of HCl, the absorption spectral of the dyes did not change significantly.

Dye **7a** which was obtained by diazotised 4-amino-*N*-hexadecyl-1, 8-naphthalimide and coupled with 2-naphthol gave maximum absorption wavelength of 520 nm in DMF and when the coupling component was changed to *N,N*-dimethylaniline give dye **7b**, the resulting dye gave maximum absorption wavelength of 540 nm in the same solvent. It is well known that the colour of dyes is influenced by the introduction of additional donor groups into the coupling component. The presence of *N,N*-dimethyl in the coupling component of dye **7b** resulted in the bathochromic effect of 20 nm when compared with dye **7a** which may attribute to the present of dimethyl. Dye **7c** was obtained by coupling *N,N*-diethylaniline with the same amine gave a maximum absorption wavelength of 440 nm

in DMF and is hypsochromic by 80 nm and 100 nm when compared with dyes **7a** and **7b** respectively in the same solvent. Replacement of coupling component (*N,N*-diethylaniline) in dye **7c** with 4-nitroaniline gave dye **7d** which absorbed at 440 nm and showed a bathochromic shift of 100 nm and 0 nm when compared with dyes **7b** and **7c** respectively. Similarly, dye **7e** was obtained by coupling 1, 8-diaminonaphthalene with the diazo component. The dye **7e** showed a strong bathochromic shift of 90 nm when compared with dye **7a** which may be attributed to the presence of electron donor ( $\text{NH}_2$ ) in the coupling component.

### Dyeing and Fastness Properties of the Synthesized Dyes

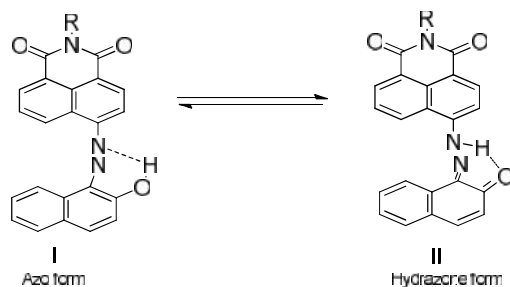
The synthesized dyes were applied onto polyester fabrics and their dyeing properties such as light fastness, wash fastness and perspiration fastness were examined. The dyes were applied by the high temperature and high pressure dyeing method. Visual evaluation of the dyeing on polyester fabrics showed that the nature of the alkyl residue in the amine group of the coupling component affects both the intensity of dyeing and their shade obtained. The highest intensity is shown by the dyes which contain *N,N*-dimethyl and *N,N*-diethyl groups. These substituents bring about an increase in the nucleophilic character of the tertiary nitrogen atom and the stability of polar forms [19] especially when there are no substituents in the phenyl ring causing steric effects. The electron donor substituent (-OH) in the ring of the coupling components in ortho position to the azo band also considerably affects the colour of the dyeing in **7a**. This phenomenon is described in terms of structures I and II.

Table 3: Mass Spectroscopy of the Intermediate and the Synthesized Dyes

Dye No.	Empirical Formula	Experimental Values of m/z Fragment	Corresponding Positive Charge Fragment	Theoretical Value
(5)	C <sub>28</sub> H <sub>40</sub> N <sub>2</sub> O <sub>2</sub>	43.2, 212.2, 295.1, 391.3, 436.4	CNO <sup>+</sup> , C <sub>12</sub> H <sub>7</sub> N <sub>2</sub> O <sub>2</sub> <sup>+</sup> , C <sub>18</sub> H <sub>33</sub> NO <sub>2</sub> <sup>+</sup> , C <sub>27</sub> H <sub>38</sub> NO <sup>+</sup> , M	436.63
7a	C <sub>38</sub> H <sub>45</sub> N <sub>3</sub> O <sub>3</sub>	43.1, 143.0, 212.1, 436.4, 591.4	CNO <sup>+</sup> , C <sub>10</sub> H <sub>7</sub> O <sup>+</sup> , C <sub>12</sub> H <sub>7</sub> N <sub>2</sub> O <sub>2</sub> <sup>+</sup> , C <sub>28</sub> H <sub>40</sub> N <sub>2</sub> O <sub>2</sub> <sup>+</sup> , M	591.78
7b	C <sub>36</sub> H <sub>48</sub> N <sub>4</sub> O <sub>2</sub>	43.1, 120.1, 212.1, 436.3, 568.4	CNO <sup>+</sup> , C <sub>8</sub> H <sub>10</sub> N <sup>+</sup> , C <sub>12</sub> H <sub>7</sub> N <sub>2</sub> O <sub>2</sub> <sup>+</sup> , C <sub>28</sub> H <sub>40</sub> N <sub>2</sub> O <sub>2</sub> <sup>+</sup> , M	568.79
7c	C <sub>38</sub> H <sub>52</sub> N <sub>4</sub> O <sub>2</sub>	43.1, 212.1, 596.5	CNO <sup>+</sup> , C <sub>12</sub> H <sub>7</sub> N <sub>2</sub> O <sub>2</sub> <sup>+</sup> , M	596.85
7d	C <sub>34</sub> H <sub>43</sub> N <sub>5</sub> O <sub>4</sub>	43.1, 212.2, 436.4, 586.4	CNO <sup>+</sup> , C <sub>12</sub> H <sub>7</sub> N <sub>2</sub> O <sub>2</sub> <sup>+</sup> , C <sub>28</sub> H <sub>40</sub> N <sub>2</sub> O <sub>2</sub> <sup>+</sup> , M	585.74
7e	C <sub>38</sub> H <sub>47</sub> N <sub>5</sub> O <sub>2</sub>	43.2, 212.2, 436.4, 605.4	CNO <sup>+</sup> , C <sub>12</sub> H <sub>7</sub> N <sub>2</sub> O <sub>2</sub> <sup>+</sup> , C <sub>28</sub> H <sub>40</sub> N <sub>2</sub> O <sub>2</sub> <sup>+</sup> , M	605.81

Table 4: The UV- Visible Spectroscopic of the Dyes

Dye No.	λ <sub>max</sub> in DMF ×10 <sup>4</sup> l mol <sup>-1</sup> cm <sup>-1</sup>	DMF	Chloroform	Ethanol	Ethanol + HCl	Change in
		λ <sub>max</sub> (nm)	λ <sub>max</sub> (nm)	λ <sub>max</sub> (nm) a	λ <sub>max</sub> (nm) b	λ <sub>max</sub> (nm) (b-a)
7a	3.06	520	465	520	520	0
7b	1.06	540	440	440	440	0
7c	1.76	440	460	440	435	-5
7d	3.18	440	410	435	435	0
7e	2.35	610	520	430	440	+10



Scheme 2: Structure of Azo form and Hydrazone form

Table 5: Dyeing Properties of the Synthesized Dyes on Polyester Fabrics

Dye No.	Light	Fastness Washing	Perspiration	
			Acid	Alkaline
7a	2	3	3	3
7b	3	3	4	4
7c	6	5	4	5
7d	2	4	4	4
7e	3	4	3	4

The fastness properties results are summarized in Table 5, the dyes gave good levelness and fibre penetration on polyester. The wash fastness rating is good to excellent with rating of 3-5. The good wash fastness obtained on polyester is due to the crystalline structure of the polyester which disallowed the migration of dye out of the fabric when this has entered the fabric. The dye 7c gave very good light fastness rating of 6 whereas other have poor to moderate light fastness rating as

shown in Table 2. Disperse dyes have a tendency to deposit on the fibre surface during dyeing as well as during cooling [20] and therefore in order to obtain a satisfactory combination of light and sublimation fastness, a judicious choice of substituent is required. The fading of azo dyes is mainly a consequence of decomposition of the –N=N– moiety either by oxidation, reduction or photolysis. The rates of these processes should be sensitive to the chemical structure of the dye, the



type of substrate and treatment conditions [21]. The dyed substrate employed in this study is polyester fabric, the fading process likely occur by oxidation [22]. The ease of oxidation of azo linkages should be a function of electron density. Therefore, electron donating substituents on this moiety should increase the fading rate while electron withdrawing groups should decrease the rate. This is in agreement with the observed results (Table 5) which demonstrate that the presence of a hydroxyl group in the dye **7a** causes decrease of light fastness to 2. The introduction of polar groups can adversely affect the dyeing properties of a dye but the hydroxyalkyl group appears to be particularly useful in increasing polarity without excessive undesirable side effects. The free hydroxyl group and also short alkyl chain favour formation of compact aggregates of dyes, which confers low surface activity and this causes dye to spread as multilayer on the surface of the fabric; the multilayer is less susceptible to fading. This may account for why the dyes from *N,N*-dimethylaniline and *N,N*-diethylaniline have acceptable results in light fastness.

## CONCLUSION

A series of monoazo disperse dyes derived from amphiphilic naphthalimide have been synthesized in good yields. The structures of the intermediate and the dyes were confirmed using elemental analysis (CHN), FTIR, <sup>1</sup>H-NMR, <sup>13</sup>C NMR, UV-visible, and mass spectroscopic techniques. Spectrophotometric investigations of the synthesized dyes in solvents of different polarities were determined in order to obtain their absorption maxima, molar extinction coefficients and solvatochromic effects. The dyes were applied on polyester fabrics using high temperature dyeing method without addition of dispersing agent and their fastness properties were examined. The wash and perspiration fastness properties were generally very good. Dyes **7a** and **7b** showed poor light fastness whereas, dyes **7b**, **7e** and **7c** showed a moderate to very good respectively.

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## **Effect of Coupling Agent on the Mechanical Properties of High-Density Polyethylene-Filled Calabash Particles Composites**

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### **ABSTRACT**

In the interest of sustainable development and production of eco-friendly bio-composites, the use of natural fillers with synthetic resins in the production of composites has been the new trend in composite manufacture. In this study, calabash particles (CP) were used as filler in high density polyethylene (HDPE) to produce bio-composites. The calabash which was grinded and sieved into 125 $\mu$ m was introduced at 10% variation up to 50% filler loading followed by compounding and thereafter compression moulded. Two types of composites were prepared, in one, Maleic Anhydride (MA) as coupling agent was introduced at 3% on the weight of the filler together with an initiator Benzoyl Peroxide (BP) at 1% to enhance the interfacial adhesion between the CP and HDPE and in another no coupling agent was used. The result of tensile, flexural, impact strength and tensile modulus shows increase from 0 to 40% filler loading of composites with coupling agent representing 21.61, 32.11, 63.16 and 42.97% increase respectively, but shows decreasing trend for composites without coupling agent with the exception of tensile modulus and hardness which increased from 0 to 50% filler loading representing 71.9% increase and 76.46% increase respectively. Also, SEM micrographs for the fractured surfaces show even dispersion of the filler particles in the composite with coupling agent and show signs of agglomeration for composites without coupling agent.

**Keywords:** Calabash Particles, HDPE, Tensile Strength, Flexural Strength, Impact Strength

### **INTRODUCTION**

Calabash (*Lagenaria Ciseraria*) is simply the dried hollow shell of a gourd used for household utensils [1]. The fresh fruit has a light-green smooth skin and a white flesh. Rounder varieties are called calabash gourds [2]. Thermoplastic polymer has formed a new trend in material development, and it has the advantages of low production cost, great diversity, sufficient sources, light weight, good physical properties, and chemical resistance, as well as various efficient manufacturing process. Therefore, there are a great number of plastic products commercially available [3]. Plant fibres are biodegradable and readily available when compared with glass fibres [4]. Most plastics are not suitable for load bearing applications by themselves due to their insufficient strength, stiffness or dimensional stability [5].

In recent years, wood fibre-reinforced plastic composites or so-called wood-plastic composites (WPCs) have attracted significant interest and they are replacing wood in many fields [6], better stability and favourable mechanical properties has caused WPCs to become a preferred building material [7].

The recent world population growth has been one of the main factors for the technological development and innovation. In fact, exponential growth has taken place in the world population, being expected to reach 9 billion in 2042, compared to 3 billion in 1960 [8].

Contrary to human needs, the earth's resources are limited. In the era of rapidly growing population and global warming, environmental issues cannot be neglected in almost all the fields of engineering. Both the industry and researchers try to utilize renewable resources in more and more products and processes. The idea of the replacement of synthetic fibres with natural-based fillers in polymer composites has been incorporated since 1970s and has recently gained popularity [9].

Eco-friendly bio-composites consisting of agro-fillers and bio-based plastics are of great importance to the materials and research world as a feasible solution to growing environmental threats and as a sustainable solution to the uncertainty of the world's petroleum supply [10].

Dissimilarity of the surface chemistry between the filler and polymer usually contribute to the decrease of the tenacity in filler-polymer systems

[11] CP are hydrophilic while HDPE is hydrophobic.

Therefore, in order to reduce the non-biodegradability of synthetic polymers, biodegradable fillers can be incorporated into synthetic polymers to produce composites that are of biodegradable, low cost and eco-friendly.

## MATERIALS AND METHODS

### Materials

High Density Polyethylene (HDPE)(Zayo-Sigma Chemicals Ltd. Jos), Maleic Anhydride (MA) 98% purity (coupling agent), Benzoyl Peroxide 98% purity (initiator), Calabash Particles (CP), Retsch Sieve Shaker Machine (Emdocatt. Model: 7416), Laboratory Mill (Model 4, Arthur H.

Thomas Company, PA., U.S.A.), Vacuum Oven (model: 60648, Cole Parmer), metallic mould, Two-roll Mill (Model: 5183 North Bergen U.S.A), Compression Moulding Machine (model: 0557) Wenzhou Zhiguang Shoes making machines CO., Ltd. China.

### Methods

#### Collection of Materials

Calabash bowls (dried seeds and whitish material removed) were obtained from Zurmi, in Zurmi Local Government Area of Zamfara State, Nigeria. All have varying sizes of between 10 to 15 litres in volume. The calabash bowls were broken into smaller pieces which were then fed into a laboratory milling machine and further crushed into smaller particle sizes. The ground calabash particles from the milling machine was sieved to 125µm particle size. Moisture was removed from the sieved particles in an oven until a constant weight was obtained thereafter stored in a desiccators.

#### Preparation of the Composites

The composites were prepared from the calabash particles and HDPE at 10% varying filler loading up to 50% while the HDPE was reduced simultaneously from 100% down to 50%, during compounding using a two-roll mill. The composite was prepared in two forms, one with coupling agent (CP/HDPE A) and the other without coupling agent (CP/HDPE B). Two roll mill was used for compounding and thereafter compression moulded and cooling of the composites were carried out for 10 minutes each.

Table 1: Percentage composition of CP (125µm) and HDPE in each of the composite formulation at different filler loadings

S/NO.	CP (%)	HDPE (%)
1	0	100
2	10	90
3	20	80
4	30	70
5	40	60
6	50	50

### Tests for the Composites

#### Tensile Strength (ASTM D-638)

Tensile strength test was carried out according to ASTM D-638 standard with multifunctional electric tensile strength tester (YG026D) at Standards Organization of Nigeria in Kaduna. The machine loadcell of 300kg capacity was used for each of the samples. The test sample dimensions were 50mm x 10mm x 4 mm.

#### Flexural Strength (ASTM D-790)

Flexural strength test was carried out according to ASTM D-790 standard with Universal Material Testing Machine (Cat Nr. Model: 26) at the Department of Mechanical Engineering, ABU, Zaria. The machine has a maximum force of 1K, and sample dimensions were 100mm x 25mm x 8mm

#### Impact Strength (ASTM D-256)

Impact strength test was carried out according to ASTM D-256 standard with Charpy Impact Testing Machine at The Department of Metallurgical and Materials Engineering, ABU, Zaria. The machine load of 15J capacity was used for each of the samples. The test sample dimensions are of 80mm x 10mm x 10 mm.

#### Hardness Test (ASTM E-384)

The hardness test was carried out according to ASTM E-384 standard with a Vickers Hardness Tester Machine Model MVI-PC YADRAN-416145, INDIA, using a diamond indenter at Shell Professorial Building, ABU, Zaria. The machine load of 500gf at 15 sec was used for each of the samples whose dimension is 30mm x 25mm x 8mm (length, width and thickness). The test was repeated three times and an average test result was calculated with its mean value and standard recorded

#### Scanning Electron Microscopy Test (SEM) (ASTM E766-98)

SEM test was carried according to ASTM E766-98 standard using fractured surface of the tensile specimens with Pro: X: Phenom world. Model number 800-07334 at the Department of Biology,

Umaru Musa Yaradua University, Katsina. To impart conductivity to the polymer composites, a sputter machine used 5nm gold to coat the sample surface. The samples were inserted into the machine using the sample holder and the morphology of the samples were viewed at 15KV, and Images were obtained at 1000X magnifications.

## RESULTS AND DISCUSSION

### Tensile Strength

From Fig. 1 the tensile strength of CP/HDPE composite with coupling agent can be seen to be increasing with increased filler loading from 27.54 MPa of the neat polymer to 35.13MPa at 40% filler loading i.e. 21.61%. The increase might be attributed to the interfacial adhesion between the filler and the matrix. Maleic Anhydride was introduced as coupling agent at 3% on the weight of the filler, it might have tailored the properties of the composite by increasing bonding between filler and matrix. The incompatibility of polar wood fibres and non-polar polyolefin leads to difficulties in obtaining uniform dispersion of wood fibres in the matrix, which in turn reduced the efficiency with which the fibres reinforce the polymer, but introduction of coupling agent into a composite system can improve mechanical properties [12].

At 50% filler loading, it can be observed that the tensile strength dropped by about 50%, this might be as a result of the matrix losing its binding ability [13]. The matrix might be approaching its saturation level which occurred at 60% filler loading based on preliminary investigation.

Composites without coupling agent show a decreasing trend from 27.54 MPa of the neat polymer to 12.20MPa at 50% filler loading, the decrease might be attributed to poor interfacial adhesion between the filler and matrix [13].

### Tensile Modulus

From Fig. 2, it can be observed that tensile modulus of CP/HDPE composite was increasing for both composites with and without coupling agent from 0.88GPa of the neat polymer to 1.21GPa (42.97% increase) and 1.11GPa (37.84% increase) for composites A and B respectively. The increase in tensile modulus might be attributed to the stiffening effect caused by introduction of rigid fillers which might have reduced the polymer chain mobility consequently increasing modulus [14].

### Flexural Strength

Fig. 3 shows similar trend with tensile strength, the flexural strength of CP/HDPE composite A was increasing with increase in filler loading from Nigerian Journal of Textiles (NJT) Vol. 5: 11 – 16

43.47MPa of the neat polymer to 64.03MPa at 40% filler loading representing 32.11% increase. The increase in flexural strength might be attributed to the incorporation of maleic anhydride as coupling agent which might have improved the bonding between the CP and HDPE. Increase in interfacial bonding between filler and matrix improved mechanical properties [15], because improved filler matrix interaction provides effective transfer of stress from matrix to filler [16]. For composites without coupling agent there is a decreasing trend from 43.47MPa of the neat polymer to 26.42MPa (39.22% decrease) at 50% filler loading, the decrease might be attributed to poor interfacial adhesion between the filler and matrix [15].

### Impact Strength

The result of impact strength is shown in Fig. 4, there is increase in impact strength with increase in filler loading of the composite for CP/HDPE composites A from 35J/m of the neat polymer to 95J/m at 40% filler loading representing 63.16% increase. This might be attributed to improved interfacial interaction between the filler and matrix when coupling agent was introduced in the composite system, maleic anhydride has the property of covalent bonding on to another polymer chain [17]. Introduction of coupling agent into a composite system with dissimilar surface chemistry between the filler and matrix improves interfacial adhesion and consequently improves mechanical properties, increase in impact strength has been reported by several researchers due to the incorporation of coupling agent into a composite system [18].

Meanwhile, for composites without coupling agent, impact strength was found to be decreasing with increased filler loading from 35J/m of the neat polymer to 12J/m (65.71% decrease) at 50% filler loading. The dissimilarity between surface chemistry between filler and matrix brings about reduction in the tenacity of composites [16], decrease in impact strength my result from the non-inclusion of coupling agent in the composite.

### Hardness

Hardness of CP/HDPE composites A and B was found to be increasing with increased filler loading from 9.37Hv of the neat polymer to 44.50Hv (71.9% increase) and 39.80Hv (76.46%) increase for composites with and without coupling agent respectively. Hardness of a material is dependent on stiffness of that material, introduction of rigid fillers impart stiffness to synthetic polymer materials consequently increasing hardness [19].

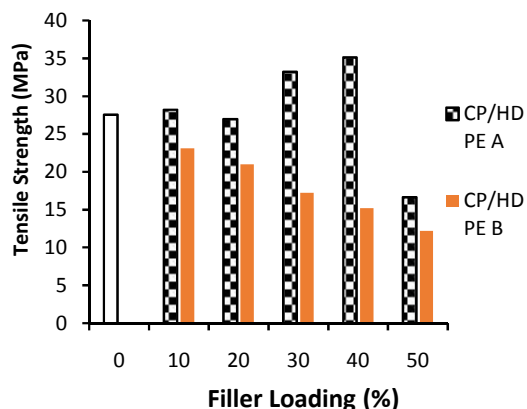


Fig. 1: Tensile strength of CP/HDPE composites A and B at varying filler loading

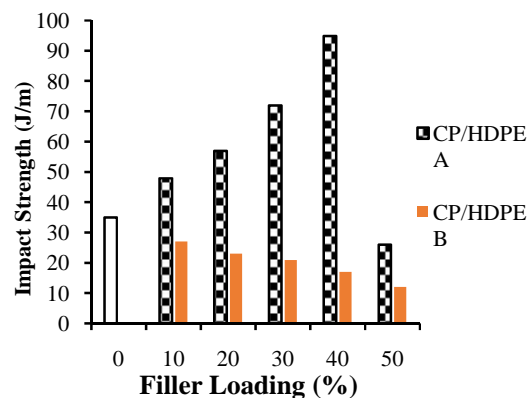


Fig. 4: Impact strength of CP/HDPE composite A and B at varying filler loading

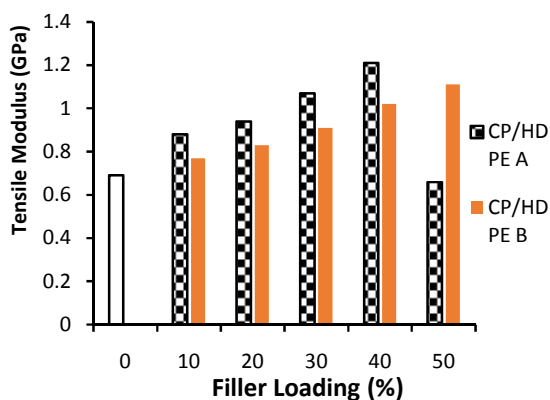


Fig. 2: Tensile modulus of CP/HDPE composites A and B at varying filler loading

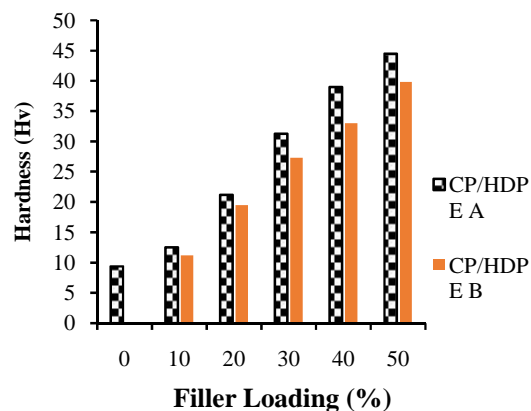


Fig. 5: Hardness of CP/HDPE composites A and B at varying filler loading

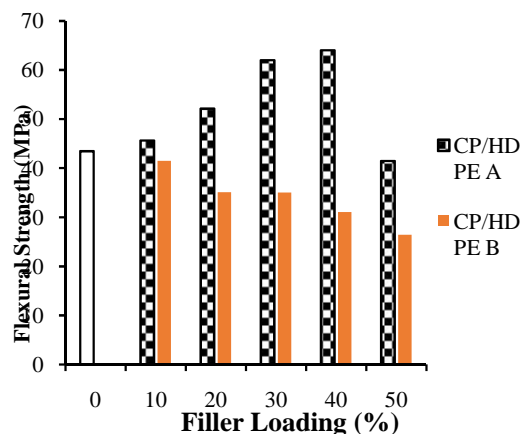


Fig. 3: Flexural strength of CP/HDPE composites A and B at varying filler loading

### Scanning Electron Microscopy

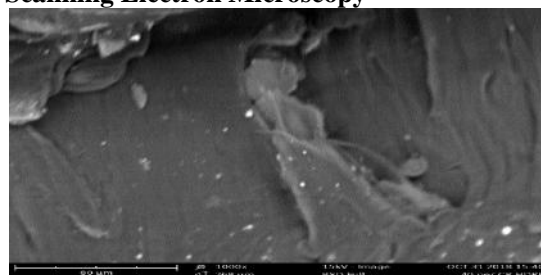


Fig. 5: SEM image of CP/HDPE composite A at 40% filler loading with coupling agent

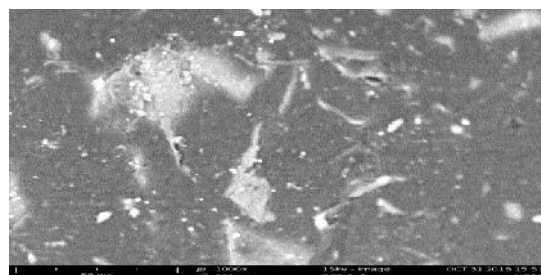


Fig. 6: SEM image of CP/HDPE composite B at 40% filler loading without coupling agent

Fig. 5 and 6 shows the SEM images of CP/HDPE composite at 40% filler loading with and without coupling agent, since 40% filler loading gave the best mechanical property, fractured surface of the tensile test specimen was used for SEM determination.

From the micrograph in Fig. 5, it can be observed that there is an even distribution and dispersion of the CP in the composite with coupling agent. One advantage of using coupling agent in composites is that it helps in making the reinforcing fillers to be fully embedded in the matrix thereby preventing debonding which leads to plastic deformation of composites [19]. Meanwhile, in Fig. 6, the SEM image shows areas of agglomeration and uneven dispersion of the filler in the composite without coupling agent which might be the reason why there is reduction in the mechanical properties of composites without coupling agent.

## CONCLUSION

The effect of coupling agent on the mechanical properties of CP/HDPE composites was studied to determine the viability of maleic anhydride in improving the mechanical properties of composites. The result of tensile, flexural, impact strength, show increasing trend from 0 to 40% filler loading which might be due to incorporation of maleic anhydride as coupling agent but shows reducing trend with composites without coupling agent. Meanwhile, tensile modulus and hardness were increasing with increased filler loading for composites with and without coupling agent. The SEM image of CP/HDPE composite A at 40% filler loading shows even distribution of fillers in the matrix which is a sign of proper bonding between the filler and matrix preventing debonding, while, CP/HDPE composite B shows uneven dispersion of fillers with areas of agglomeration. From the result of the mechanical properties obtained, it can be deduced that the composites can be used for applications where low to moderate mechanical properties are required.

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## **Industrial Design in Nigeria: A Mechanism for Expanding the Roles of Industrial Sector as Empowerment for Youth and National Development**

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### **ABSTRACT**

*The Industrial design in Nigeria is a potential and an agent for expanding various roles of industrial sectors in order to empower the youth that is skillful in Industrial Designs. This paper focuses on the industry (relatively low capital intensively low investment cost, that are able to use low skilled labour as a means that the industry is relatively footloose as well as able to adjust and to manage the market condition quickly as possible. It explains the manufacturing industries as the process of creating designs for mass production purposes in the industries. It further explains the uses of computer-aided design softwares for designing products as well as expressing design industries as agent of National development. It expresses in details the social aspect of Industrial design as well as the challenges facing industrial design in Nigeria which include Infrastructures and corruptions among others. Based on the above reasons, the paper focuses on expanding the roles of industrial design and likewise the attempts to begin the assessment of the impact of long-term policies on the design Industries as sectors that can employ on a largely supply of unskillful workers from formal and informal labour forces and proffers recommendations and conclusion.*

**Keywords:** Industrial Design, Agents, Empowerment, National Development.

### **INTRODUCTION**

Industrial design form a major part of manufacturing, employment and trade in many developing countries. This paper will examine the impacts and role of industrial design in growth and development strategies for developing countries. The Industrial design is one of the oldest, largest and most global industries in the world. It is the typical 'starter' industry for countries that engaged in export-orientated industrialization [6] and is labour-intensive. The technological features of industrial design have made it suitable as the first step on the 'industrialization ladder' in poor countries some of which have experienced a very high output growth rate in the sector, such as Bangladesh, Sri Lanka, Vietnam and Mauritius, and have since become middle income countries [10].

A number of reasons [10] why the designing sector has played such an important role in economic development. The sector absorbs large numbers of unskilled labour, typically drawing them from rural agricultural households to urban locations. Despite relatively low start-up investment costs, expansion of the sector provides a base upon which to build capital for more technologically demanding activities in other

sectors. Growth of the sector allows imports of more advanced technologies to be financed through revenues gained from products exports.

The industrial design was major sources of export, income and employment for the country before the discovery of oil exploration and exploitation. This discovery of oil (Petroleum) led to government' negligence to the people's dignity – farming resulting to corruption, poor management, white collar job perception, certification preferences over vocational and apprenticeship careers. These were influenced by the Nigerian government policies and implementation. However, the oil boom period promotes industrial and infrastructural development, and higher revenue income generation but the Nigerian government failed to grow and develop as well the industries and comparative business advantage of the regional economy. These policies and actions portrayed by government caused the total collapse of almost all indigenous design industries leading to the present mono-economy situation Nigeria is experiencing today. Diversification of economy to solid minerals and agriculture and also, the revitalization of the role of design in order to engage our teaming unemployed youth have been the media jingles and Government's mantra.

However, the characteristics of the industry (relatively low capital intensity; low investment costs; and use of low skilled labour), also means that the industry is relatively footloose and able to adjust to changing market conditions quickly [10]. Government policy regulations have had a major impact on the pattern of Industrial design production and are likely to do so in the near future. China has become a very important player now that restrictions on its trade are progressively being lifted. This has intensified competition for traditional designers and producers especially in small and remote countries. This paper therefore provides Nigerian government and industrial designers' advice for a sustainable development of our designing industries.

## **DESIGN**

In normal usage, the word design can be taken to mean a plan or scheme, which may be written or drawn, showing how something is to be constructed, or how the elements of an item or article are arranged. In the legal terms, a design is defined by reference to the provisions applicable to either the registered design or the design right, as appropriate.

According to [5], Industrial design is any combination of lines or colours or both and any three dimensional form, whether which is intended by the creator to be used as a model or pattern to be multiplied by industrial process and not intended solely to obtain a technical result. Industrial designs are primarily those elements incorporated into mass produced products that aim to enhance their attractiveness by their appearance. The primary objective of the law governing industrial design is to protect a design that is new or essentially better in some ways than what was created before. This protection is of immense importance to artists, lace designers and designers of other types of products. When goods are similar, designs become an important distinguishing factor.

### **Definition of Industrial Design**

Industrial design is the application of arts and science for the improvement of aesthetics, architecture, ergonomics, functionality and usefulness of the specific product, [4]. Industrial design can be used for the improvement of the competitiveness of a product and its manufacturing. The major role of an industrial designer is the creation and development of the most successful project solutions related to the shape, usability, physical ergonomics, marketing, brand development and sales, [7]. Industrial

design is an integral part of the scientific progress and development of business. Sometimes, companies have to change the shape, functions and usability of their products in order to attract new customers and compete with other similar firms on the same sector of the market. Although everyone knows at least something about the foundations of industrial design, very few people are aware about the roles of this matter.

### **The Role of Industrial Design**

**Industrial design** is essentially the process of creating designs for the industry, for mass production purposes. Industrial designers are involved with the production of these designs, which are used, sometimes repetitively, by the industries. The field encompasses the actual pattern making while supervising the production process. In other words, industrial design is a process from the raw material into finished product. Industrial design fulfils a variety of purposes in our lives. For example, our clothing, carpets, handsets, laptops, electronics, cars, towels, rugs, etc are all results of industrial design.

These examples illustrate the significance of industrial design in our daily lives. The creations of product are not only important for their use, but also for the role they play in the development of the country. Industrial designers have the ability to inspire collections, trends, and styles and the industry, while being a creative art form, is a business opportunity for the sector. Industrial designers marry a creative vision of what a finished product will look like with a deep understanding of the technical aspects of production and the properties of fibre, yarn, and dyes. The creative process often begins with different art mediums to map concepts for the finished products. Traditionally, drawings of woven textile patterns were translated onto special forms of graph paper called point papers, which were used by the weavers in setting up their looms [6].

Today, most professional industrial designers use some form of computer-aided design softwares created expressly for this purpose. Some of the latest advances in printing have been in the area of digital and 3D printing. The process is similar to the computer controlled paper printers used for office applications. In addition, heat-transfer printing is another popular printing method to be used for design [7].



*Fig 1: Product Development Circle [11].*

### **DESIGN INDUSTRIES AS AGENTS OF NATIONAL DEVELOPMENT**

The development of a nation cannot be discussed without the factors that contribute to such development which are usually embedded in diverse areas of a nation's economy. The need to actualize and stabilize Nigerian economy implies harnessing all resources and factors that can reshape its economy to achieving a purposeful growth. In this regard, industrial designs have been identified as a dependable employer of labour and a source of financial security for most Nigerian youths [1].

Apart from food and shelter, industrial design (products) has been identified as the most important in the hierarchy of man's need [8]. In Nigeria, the production of items had flourished at both cottage and industrial levels. Studies show that the industry had been a major employer of labour (about 60% of the labour force) in the manufacturing sector and contributed immensely to the socio-economic and cultural development of the country [2], [7]. It is unfortunate; however, that the current economic low-ebb in Nigeria has

developed adversarial forces clamping the proper functioning of these industries as a resourceful economic contributor.

### **THE IMPACT OF INDUSTRIAL DESIGN ON THE ENVIRONMENT**

Industrial design is changing rapidly in all areas of sectors as a basic fact as seen in all manufacturing industries. However, the product such as clothing, ceramics wares, cars were available to us in the market are at present are more expensive than they were few years ago. In the 1980s, for example, products choices were based on the functionality: products were selected according to certain attributes such as comfort, durability and versatile characteristics. Currently consumers buy products impetuously, irrespective of whether they meet the attributes outlined above [9].

### **The Social Aspect of Industrial Design**

As an industrial designer, understanding the mindset of the customer is critically important to what you do. Social innovation occurs when the voices, expertise, and insights of key players in a

market space form a single, dynamic community, which influences, guides, and fosters the development of innovative industrial designs. Social innovation is so vital to the development of successful industrial designs that most leading manufacturers have formal voice of the customer programs. Accurately capturing the customers point of view is a social, iterative process, demanding a social-networked approach to industrial design that allows you to manage and control these communications, so vital information and innovative ideas don't get lost.

### **CHALLENGES FACING INDUSTRIAL DESIGN IN NIGERIA**

The industrial design sector in Nigeria accounts for a tiny proportion of economic activity (6 per cent) while the manufacturing sector contributes only 4 per cent to Government Developmental Programs (GDP) in 2011. There are some challenges faced with industrial design leading to inadequate development in Nigeria. Among these issues, three in particular top the current policy debate and these are; the development of the critical infrastructure, tackling corruption and ensuring national security.

#### **Infrastructure**

The current infrastructure base in Nigeria is grossly inadequate in terms of capacity and quality and is not capable of catering for the anticipated industrial development. Despite government investments, Nigeria still has huge infrastructure deficits, particularly with regards to power generation. The current power generation capacity is less than 2000 Megawatt, which is about 20 per cent of the estimated national demand [8]. A key challenge for government and the private sector is to build a modern, efficient, and effective infrastructure network within the next five to ten years.

Electricity outage is the key factors constituting impediments to firm growth. Nigerian manufacturing firms suffer acute shortages of infrastructure such as good roads, portable water, and, in particular, power supply. Electricity outages and voltage fluctuations are commonplace, causing damage to machinery and equipment. Consequently, most firms rely on self-supply of electricity by using generators, which escalates their costs of production and erodes their competitiveness relative to foreign firms.

#### **Corruption**

Nigeria ranks highly in the Corruption Perception Index [3]. This has implication for investment

flow into the country. Previous anti-corruption policies implemented in Nigeria have been targeted at enforcement measures rather than addressing the root causes. The root causes of corruption in Nigeria have been identified to include social insecurity and over-centralization of resources at the centre. Even though there are suitable laws and viable institutions to fight corruption in Nigeria, the greatest challenge is in formulating a strategic plan of action to deal with the root causes.

#### **National Security**

The internal security of Nigeria has become a very big challenge in recent times. Internal conflicts, including religious, ethnic and political, have had debilitating effects on the economy, most notably by scaring investors from certain parts of the country. Even though insecurity of lives and properties had become noticeable following the civil war and the subsequent military regimes which directly intensified urban violence, the recent upsurge of violence and insurgency (Boko-Haram, Avengers and Biafra Agitators) in the country heightens the need to comprehensively address the persistent causes of social tension as a risk factor to Nigeria as an investment destination.

### **THE FUTURE OF NIGERIAN YOUTH INDUSTRIAL DESIGN**

There are huge learning opportunities for designers locally, in our various institutions, such as Ahmadu Bello University (ABU), Zaria, Federal University of Technology, Akure (FUTA), and Kogi State Polytechnic, Lokoja where Students can learn and practice product development to the value of a designer. Most of the products developed were left without been used by the industries as expected. It is hoped in the future that these designs can translate to product developed for mass production in industries like Textile Industry, Ceramics Industry and Garment Industry.

In Nigeria, most of the graduate Industrial Designers are not practicing, as there are no availability of equipments to practice, insecurity, lack of infrastructure and materials, in order to showcase their knowledge as industrial designers. In the future, these Nigerian youths of Industrial Designers will be the most employers of labour, because in all industries, they cannot produce any item without the input of Industrial designers that consist of Nigerian youth.

## RECOMMENDATION

- Government should rationalize and strengthen production chain where necessary by setting up institutions to channel raw materials and finished products.
- Diversifying product base: the Industrial designers in the factories can diversify their outdated product base such as changing bottles to different shapes, changing dress wares into fashion materials like shoes, interior decoration, old ceramics works to flower pots or tiles. Industrial designers studied the customers and diversify the product development to their present interest needs. Instead of most manufacturing industries to close down, the knowledge of Industrial designers can be employed, so that the Industries that have closed down can diversify their products as subsidiary firms that can grow and re-establish as a viable Industry progressing over years respectively. That will also boost the export of Nigerian made products overseas.
- Promoting an export culture thus growing domestic and export demand for local industries. Although Nigeria is the destination market for most trading partners such as China, U.S and Holland for the products, ultimately to improve our share of the market on the world scale, Nigeria must export whether to the sub region or the further parts of the world. All global players operate in international markets. China is said to be aiming to increase her foreign trade volume by outpacing world average by 4% annually to remain above 10% in fifteen years [11]. Many experts also say Nigeria must start growth targeting, whether in exports or overall trade. Export promotion will be linked to other things like tourism promotion to promote local products to international status the way Chinese, Japanese and Italian products have become international.
- Industrial friendly policies such as reduction of tax should be adopted to ensure that improve economic incentives.
- Government should focus mainly on providing strong support for technical training as well as educational programs related to the industrial design just like the Chinese design industries who have invested much on training the workforce in modern technology and management skills.
- The government should demand an improvement in the output and productivity from design industries to encourage innovations, and innovators periodically come

together and sell their innovations to other industries [7].

## CONCLUSION

Nigerian design industries have the capability of making the nation grow if given proper handling and attention. Both traditional and contemporary industries are essential to national growth. Also, the problems of power, transportation and other basic infrastructures have weakened the industry's potentials. We would imagine that this is one thing that could be taken care of, given some will to do so. It is almost redundant these days to mention power, transportation and other basic infrastructure. But yes, these are going to be crucial to any resuscitative attempts.

The Nigerian government's determination and political-will to be active player in industrial design will help to fully realize its potentials. The patronage of smuggled products by some Nigerians indicates that Nigerian Industries are to be closed-down. But if Nigerians have a change in attitude and start patronizing made in Nigeria products that will create employment and sustainable development for the young industrial designers in Nigeria. The fact that Nigeria is already acknowledged as a big market makes this a worthwhile venture. The involvement of the Indians and Chinese might just be the catalyst that is needed.

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## Service Properties of Selected Locally Manufactured Carpets

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### ABSTRACT

The service properties of selected made in Nigeria carpets have been investigated. Properties studied include; constructional details, fastness properties (wash fastness and light fastness), abrasion resistance, tuft withdrawal force, flammability test, static and dynamic loadings, electrostatic properties and soil retention properties. Assessment was carried out on those properties among the carpet samples and the results obtained shows that the density of the base materials, the characteristics of the pile yarns, the stitch density and the type of fibres used for the carpet as well as the type of finishing impacted on the carpet influenced the service properties of the carpet.

**Keywords:** Flammability, Carpet, Finishing, Electrostatic, Pile, Wash fastness, Light fastness.

### INTRODUCTION

Carpets are thick coverings for floor or stairs, usually woven, knitted or tufted and are made of wool or other fibres which may be plain or having designs woven unto them. It could also be a textile soft floor covering that is produced by permanent orientation of fibres (synthetic or natural) into a substrate [1]. Often times, the term carpet is interchangeably used with the term “rug” although rug is a loose laid carpet traditionally smaller than room dimensions in size. A carpet is usually valued not only for its appearance but also for its performance during use as it offers great values along with the general benefit of security, aesthetic beauty, comfort underfoot, insulation factors, sound absorption and even some respiratory health advantage [2].

The end use performance of carpet is characterized by both mechanical and optical characteristics which make up the physical properties of the carpet and ultimately influence their performance significantly [3]. While the mechanical properties are used to explain the walking comfort and abrasion related wear, the optical properties are used to explain appearance. The major properties of importance in carpet assessment are durability, appearance retention, pile height and pile density [4]. Structural parameters for example were investigated through experiments to evaluate carpet appearance loss [5]. Mathematical models were established to understand wear mechanisms and to predict wear life of cut-pile and loop pile carpets [6].

However, in this study, the service properties such as tuft withdrawal force, abrasion resistance, light and wash fastness, flammability, electrostatic properties, soil retention, static and dynamic loadings, pile height and pile density of some locally manufactured (made in Nigeria) carpets were measured and the results obtained were used to rate the carpet by virtue of their appearance, performance and end use serviceability.

### MATERIALS AND METHODS

#### Materials

The materials used for this study were five different tufted carpet samples manufactured by Nobel Carpet Company, Lagos, Nigeria. The carpets include; Hilite Red, Imperial Blue, Winners Brown, Prestige Red and Pentagon Red. These samples were labelled X<sub>1</sub>, X<sub>2</sub>, X<sub>3</sub>, X<sub>4</sub>, and X<sub>5</sub> respectively.

#### Equipment

Some of the equipment that were used for this study include but not limited to; Grey scale, weighing balance, counting needle, SDL Carpet abrasion tester (HD-D125), SDL WIRA Tuft withdrawal (K224), Gyrowash (FH 234), pile height gauge, Xenon arc lamp (M233) etc.

#### Conditioning of Samples

All the carpet samples were conditioned in a standard atmosphere of relative humidity 65±2% and temperature of 20±2°C for 172800 seconds prior to testing.

## Methods

### Construction Details

The method of determination of the construction details was in accordance with the procedure described in [7].

### Pile Density and Pile Height

The usual method of counting which involves using a counting glass and counting needle was adopted by counting the piles (cut or uncut) in a unit area e.g. square centimeter over about ten different areas on the tufted carpet sample. The average of the number was then calculated which gave the pile density. Pile height was measured using the pile height gauge and the value calculated to the nearest mm. The results are shown in Table 1.

### Total Pile Weight

100 tufts were weighed and the total pile weight was estimated from the weight. The results are shown in Table 1.

### Abrasion Resistance

The test was carried out using the WIRA Carpet Abrasion tester in accordance with the procedure described in [8]. A standard abrasive cloth was used to abrade the carpet specimen to rupture point and the number of rubs were recorded. Ten tests were carried out and the results tabulated.

### Tuft Withdrawal Force

The test procedure was as described in [8]. The withdrawal force required to remove tufts from the carpet specimen was determined using WIRA Tuft Withdrawal Tensometer with a 550N load cell and accuracy of 5%. The force required breaking the bond between the tuft and the backing structure was recorded. Ten tests were carried out and the results tabulated.

### Light Fastness Test

The test was carried out according to the procedure in [9]. The test specimen was exposed to artificial light source from Xenon arc lamp alongside with standard dyed material of known light fastness (blue wool standard).

### Wash Fastness Test

The test procedure was in accordance with [10]. The test specimen of dimension 5cm X 4 cm was placed between two specified pieces of undyed cloth of dimension 10 cm X 8 cm. The three pieces were stitched together to form a composite specimen. The composite specimen was placed in a container containing 5 g/l soap and 2 g/l Na<sub>2</sub>CO<sub>3</sub> solution previously heated to a temperature of 60±2°C to give a liquor ratio of 50:1. The

composite specimen was removed, rinsed and assessed. Assessment of change in colour of the specimen and the staining of the adjacent fabric was carried out using a grey-scale.

### Soil Retention Test

A predetermined amount of soil of weight 0.8 g containing a mixture of dust under carpet, oil and clay was applied to each specimen of dimension 5 cm x 3 cm. It was then allowed to aerate for 86400 seconds. A standard solution of soap (containing 5 g/l soap and 2 g/l Na<sub>2</sub>CO<sub>3</sub>) was used to wash each specimen. The time taken for complete removal of the soil from each specimen was recorded. Ten tests were carried out for each sample and the results tabulated.

### Flammability Test

The test was carried out in accordance with the procedure in [8]. The vertical strip test method was employed. The test specimen of dimension 4 cm x 1 cm was suspended in air-free cabinet and held at the top and over the topmost wire by clips. The flame from a candle-stick was put at one inch before the lower end of the specimen. With the aid of a stop-watch, the time taken to consume the specimen from its lower end to the top end was recorded and used for the carpet flammability grading. Ten tests were carried out for each sample and the results tabulated.

### Dynamic Loading Test

The test procedure was in accordance with [11]. The WIRA Dynamic Loading Machine was employed for this test. A cam-operated load of nearly 1.3 kg was dropped freely at regular intervals of about 4 seconds from a height of 64 mm onto the carpet specimen. Thickness loss of the carpet specimen expressed as a percentage of the pile height after 1000 impacts was recorded. Ten readings were taken for each sample and results tabulated.

### Static Loading Test

The test was carried out according to procedure described in [12]. A pressure of 7 kg/cm<sup>2</sup> was applied to a small foot on top of the carpet specimen for 86400 seconds and the indentation was measured after 86400 seconds of recovery. Ten readings were taken and results tabulated.

### Electrostatic Properties

The test procedure is as described in [12]. The method of capacitor discharge was used. The resistivity of the carpet sample was measured along the longitudinal direction. The resistivity and the time were recorded and used to obtain the anti-static rating for the carpet samples.



**RESULTS AND DISCUSSION**

**Carpet Construction Details**

From Table 1, the carpet samples show acceptable properties in terms of pile height, pile type, pile density and total pile weight. Sample X<sub>3</sub> shows the best result having the highest pile density and one of those with high pile height while sample X<sub>1</sub>

shows the least result having a low pile height and density. The carpet pile density and pile height are the main structure parameters of carpets, which have an influence on resulting deformations during compression. Any variation of these two mechanical parameters will have an influence on the end – use properties of carpets [13].

Table 1: Carpet Construction Details

Sample	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	X <sub>4</sub>	X <sub>5</sub>
Pile height (mm)	4	5	5	5	4
Pile type	Cut	Loop	Cut	Cut	Loop
Pile density (tufts/cm <sup>2</sup> )	23	24	26	24	23
Total pile weight (g/cm <sup>2</sup> )	0.3560	0.3610	0.3680	0.3620	0.3650

**Abrasion Resistance**

The abrasion resistance results in Table 2 show that, carpet sample X<sub>3</sub> has the highest abrasion resistance while sample X<sub>5</sub> has the least. It was observed that the pile density and pile height influenced abrasion resistance. Thus, sample X<sub>3</sub> with the highest abrasion resistance has the highest pile density and one of those with a high pile height. The order of abrasion resistance is X<sub>3</sub> > X<sub>2</sub> > X<sub>4</sub> > X<sub>1</sub> > X<sub>5</sub>. Generally, abrasion resistance results were better for the more dense carpets.

Table 2: Abrasion Resistance

Test Samples	Mean No of Rubs	Standard Deviation	Coefficient of Variation
X <sub>1</sub>	5803	8.63	0.148
X <sub>2</sub>	8350	7.07	0.085
X <sub>3</sub>	20500	12.10	0.009
X <sub>4</sub>	8150	4.12	0.051
X <sub>5</sub>	4000	3.53	0.088

**Tuft Withdrawal Force**

From Table 3, the results of the tuft withdrawal force for the carpet samples followed this trend in order of increasing values: X<sub>5</sub> > X<sub>1</sub> > X<sub>3</sub> > X<sub>2</sub> > X<sub>4</sub>. It was observed that the pile height has no significant influence on the force required to remove tufts from the carpets. The variation in the tuft withdrawal force for the samples may be attributed to the type of binder or latex used in coating the backing material.

Table 3: Tuft Withdrawal Force

Test Samples	Mean Tuft Withdrawal Force (Kg)	Standard Deviation	Coefficient of Variation
X <sub>1</sub>	2.24	0.0158	0.7058
X <sub>2</sub>	1.92	0.0057	0.2968
X <sub>3</sub>	2.13	0.0043	0.2018
X <sub>4</sub>	1.80	0.0151	0.8388
X <sub>5</sub>	3.83	0.0032	0.0835

**Light Fastness**

The results in Table 4 shows that sample X<sub>5</sub> has the highest light fastness rating while sample X<sub>2</sub> has the lowest. The colour change of the carpets appeared to be influenced by the depth of shade of the dyes applied. All the carpet samples have an acceptable light fastness rating. The deeper the shade the more the change in colour as seen on the tabulated results.

**Wash Fastness**

The results in Table 4 show the wash fastness of the samples tested. The whole samples have very good to excellent wash fastness with sample X<sub>1</sub> and X<sub>4</sub> having the best wash fastness results. Wash fastness is usually influenced by the rate of diffusion of dyes and the state of dyes inside the fibre. In this case, the high wash fastness rating could be attributed to the better affinity of the dyes on the fibre and their tendency to aggregate thereby forming large molecular sizes within the fibre [14]. The colour change of the carpets appeared to be influenced by the depth of shade of the dyes applied. The deeper the shade the more the change in colour as seen on the tabulated results.

Table 4: Light and Wash Fastness Ratings

Test Sample	Light Fastness Rating	Wash Fastness Rating	Change in Colour	Change in Staining
X <sub>1</sub>	5	4 - 5	4 - 5	3
X <sub>2</sub>	4	4	4	2
X <sub>3</sub>	5	4	4	2 - 3
X <sub>4</sub>	5	4 - 5	4 - 5	3
X <sub>5</sub>	6	4	4	2 - 3

**Soil Retention Property**

The results in Table 5 gave an indication that the higher the time taken to wash off the dirt completely from the carpet sample, the higher the

soil retention property of the sample. The order of decreasing soil retention property is:  $X_1 > X_3 > X_5 > X_2 > X_4$ . The fibre type, as well as, the electrostatic properties of the samples may have influenced the soil retention property of the carpets [15].

Table 5: Soil Retention

Test Sample	Time (Sec.)	Standard Deviation	Coefficient of Variation
X <sub>1</sub>	29	1.825	6.2956
X <sub>2</sub>	16	0.974	6.0875
X <sub>3</sub>	21	1.032	4.9142
X <sub>4</sub>	15	1.012	6.7466
X <sub>5</sub>	18	1.001	5.5611

**Flammability Rating**

The test results in Table 6 show that all the carpet samples tested are flame retardant to different extent. The order of flame retardancy is:  $X_1 > X_5 > X_2 > X_3 > X_4$ . The variation in flame retardancy may be attributed to the pile material used, as well as, the type and extent of the flame retardancy finish imparted on the carpets [16].

Table 6: Flammability rating

Test Sample	Mean time of consumed specimen (Sec.)	Standard Deviation	Coefficient of variation
X <sub>1</sub>	86	5.416	6.297
X <sub>2</sub>	66	1.732	2.624
X <sub>3</sub>	62	2.380	3.839
X <sub>4</sub>	47	2.236	4.757
X <sub>5</sub>	71	2.309	3.253

**Dynamic Loading**

Table 7 shows that sample X<sub>4</sub> have the highest percentage loss in thickness expressed as a percentage of the pile height while sample X<sub>3</sub> has the least. The order of percentage loss in thickness is  $X_4 > X_2 > X_1 > X_5 > X_3$ . The dynamic loading test stimulates the two actions of walking (compression) and the shearing effect at the edge of the shoe. From the results obtained, it was observed that the percentage loss in thickness expressed as a percentage of the original pile height decreases with increasing surface pile density. Thus, the percentage loss in thickness of the different carpet may be due to the surface pile density, pile weight and the resilience properties of the pile material [17].

Table 7: Dynamic loading

Test sample	Loss of thickness	Standard variation	Coefficient of variation
X <sub>1</sub>	24	8.63	0.148
X <sub>2</sub>	28	7.07	0.085
X <sub>3</sub>	10	12.10	0.009
X <sub>4</sub>	30	4.12	0.051
X <sub>5</sub>	20	3.53	0,088

**Static Loading**

Table 8 shows the recovery of the tested samples after 24 hours recovery period. Sample X<sub>3</sub> shows a better recovery of the pile after 24 hours than the other samples, while sample X<sub>2</sub> shows the least recovery. The superior recovery of samples X<sub>3</sub> over the other samples may be due to its superior resilience property and its pile density. This is in agreement with the findings of [18].

Table 8: Static loading

Test Sample	Original Thickness (mm)	24 Hours Recovery (mm)	Standard Deviation	Coefficient of Variation
X <sub>1</sub>	6	1.05	0.0031	0.2952
X <sub>2</sub>	6	0.62	0.0002	0.0322
X <sub>3</sub>	8	1.07	0.0014	0.1372
X <sub>4</sub>	6	1.00	0.0034	0,3400
X <sub>5</sub>	6	1.04	0.0024	0.2307

**Electrostatic Properties**

The results in Table 9 show that the resistivity's of the pile materials were less than  $10^{10}$  ohms indicating that the carpet samples have good anti-static rating. Also, it was observed that the resistance along the carpet sample determined the rate of leakage of charges from the electrified carpet sample.

Table 9: Electrostatic Properties

Test Samples	Thickness (mm)	Along Carpet Longitudinal Axis	
		Resistivity (ohms) X 10 <sup>9</sup>	Time (sec) x 10 <sup>-2</sup>
X <sub>1</sub>	6	7.00	5.00
X <sub>2</sub>	6	5.00	2.50
X <sub>3</sub>	6	1.00	3.50
X <sub>4</sub>	6	1.50	1.80
X <sub>5</sub>	6	1.20	2.50

Static electricity in carpet has been higher from carpets containing stainless steel fibres [19]. The charges developed on shoe-sole materials and on human body have been investigated for carpets with and without steel fibres. The static electricity in carpets is affected by the following factors such as; relative humidity, fibre type, shoe-sole materials, carpet backing and underlay, carpet wear and human walking idiosyncrasies [15].

**CONCLUSION**

This study has been carried out in order to determine the physical properties of some selected carpets; and to assess the influence of these properties on the end use performance of these carpets. The end use performance of the carpets was greatly influenced by the constructional parameters, such as pile height, pile density, type of pile

material, pile weight, pile anchorage, type of latex coating, resilience property of the pile material and the colour fastness properties of the dyes employed. These parameters were mostly interwoven and tend to influence one another. The dynamic and static loading properties of the carpets were influenced by the resilience property of the pile material, while the pile density influences abrasion resistance and also, the dynamic and static loading properties of the carpets. Finally, the end use performances, that is, the service properties of the carpets are mainly dependent on the constructional variables amongst other variables.

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## Synthesis and Application of Monoazo Acid Dyes Derived from 5-amino-4-cyano-2-methyl-N-phenylthiophene-3-carboxamide on Chrome Tanned Leather and Nylon 6,6 Substrate

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### ABSTRACT

A series of new mono azo dyes have been synthesized in moderate yield from diazotization of thiophene and coupling with R, H, J and acid. The dyes were characterized by spectral studies. The dyeing potentials were assessed by applying them on chrome tanned leather and nylon 6,6 substrates. The dyes were found to give shade of red dark blue and dark maroon. The synthesized dyes gave a maximum absorption within the UV- visible region with  $\lambda_{max}$  515-597 nm and molar extinction coefficient ranging from  $(3.217-4.221 \times 10^4 \text{ l/mol}^{-1} \text{ cm}^{-1})$  and the infra-red spectra showed peaks at  $1582-1421 \text{ cm}^{-1}$  (due to azo group (N=N) stretching vibration), at  $3400 - 3200 \text{ cm}^{-1}$  due to (OH&NH) medium & broad stretching vibration), at  $2830-26-95 \text{ cm}^{-1}$  (due to aromatic group (C-H) stretching vibration), at  $1760-1640 \text{ cm}^{-1}$  (due to carbonyl group (C=O) stretching vibration), at  $1342-1266 \text{ cm}^{-1}$  (due to aromatic amine group (C-N) stretching vibration) and at  $2260-2222 \text{ cm}^{-1}$  (due to nitrile (C N) stretching vibration). The dye exhibited good depth, affinity, levelling and brightness properties on the substrates. The dye gave a good percentage exhaustion on nylon 6.6 and on chrome tanned leather substrates ranging from (78-87%) and (63-75%) respectively. The chrome tanned leather gave a wash fastness rating ranging from (3 -5) good, very good to excellent respectively and nylon 6,6 gave a wash fastness rating ranging from (4-5) very good to excellent fastness properties and the light fastness properties of chrome tanned leather gave a fastness rating (4-7) moderate, good, very good and excellent and rubbing fastness rating (4-5) very good to excellent on both substrates. The good shades observed may be due to better substantivity on chrome tanned leather and nylon 6,6 fabrics.

**Keywords:** Monoazo dyes, Synthesis, Exhaustion, Fastness properties, Diazotization

### INTRODUCTION

Azo-functionalized dyes bearing aromatic heterocyclic components [1] have attracted ever increasing attention in recent years due to their range of colour, brightness, simplicity, ease of manufacturing, good dyeing performance [2]. Azo compounds are a class of chemical components that are continually receiving attention in scientific research [3] [4] [5]. They are usually strong coloured compounds which can be intensely yellow, red, orange, blue and green etc. depending on the exact structure of the molecule. These dyes have characteristically good tinctorial strength as well as stability. Their preparation procedures by the classic diazotization and coupling reactions, are very simple and of low cost. They have found wide application in dyeing of protein fibers such as wool, angora, cashmere, and silk, as well the milk protein fiber called "Silk Latte", the soy protein fiber called "Soy Silk", and the synthetic polyamide fiber nylon [6 -14]. As a result of their colour, azo compounds are tremendously important as dyes and as Nigerian Journal of Textiles (NJT) Vol. 5: 28 – 36

pigments for a long time [13]. Infact, about half of the dyes in industrial use are azo dyes which are mostly prepared from diazonium salt [15-16]. Azo dyes, account for more than half of the dye which contain phenol as intermediates. The aim of the study was to synthesize novel monoazo acid dyes and the assessment of their fastness properties on chrome tanned leather and nylon 6,6 derive from 5-amino-4-cyano-2-methyl-N-phenylthiophene-3-carboxamide using coupling component such as R, H, J and acid.

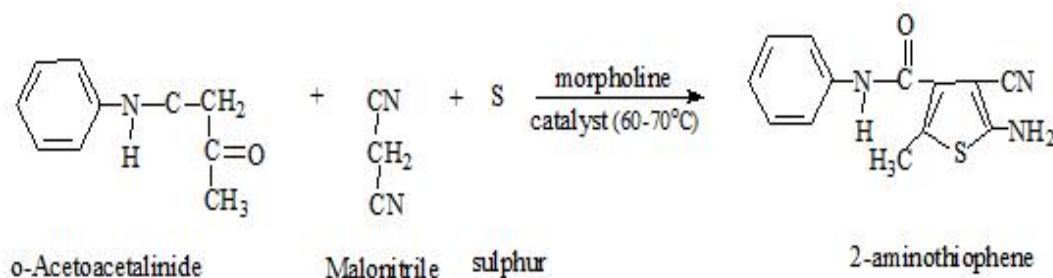
### MATERIAL AND METHODS

#### Materials

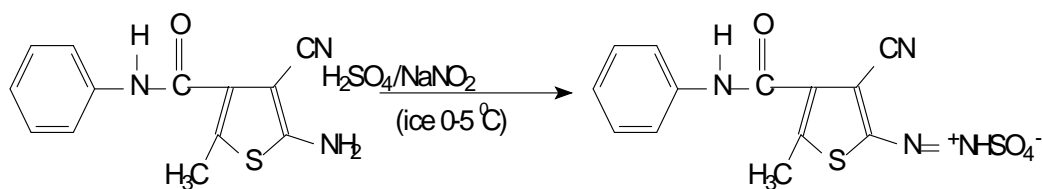
All commercial products were purchased from Sigma-Aldrich. Such as H-acid, R-acid, J-acid. Gamma-acid, acetic acid, anionic detergent. FT-IR spectrophotometer (Agilent CARRY 630 FT-IR Spectrophotometer), GC-MS spectrometry (7890B GC System),

### Synthesis of dye Intermediate (2-Aminothiophene Intermediate)

The dye intermediates were synthesized as follows: o-acetoacetanilide (20.0 g, 0.1 mol), Malononitrile (6.50 g, 0.1 mol), Sulphur (3.0 g, 0.1 mol), morpholine (8.50 g, 0.1 mol) were dissolved in 200 cm<sup>3</sup> of ethanol and introduced into a round bottom flask and heated at 60-70 °C for 3-4 hrs. The resulting thick dark solution is cooled and stored overnight in a refrigerator, The light brown powder obtained was crystallized from ethanol [17]



Equation 1: Schematic route for the synthesis of intermediate (Aminothiophene)



Scheme 1: Schematic route for the synthesis of diazonium salt

### Coupling of Diazonium Salt Solution [19]

H-acid (2.0g, 0.01 mol) is dissolved in 10 cm<sup>3</sup> of distilled water and the solution was cool to 0-5°C. To the well stirred solution, the freshly prepared diazonium salt solution was added drop wise over 45 minutes maintaining the temperature below 0-5°C with vigorous stirring. The stirring was done for 1 hour at 0-5 °C, maintaining pH 4-5 using sodium carbonate solution (10% w/v). The resulting dye was collected, wash several times with water to ensure a free acid and dry in an oven at 40 °C. The result was repeated for R, J and acid.

### Structures of Synthesized Dyes

#### Purification of the Dyes

The dyes were purified through the same process of recrystallization as highlighted above for the intermediate. Some of the dyes required a mixed solvent of ethanol, methanol /DMF (9/1 solvent mixture) according to a procedure suggested by [20]. The purity of each synthesized dyes were confirmed by melting point.

### Synthesis of Diazonium Salt Solution [18]

Sodium nitrite (1.38g, 0.02mol) was added portion wise to mixture 50cm<sup>3</sup> of nitrosyl sulphuric acid added to propionic acid and acetic acid (2:1) cooled to 0-5°C in ice bath and a mixture added to the (2.57g, 0.01) synthesized intermediate. The reaction mixture was then cooled to 0-5°C and then added portion wise and stirring continued for 2 hrs at 0-5°C to avoid excess heat that will destroy the diazonium salt. The resulting diazonium salt solution was obtained.

### Percentage Yield of Dyes and Intermediate

The percentage yield of the synthesized dyes and intermediates were determined using the formula shown below [21]

$$\% \text{ Yield} = \frac{M}{M} \div \frac{M}{M} \times 100$$

where;

- MP is the mass of the product
- MMP is the molar mass of the product
- MR is the mass of the reactant
- MMR is the molar mass of the reactant

### Melting Point of the Dyes

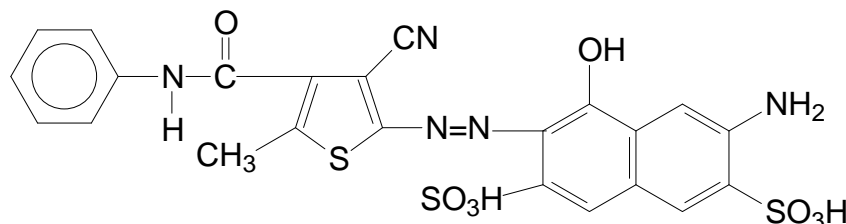
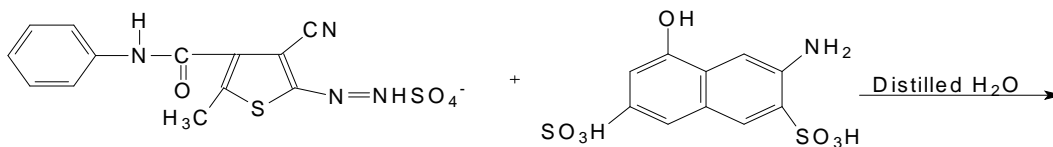
The melting point of each dye was determined using Gallenkamp melting point apparatus. Small amount of each dye was filled into a capillary tube each placed in to the apparatus, the melting point of each dye was obtained by consistently focusing on the apparatus as the apparatus gradually heated the dye in the tube.

### Dyeing of Nylon 6, 6 Fabric

For the nylon 6,6 fibre, a dispersing agent (anionic detergent) was used to facilitate the dyeing process. The fabric was wetted and thoroughly squeezed to

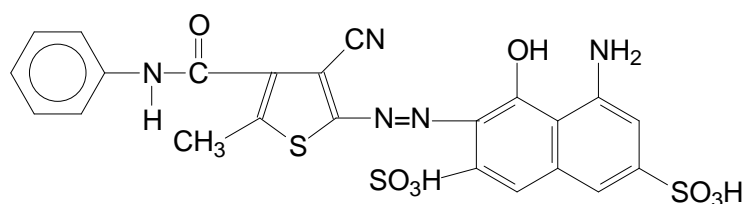
remove excess water. It was then immersed into the bath at 70°C and allowed to reach the boil within 15min. Dyeing was carried out for one hour at a temperature of 100°C with agitation. At the end of

the dyeing process, the substrate was removed, squeezed and rinsed thoroughly under running tap water and allowed to dry at room temperature [22].



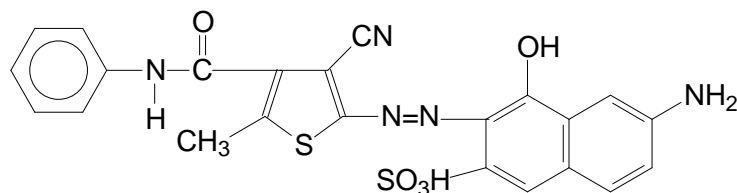
Dye 1a

3-amino-6-((3-cyano-5-methyl-4-(phenylcarbamoyl)thiophen-2-yl)diazenyl)naphthalene-2,7-disulfonic acid



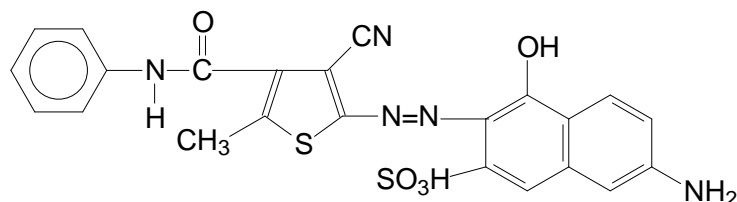
Dye 1b

5-amino-3-((3-cyano-5-methyl-4-(phenylcarbamoyl)thiophen-2-yl)diazenyl)-4-hydroxynaphthalene-2,7-disulfonic acid



Dye 1c

6-amino-3-((3-cyano-5-methyl-4-(phenylcarbamoyl)thiophen-2-yl)diazenyl)-4-hydroxynaphthalene-2-sulfonic acid



Dye 1d

7-amino-3-((3-cyano-5-methyl-4-(phenylcarbamoyl)thiophen-2-yl)diazenyl)-4-hydroxynaphthalene-2-sulfonic acid

### Dyeing of Leather

The synthesized dyes were used in dyeing of chrome tanned leather. The standard method of dyeing leather was followed using the recipe.

- i. 120 % of water (60 °C)
- ii. 2 % of dye
- iii. 0.1 % of formic acid

A solution of the dye sample (2%) was made with distilled water using heating mantle and then was raised to 60 °C. The pH of the bath was adjusted to 5.5 with formic acid. The leather samples were introduced into the bath and run for 1 hour in a mechanical shaker at a controlled speed [23].

### Application of dyes solution

A solution was prepared by dissolving about 1g of dried dye powder in 100mls of distilled water. To determine the qualities required for the experiment the following parameter was utilized.

Number of ml of stock solution required

$$= \frac{W \times P}{C}$$

where W = weight (in g) of sample to be dyed

P = percentage of dye to be used

C = concentration (%) of stock solution [24]

General procedure of dyeing chrome tanned leather:

1 % of dye (on weight of the materials)

50 + 2°C dyeing temperature

0.1 % of formic acid on weight of the dye

Time of dyeing: one hour

Liquor ratio 20:1

### Determination of Dye Bath Exhaustion

Dye uptake was determined by measuring the absorbance of diluted dye bath samples at the wavelength of the maximum absorption. The bath was sampled before and after dyeing. A 1ml aliquot was taken from the bath and diluted in 20ml of distilled water. This is to ensure that the absorbance falls within the readable range of 1.5. The percentage dye bath exhaustion (%E) for each substrate was calculated using equation below [25].

$$\%E = \frac{A_0 - A_1}{A_0} \times 100$$

where  $A_0$  and  $A_1$  are the absorbance at  $\lambda_{max}$  of the dye bath prior to dyeing and after dyeing respectively.

### Light Fastness of Dyed Substrates (Fastness to Sunlight) SLT 401 (IUF 401)

This test was carried out in accordance with the Society of Leather Technology and Chemists (SLTC). The test was carried out on the dyed

samples under daylight facing south sloping at an angle 45° from the horizontal. The procedures involved aligning both the eight dyed wool standards together with a 2 cm x 2cm cut pieces of the dyed samples on a card and covering one third of the setup with opaque cover.

Exposure was carried with regular inspection noting when the non-exposed part and exposed part of each dyed sample had contrast equivalent to grade 4 of ISO grey scale after which and opaque cover was placed covering part of the exposed sample part, exposure was continued till the blue standard 7 faded. The rating of the dyed samples was taken to be those equivalents to- that of standard dyed blue wool samples, which faded to the same extent as the specimen at the same time.

### Wash Fastness of the Dyed Substrates

This is to assess the dyed leather resistance to prolonged action of water. 2 x 2cm of the dyed leather was dipped into solution made by 5 g (0.5 %) of detergent powder into 200 cm<sup>3</sup> of distilled water. This was placed in water bath and the temperature allowed to rise to 40 °C. This was vigorously stirred for about 30 min and the leather was transferred to 100 cm<sup>3</sup> distilled water for washing and rinsing was done five times and dried. The change in colour was assessed by the standard grey scale .

### Rubbing Fastness Test of the Dyed Substrates

This method is intended for determining the behaviour of the surface dyed texture of a leather rubbing with felt. The leather to be tested is rubbed with pieces of structure wool felt under a given pressure with a given number of forward and backward motion. 2x2cm piece of the leather sample were cut and dipped on rub fastness tester (Machine Model STM). The number of rub on a fixed position was varied to assess the degree to fastness to rubbing. The change in colour of the felt and leather was assessed with the standard grey scale.

## RESULT AND DISCUSSION

**Table 1: Physical Properties of the Synthesized Dye Intermediate**

Name of Intermediate	Colour	Melting Point (°C)	Percentage Yield (%)	Molecular Weight (g/mol)	Molecular Formula
5-amino-4-cyano-2-methyl-N-phenylthiophene-3-carboxamide	Dark brown	188-194	97	257	C <sub>13</sub> H <sub>11</sub> ON <sub>3</sub> S

**Table 2: Physical Properties of the Synthesized Mono Azo Acid Dyes**

Dye no	Molecular Formula	Shade Crystal	Molecular Weight(g/mol)	Melting Point (°C)	Percentage Yield (%)
1a	C <sub>23</sub> H <sub>17</sub> O <sub>8</sub> S <sub>3</sub> N <sub>5</sub>	Red	587	200-202	76
1b	C <sub>23</sub> H <sub>17</sub> O <sub>4</sub> S <sub>3</sub> N <sub>5</sub>	Darkblue	587	191-194	62
1c	C <sub>26</sub> H <sub>17</sub> O <sub>5</sub> S <sub>2</sub> N <sub>5</sub>	Dark maroon	507	120-122	68
1d	C <sub>26</sub> H <sub>17</sub> O <sub>5</sub> S <sub>2</sub> N <sub>5</sub>	Red	507	212-214	82

**Table 3: Spectra Data of the Synthesized Mono Azo Acid Dyes using Dimethylformamide and Ethanol**

Dyes no	max in (nm) DMF	max in (nm) Ethanol	Molar Extinction Coefficient in (DMF) x 10 <sup>4</sup> (l/mol <sup>-1</sup> cm <sup>-1</sup> )
1a	530	515	4.181
1b	597	572	4.176
1c	580	577	3.217
1d	565	527	4.145

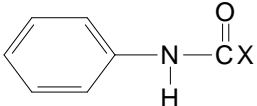
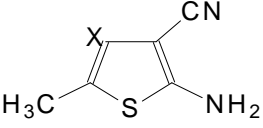
**Table 4 : Infrared Spectra of the Synthesized Intermediate**

Functional group	NH cm <sup>-1</sup>	C-H cm <sup>-1</sup>	C=O cm <sup>-1</sup>	C-N cm <sup>-1</sup>
Types of vibration	Stretching	Stretching	Stretching	Stretching
	3459.0	2967.0	1699.7	2214.0
	3354.6	2870.1	1666.1	
	3205.5		1602	

**Table 5: Infrared Spectra of Mono Azo Acid Dyes**

Functional group	OH & NH	Aromatic C-H	Carbonyl C-O	Aromatic amine C-N	Azo N=N
Types of vibration	Stretching	Stretching	Stretching	Stretching	Stretching
1a	3347.1		1636.3	1315.8	1488.6
1b	3377.0		1636.3	1367	1499.4
					1529.2
1c	3373.2		1703.4	1319.5	1468.5
			1654.9		
1d	3410.5		1636.3		1535.7
					1468.6

**Table 6: Gas Chromatography Mass Spectroscopy of Synthesized Intermediate**

Sample value	Fragmentation (m/z)	Calculated
		
Intermediate	(C <sub>7</sub> H <sub>6</sub> NO) <sup>+</sup> = 120 but m/z = 123 ± 3	257g/mol
		
	(C <sub>6</sub> H <sub>5</sub> N <sub>2</sub> S) <sup>+</sup> = 137 but m/z = 140 ± 3	



**Table 7: Percentage Exhaustion the Dyes on Nylon 6,6 and Chrome Tanned Leather Substrate**

Dyes No.	% E for Nylon 6,6	% E CTL
1a	80.10	63.10
1b	78.50	68.50
1c	84.00	75.30
1d	87.20	68.20

**Table 8: Light and Wash Fastness Properties of Chrome Tanned Dyed Leather**

Dyed No.	Light Fastness Rating	Wash Fastness Rating
1a	5-6	4-4
1b	5	4-5
1c	5-6	4-5
1d	3-4	3-4

**Table 9: Rubbing Fastness Properties on Chrome Tanned Dyed Leather**

Dyed no	Number of Rubs	Fastness Rating	Staining of Adjacent un-dyed Cotton
1a	100	5	4
1b	100	5	4
1c	100	5	3
1d	100	5	3

**Table 10: Rubbing Fastness Properties of Dyed Nylon 6.6 Fabric**

Dye No.	Dry Treatment Change in Colour	Wet Treatment Change in Colour	Staining	Change in Colour	Staining
1a	5	5	5	5	5
1b	5	5	5	5	5
1c	5	5	5	5	5
1d	5	5	5	5	5

**Table 11: Light and Wash Fastness Properties of Nylon 6,6 Fabric.**

Dyes	Light Fastness Rating	Wash Fastness Rating	Staining on of Adjacent undyed Fabric
1a	6	3	3
1b	6	3	3
1b	6	4	4
1d	7	4	3

### The Spectral Data Analysis of Synthesized Monoazo Acid Dyes

The monoazo acid dye as shown on Table 3: 1a, 1b, 1c and 1d absorbed at 530, 597, 580 and 565 nm respectively in dimethylformamide. Dye 1b for instance is more bathochromic compared with dye 1a, 1c and 1d. Dye 1b was synthesized from the coupling component of 8- amino-1-naphthol 3-6-disulfuric acid. This is followed by dye 1c containing 2, amino-8-naphthol sulfuric acid as coupling component which absorbed at 580nm and is bathochromic compound with dye 1a and 1d which absorbed at 530 and 565 nm containing 2-

naphthol-3-6 disulfuric acid and 2-amino-5-naphthol-7-sulfuric acid respectively, The substituent shifts of maximum wavelength ( $\lambda_{max}$ ) absorption to shorter wave length, with reduced absorbance energy, intensified the colour of dyes. The colour change observed for each dye is due to the oscillation of electrons and the presence of additional substituents. The higher HOMO energy values show the molecule is a good electron donor, in other hand, the lower HOMO energy values indicate that, a weaker ability of the molecules for donating electron. LUMO energy presents the ability of a molecule for receiving electron.

Maximum absorption moves to longer wavelength as the amount of delocalization increases, therefore the maximum absorption moves to shorter frequency, the absorption needing less energy gap between the bonding and anti-bonding orbitals [24].

#### Assessment of FT-IR Group Absorption Spectroscopic Properties of Synthesized Intermediate and Acid Dye

It is obvious as shown on Table 4: that dye intermediate which is 2-aminothiophene gave absorption peaks due to (N-H) group at  $3459.0\text{ cm}^{-1}$ ,  $3354.6\text{ cm}^{-1}$ , and  $3205.5\text{ cm}^{-1}$ , (C-H) group at  $2967\text{ cm}^{-1}$ , (C=O) group at  $1699.7\text{ cm}^{-1}$ ,  $1666.1\text{ cm}^{-1}$ , and  $1602\text{ cm}^{-1}$ , and (C≡N) group at  $2214.0\text{ cm}^{-1}$ , stretching vibration. Dye 1a-d were obtained by diazotization of 2-aminothiophene and coupling with 2-naphthol-3-6 disulfuric acid, 8-amino-1-naphthol-3-6, disulfuric acid, 2-amino-8-naphthol sulfuric acid and 2-amino-5-naphthol-7 sulfuric acids respectively. They absorption peaks due to (O-H and N-H) group at  $3347\text{ cm}^{-1}$ , (C=O) at  $1636.3\text{ cm}^{-1}$ , (C-N) at  $1315.8\text{ cm}^{-1}$ , and (-N=N-) group at  $1488.6\text{ cm}^{-1}$ , stretching vibration in dye 1a. dye 1b which is obtained by diazotization of diazonium salt coupling with 8-amino-1-naphthol-3-6-disulfuric acid showed absorption peaks due to (O-H and N-H) group at  $3377.0\text{ cm}^{-1}$ , (C=O) group at  $1636\text{ cm}^{-1}$ , (C-N) at  $1367\text{ cm}^{-1}$ , and (-N=N-) group at  $1499.4$  and  $1529.2\text{ cm}^{-1}$ , stretching vibration. Dye 1c which is obtained by coupling diazonium salt with 2-amino-8-naphthol sulfuric acid gave absorption peak due to (O-H) and (N-H) group at  $3373.2\text{ cm}^{-1}$ , (C=O) group at  $1703.4\text{ cm}^{-1}$ , and  $1654.9\text{ cm}^{-1}$ , (C-N) group at  $1319.5\text{ cm}^{-1}$ , and (N=N) group at  $1468.5\text{ cm}^{-1}$  stretching vibration. Dye 1d which obtained by coupling diazonium salt with 2-amino-5-naphthol-sulfuric acid showed absorption peak due to (O-H and N-H) group at  $3410.5\text{ cm}^{-1}$ , (C=O) group at  $1636.3\text{ cm}^{-1}$ , (-N=N-) group at  $1535.7\text{ cm}^{-1}$ , and  $1468.6\text{ cm}^{-1}$ , stretching vibration.

#### Molar Extinction Coefficient (€)

From the result indicated on Table 5: it is clear that the dyes possess high molar extinctions coefficient. From the result shown, it can be deduced that majority of the synthesized dyes gave molar extinction coefficient values greater than  $4.181 \times 10^4\text{ mol}^{-1}\text{ cm}^{-1}$ . The higher molar extinction coefficient value of the synthesized dyes probability for more electronic transition and deepness or tinctorial of the shade on the substrate.

#### The Gas Chromatography Mass Spectrophotometry of the Synthesize Dye Intermediate

This elucidate structural information of the synthesized intermediate molecular ion of the intermediate is shown in Table 6; Which underwent fragmentations to produce peaks at  $120\text{ g/mol}$  but  $m/z$  is  $123 \pm 3$  and  $137\text{ g/mol}$  but  $m/z$  is  $140 \pm 3$  corresponding positive charge fragment are  $\text{C}_6\text{H}_7\text{NO}^+$  and  $\text{C}_6\text{H}_5\text{N}_2\text{S}^+$  respectively and its molecular mass is ( $257\text{ g/mol}$ ). The difference between the experimental ( $263\text{ g/mol}$ ) and the calculated value ( $257\text{ g/mol}$ ). May be due to isotropy relative abundance  $\pm 6$ . The slight difference in numerical value of the molecular weight of the intermediate can be attributed to certain elements with high isotopic abundance [25].

#### Percentage Exhaustion on Nylon 6, 6 and of Chrome Tanned Leather Substrates

The percentage exhaustion and fixation reported according to [26] of 2% (o.w.f) dyeing of nylon 6.6 and percentage exhaustion on chrome tanned leather substrate is summarized in Table 7. Dye 1a-1d which is monoazo acid dyes is obtained by coupling diazonium salt (2-aminothiophene) with coupling component such as 2-naphthol-3-6-disulfonic acid, 8-amino-1-naphthol-3-6 disulfuric acid, 2-amino-8-naphthol sulphonic acid and 2-amino-5-naphthol-7-sulphonic acids. Indicates higher percentage exhaustion values on nylon 6,6 ranges from 78-87 while the percentage exhaustion on chrome tanned leather ranges 75-63% in which dye 1d show maximum exhaustion and dye 1c, 1a, and 1b shows minimum percentage exhaustion values 84, 80, 78% respectively due to the amorphous nature of the fabric. While dye 1c show maximum percentage exhaustion value 75% and dye 1b, 1d, and 1a shows minimum percentage exhaustion values 68, 68 and 63% respectively on chrome tanned leather respectively due to the crystalline nature of the substrate. Which indicate high levelness, penetration, substantivity and affinity due to solubility and high rate of diffusion of dye molecules.

#### Light Fastness

Light fastness rating of the dyed fabric is shown in Table 8 and 10. The test is carried out by exposing each dyed sample to daylight alongside eight blue wool standards. It was observed that light fastness of dye 1a, 1b, 1c and 1d which is obtained by coupling diazonium salt with 2-naphthol-3-6 difulfonic acid, 8-amino-1-naphthol-3-6 disulfonic acid, 2-amino-8-naphthol sulfonic acid and 2-amino-5-naphthol-7-sulfonic acid to obtained monoazo acid dyes gave a light fastness rating (3-5) moderate, good and very good on chrome tanned

leather which is attributed to hydrophobic texture of the substrate, while light fastness rating (6-7) very good and excellent on nylon 6,6 is attributed to hydrophilic nature of the fabric.

#### Washing Fastness

Tables 8 and 10 shows the wash fastness of the dyes coupled with diazonium salt, 2-naphthol, 3-6 disulfonic acid, 8-amino-1-naphthol; 3-6. Disulphonic acid, 2-amino-8-naphthol sulphonic acid and 2-amino-5-naphthol-7-sulphonic acid. These dyes gave purple, brown, pink, deep blue to khaki hues with brighter and deeper shades, high tinctorial strength and excellent levelness on the substrates ranging from good (3), very good (4), to excellent (5). Dye 1a-1d gave a wash fastness rating on chrome tanned leather (3-5) good, very good and excellent which is attributed to dyed in difficult to wash out, while on nylon 6,6 gave a wash fastness rating (3-4) good to very good. It could be seen that chrome tanned leather gave better wash fastness than nylon 6,6, which could be as result of the texture of the fabric which in nylon 6,6 involved high diffusion and penetration and in chrome tanned leather which its interweave involve slow diffusion and penetration.

#### Colour Fastness to Rubbing of the Synthesized Dyes on Chrome Tanned Leather and Nylon 6,6 Substrates

Rubbing fastness was designed to determine the degree of colour which may transfer from the surface of a colour fabric to a specific test substrate for rubbing. Rubbing fastness of all the dyes was very high ranging from (4-5) and the staining rating of adjacent fabric was (3-4). The results obtained from the rubbing fastness test of the dyed fabric is given in Table 11. All the dyed sample gave a fastness rating (4-5) very good to excellent results. This may be due to the solubilisation of a part of the dye and its migration to the surface of coloured fabric (Clark, 2011) substantivity, penetration, levelness and durability in use.

#### CONCLUSION

A series of new monoazo dyes have been synthesized in moderate yield from diazotisation of 2-aminothiophene and coupling with H, R, J and acids and their different properties examined in solution and applied on chrome tanned leather and nylon 6,6 substrates. The dye gave wide range of fast shade like, red, darkblue and maroon. The chrome tanned leather gave a wash fastness rating ranging from (3-5) good, very good to excellent respectively and nylon 6,6 gave wash fastness rating ranging from (4-5) very good to excellent fastness properties. The light fastness properties of chrome tanned leather gave fastness rating of (4-7)

moderate, good, very good and excellent as well as rubbing fastness rating from (4-5) very good to excellent on both substrates.

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## **Information Communication Technology and Dress Culture among Senior Secondary School Students in Osun State, Nigeria**

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### **ABSTRACT**

*This study was carried out to examine the involvement of Senior Secondary School Students on ICT related activities as well as the influence of ICT on their clothing culture. Two public and two private schools were randomly selected. Two hundred students completed the questionnaire. Relevant information was obtained using structured questionnaire. Data was analysed using descriptive and inferential statistics. The respondents were equally distributed in terms of gender. Based on their perception of ICT on dress culture, 74.5% of the respondents agreed that frequent use of ICT has increased their level of fashion consciousness, while 60.5% were motivated by the images and dressing styles in magazines, on TV and the internet. The result further shows that male students were significantly more engaged in ICT related activities than females ( $t = 1.29, P < 0.05$ ), whereas there is no significant difference in the involvement in ICT activities between private and public school students ( $t = 0.325, P > 0.05$ ). The study adds to existing literature on the rationale for clothing selection and factors affecting such, one of which is ICT. Since ICT has influence on dressing, our traditional dress should be promoted on mass media.*

**Keywords:** Dress Culture, Adolescent Clothing Selection, Fashion Trend, ICT, Role Model

### **INTRODUCTION**

Information and Communication Technology (ICT) includes any communication device and systems such as the internet, television, cell phones, digital cameras etc that enhance greater access to information [1]. It is composed of the computer hardware and software as well as other devices. ICT has influenced many areas of human life and endeavour such as communication, transportation, education and business as well as the life style of youths. Youths of today live in a world characterized by dramatic cultural, economic, social and educational differences [2]. Young people are often first adopters of new technologies. They have more access to internet, mass media and telephone as all these provide opportunities for informal education as well as job opportunities. They are the most exposed to ICT because of their passion for information.

The importance of clothing and appearance cannot be overemphasised. Protection from harsh weather, beauty and aesthetics are among the roles played by dress. It enhances identity and communicates to the society. Dress practices can shape individual self-concept, influence behaviour and values. They serve as symbols of mood as well as socio-economic status [3].

Dressing styles, fashion consciousness and choice of role model are key areas that ICT influences

among youths. They tend to select their favourite stars and emulate them. Studies on clothing selection have shown that pre-adolescents compare their physical attractiveness with that of models in advertisements [4]. The television (TV) has greater influence on adolescent's clothing selection than any other media form; however, younger male adolescents indicate more influence on clothing choice from television than females [5].

Globally, most youths have access to broadcast technologies [6]. In Chile for example, 62% of schools are online and surf the net through mobile phones [7]. In Nigeria, private and public schools are adopting the e-learning system. Most secondary school students possess phones and laptops. They are involved in browsing and visiting various websites on sports, fashion, movies and games. The dressing and appearance of the youth therefore tend towards western styles with little traditional touches. There is likelihood that the dressing of these youths is being influenced by ICT. This study therefore sets out to examine the involvement of Senior Secondary School Students in ICT related activities as well as the influence of ICT on their clothing culture.

### **METHODOLOGY**

The population of the study consist of all private and public secondary school students in Ife-East Local government. Four secondary schools

including two private and two public schools were randomly selected. Fifty students in senior level were selected randomly (Senior Secondary School 1-3). A well-structured questionnaire was administered to elicit information on ICT activities and its influence on dress culture. Data collected were analysed using descriptive and inferential statistics. Pearson Correlation was used to measure association between dress culture and use of ICT. T-test was used to measure significant difference in the level of involvement in ICT between private and public schools.

## RESULTS

### Personal Characteristics of Students

Table 1 shows that most (52.0%) of the respondents are within the age of 16-20years. 50.0% of the respondents were male while 50.0% were females. 25.0% of the respondents attended Acada High School Modakeke, 25.0% attended Modakeke High School, and 25.0% attended Saint Timothy Academy while 25.0% attended The Apostolic Grammar School. The table further shows that 79.0% of the respondents were Christians, 20.0% were Muslim and 1.0% were traditional worshippers. The distribution of students in SSS1, SSS2 and SSS3 is as shown in the Table. Overwhelming majority of the respondents (99%) was of Yoruba origin. This could be associated with the fact that Modakeke is located in Yoruba land.

### Involvement in ICT Related Activities

Table 2 shows that 79.0% of the students were computer literate. 79.5% had access to ICT facilities and 93.5% browsed with their phones. It was also observed that 78.0% chatted with friends online, 64.5% had face book accounts and 77.0% had 2go account. Those that read fashion magazines accounted for 90.5% while 32.0% downloaded games, images and movies from the internet.

From Table 5, it is observed that about 60.0% of female students wore skirts and blouses always and 87.0% wore under-wears. Sometimes, 58.0% wore *iro* and *buba*, 45.0% wore suits, 40.0% wore fitted shirts and 37.0% wore sport wears. The Table further shows that 50.0% did not wear *oleku*, 58.0% did not wear off shoulder, 60.0% did not wear spaghetti top, 63.0% did not wear *jalamia*, 69.0% did not wear jackets and trousers, 68.0% did not wear carrot trousers, 82.0% did not wear baggy trousers, 64.0% did not wear knickers, 48.0% did not wear sport wears and 76.0% did not wear crazy jeans.

Table 6 shows there is no significant difference in the involvement in ICT by male and female students. Since the significant value of 1.29 is greater than 0.05, it can be safely concluded that the average of 0.40 male students were more engaged in ICT related activities than female students. Thus, there is a significant difference in the level of ICT involvement between male and female students in Ife-East local government.

Table 4 shows that 66.0% of male respondents did not wear *agbada*, 49.0% did not wear *buba* and *sokoto* and 60.0% did not wear *jalamia*. 38.0% did not wear pant (a readymade trousers usually called pant trousers among students), 47.0% did not wear baggy trousers and 70.0% did not wear crazy jeans. The Table also reveals that 56.0% of male students sometimes wore suits. 53.0% wore jacket and trousers, 60.0% wore fitted shirts, 53.0% wore sport wears, 55.0% wore carrot trousers and 85.0% wore under wears.

**Table 1: Distribution of Respondents by Personal Characteristics**

Variable	Frequency (200)	Percentage (%)
<b>Age</b>		
11-15	95	47.5
16-20	104	52.0
21-25	1	0.5
<b>Sex</b>		
Male	100	50.0
Female	100	50.0
<b>School</b>		
Acada High School	50	25.0
Modakeke High School	50	25.0
Saint Timothy Academy	50	25.0
The Apostolic Grammar School	50	25.0
<b>Class</b>		
SSS1	52	26.0
SSS2	51	25.5
SSS3	97	48.5
<b>Religion</b>		
Christianity	158	79.0
Islam	40	20.0
Traditional	2	1.0
<b>Ethnicity</b>		
Yoruba	198	99.0
Igbo	2	1.0
Hausa	0	0.0

**Table 2: Distribution of respondents by involvement in ICT activities**

<b>Variable</b>	<b>Frequency (200)</b>	<b>Percentage (%)</b>
Computer Literacy	158	79.0
Access to ICT facilities	159	79.5
Face book account	129	64.5
2go account	154	77.0
Read news paper	181	90.5
Read fashion magazine	144	72.0
Watch movies	181	90.5
Browse the internet in the café	168	84.0
Chatting with friends on line	156	78.0
Learning computer application program	80	40.0
Browsing with phone	187	93.5
Playing games	146	73.0
Listen to radio	184	92.0
Downloading games, images and movies	64	32.0

**Table 3: Distribution of Respondents by Perception of Influence of ICT on Dress Culture**

<b>Statements</b>	<b>Strongly Agree (%)</b>	<b>Agree (%)</b>	<b>Undecided (%)</b>	<b>Disagree (%)</b>	<b>Strongly Disagree (%)</b>
ICT has influenced the way I dress	34.0	41.5	4.5	11.5	8.5
Frequent use of ICT has increased my level of fashion consciousness	29.0	45.5	4.5	14.0	7.0
Buy clothes that look similar to what I have seen on the TV and other media	25.0	33.5	8.5	22.0	11.0
Dress like super stars in the movies and sports	24.5	29.5	9.0	26.0	11.0
Motivated by the images and dressing pattern in magazines, on TV and the internet	23.0	37.5	9.50	19.0	11.0
Sample for dress online before purchase	16.0	29.5	8.0	22.5	24.0
ICT makes me choose clothes that look sexy	18.0	23.0	12.0	24.0	23.0
ICT influence me to choose cloth that expresses my personality	21.0	36.5	11.5	17.5	13.5
ICT motivates me to choose clothes that express my ethnicity	30.5	30.5	12.5	17.0	9.5
Friendson line influence my dressing	28.0	31.5	12.0	14.5	14.0

**Table 4: Distribution of male respondents by fashion trend**

Dress Items	Always	Sometimes	Never
<i>Agbada</i>	5.0	29.0	66.0
<i>Buba and sokoto</i>	5.0	46.0	49.0
<i>Jalamia</i>	23.0	17.0	60.0
Suits	29.0	56.0	15.0
Jacket and Trousers	53.0	38.0	9.0
Fitted shirts	60.0	28.0	12.0
Carrot trouser	55.0	21.0	24.0
Pant trousers	33.0	29.0	38.0
Baggy trousers	17.0	36.0	47.0
Three quarter	33.0	54.0	13.0
Knickers	36.0	44.0	20.0
Sport wears	53.0	32.0	15.0
Crazy jeans	19.0	11.0	70.0
Underwear	85.0	5.0	10.0

**Table 5: Distribution of Female Respondents by Dress Items**

Dress Items	Always	Sometimes	Never
<i>Iro and buba</i>	5.0	58.0	37.0
<i>Oleku</i>	15.0	35.0	50.0
<i>Jalamia</i>	18.0	19.0	63.0
Hijab	15.0	10.0	75.0
Suits	39.0	45.0	16.0
Skirt and blouse	65.0	22.0	13.0
Off shoulder	12.0	30.0	58.0
Spaghetti top	20.0	20.0	60.0
Jacket and Trousers	14.0	17.0	69.0
Fitted shirts	32.0	40.0	28.0
Carrot trouser	16.0	16.0	68.0
Baggy trousers	3.0	15.0	82.0
Three quarter	9.0	27.0	64.0
Knickers	16.0	23.0	61.0
Sport wears	15.0	37.0	48.0
Crazy jeans	10.0	14.0	76.0
Underwear	87.0	9.0	4.0

Table 7 shows that there is no significant difference in the involvement in ICT activities between private and public school students. Since the significant value of the test is greater than 0.05, it can be concluded that the average of 0.32 Private school students in Ife-East local government were more involved in ICT related activities than Public school students. Thus, a significant difference exists in the involvement in ICT activities between private and public schools students in Ife-East Local Government.

Table 8 shows that there is no significant relationship between ICT usage and dress culture. The correlation reported in the Table above is negative, although not significantly different from 0 because p- value of 0.401s is greater than 0.10. This suggests that there is an appreciable effect of ICT usage on dress culture of senior secondary school students in Ife-East Local Government.

**Table 6: Involvement in ICT by Male and Female Students**

Sex	N	Mean	SD	Df	Sig. value (p)	t- test
Male	100	13.24	1.7759	198	1.29	1.52
Female	100	12.84	1.9317	198		

**Table 7: Involvement in ICT Activities Between Private and Public School Students**

Schools	N	Mean	SD	Df	Sig. value (p)	t-test
Public	100	12.88	1.9189	198	0.225	1.217
Private	100	13.20	1.7978	198		



**Table 8: Relationship between ICT usage and dress culture**

		ICT Usage	Dress Culture
ICT Usage	Pearson correlation	1	-.060
	Sig. (2-tailed)		.401
Dress Culture	Pearson correlation	-.060	1
	Sig. (2-tailed)	.401	

## DISCUSSION

Majority of senior secondary students were found to be involved in ICT related activities such as browsing the internet, chatting, with friends, downloading games, images, and films, playing games, watching the TV, listening to radio etc. However, male students were found to be more involved in ICT than female with a mean difference of 0.40. In the same vein, private school students were also found to be more engaged in ICT than public school students with a mean difference of 0.32. Nevertheless, the active involvement of the students was in concordance and partial contrast to [6] who proposed that, the great majority of world's young people have access to broadcast technologies. The majority of young people have access to telephony, but still a small minority that have access to the web. Access to radio and TV has long been widespread, as many as 80 percent of the population of the developing world listens to the radio at least once a week.

Response to questions that tested the level of agreement to the influence of ICT on dress culture showed that ICT has a great influence on dress culture of secondary school students. This was justified by the agreement of about 75.5% of students whose dress had been influenced by ICT. The students agreed that frequent use of ICT had increased their level of fashion consciousness. ICT and media can provide information on fashion, beauty and body satisfaction, and has the potential to provide either positive or negative images for adolescents in the process of their social development and emerging sense of identity, of which fashion decisions are a part [9]. About 60.5% of secondary school students were more motivated by the images and dressing patterns in magazines, on TV and the internet. Majority of them even had online friends suggesting dresses for them. The study is in line with [10].

## CONCLUSION

From this study, it was found that ICT has a great influence on dress culture of senior secondary school students. A significant difference exist in the involvement in ICT activities between private and public schools students in Ife-East Local Government. A significant relationship was also found between the use of ICT elements and dress culture. A significant difference was found in the

level of involvement in ICT activities by male and female students. Male students tend to be more involved in ICT than female students.

Based on the result of this study, it is recommended that further studies should be carried out to assess the influence of ICT on dress culture of senior secondary school students in other part of Nigeria and since the youth copy western dress on media, our traditional culture of dressing should be promoted through ICT

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## Study of the Adsorption of Anionic Dye onto Activated Bone Char

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### ABSTRACT

*The adsorption of reactive dye onto activated carbon obtained from cow bones was investigated. The effects of dye concentration and pH on the adsorption of the dye onto the activated carbon were similarly investigated. The optimum adsorption was obtained at dye concentration of 45 mg/L and pH 6 respectively. Langmuir and Freundlich adsorption isotherms were employed to provide qualitative information on the capacity of the adsorbent as well as the nature of the solute-surface interaction. The correlation coefficient obtained for Freundlich isotherm ( $R^2 = 0.6369$ ) and Langmuir isotherm ( $R^2 = 0.5823$ ) indicates that Freundlich isothermal model is more fit for the adsorption process. By implication, the adsorption of Reactive Yellow FG onto the activated carbon can be referred to as a multilayer adsorption since the assumption of Freundlich's equation is based on heterogeneous distribution of active sites.*

**Keywords:** Adsorption, Langmuir Isotherm, Freundlich Isotherm, Wastewater, Reactive Yellow FG

### INTRODUCTION

The presence of colour and colour-causing compounds has always been undesirable in water for any use. It is therefore, not at all surprising to note that the colour in wastewater has now been considered as a pollutant that needs to be treated before discharge. Thus, colour removal is one of the challenges faced by the textile finish, dye manufacture, pulp and paper industries, among others. Adsorption process is one of the most effective and economically feasible methods for the removal of dyes from aqueous solutions [1].

Adsorption isotherms are mathematical models that describe the distribution of the adsorbate specie among liquid and solid phases, based on a set of assumptions that are related to the heterogeneity/homogeneity of the solid surface, the type of coverage, and the possibility of interaction between the adsorbate specie [2].

There are several isothermal models which includes: Langmuir, Freundlich, Temkin, Sips and Redlich-Peterson isothermal expressions. The studies of adsorption isotherms are carried out on two well-known isotherms, the Langmuir and the Freundlich adsorption isotherm models and the applicability of their isotherm equation is compared by judging from the values of their correlation coefficients ( $R^2$ ) [3].

Several factors affect the adsorption of dyes onto activated carbon, therefore this research is focused on the investigation of the effect of dye concentration and solution pH on the adsorption efficiency of bone char activated with  $\text{CaCl}_2$  on Reactive Yellow FG dye.

### MATERIALS AND METHOD

#### Materials

Commercial grade chemicals were utilized all through the research. The NaOH (96.0% purity) and the HCl (37.0% purity) were purchased from Guangdong Guanghua chemical factory China, Reactive Yellow FG dyestuff obtained from African Textile Manufacturers (ATM) Limited, Kano was purchased from PJS products Ltd, Lagos.

#### Equipment

The major equipment used for this work are: UV-Visible spectrophotometer (Cary 300), pH-meter, electronic weighing balance, thermometer, water bath constant temperature vibrator (SHA-C).

#### Preparation of Stock Solution of Dye

Stock solution of about 1g/L was prepared by properly dissolving about 1g of the dye in distilled water inside a volumetric flask and filling it up to the 1000ml mark, this is allowed to stand for 24hours [4].

### Wavelength of Maximum Absorption ( $\lambda_{max}$ ) and Calibration Curve

The stock solution (3ml) was repeatedly diluted with distilled water until its absorbance could be read by the UV-visible spectrophotometer, after scanning, the  $\lambda_{max}$  of 416nm was obtained for the dye. Using serial dilution formula (equation 1), five standard concentrations were obtained from the stock solution and used in plotting the calibration curve alongside the calibration equation which are used to determine the concentration of the dye solution for subsequent adsorption process.

$$C_1V_1 = C_2V_2 \quad (1)$$

where  $C_1$  = original concentration of stock solution,  $V_1$  = volume required from the stock solution,  $C_2$  = concentration required,  $V_2$  = volume of the new concentration required. The removal efficiency (%) and adsorption capacity ( $Q_e$ ) in mg/g were calculated using equation 2 and 3 respectively.

$$R_e \quad (\%) = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (2)$$

$$Q_e = \frac{(C_0 - C_e)}{m} \times V \quad (3)$$

where  $C_0$  is the initial concentration of adsorbate,  $C_e$  is the equilibrium concentration of adsorbate,  $V$  is the volume of adsorbate,  $m$  is the mass of the adsorbent.

### Assessment of Effect of Dye Concentration on the Adsorption of the Dye

The activated carbon used for the adsorption was produced using the method of [5]. By adopting [4], a predetermined weight of the adsorbent (0.5g) was constantly agitated with 25ml of the dye solutions for 100 min for each batch experiment with an initial dye concentration of 35, 45, 55, 65 and 75mg/L respectively.

### Assessment of Effect of pH on the Adsorption of the Dye

The effect of pH was studied by agitating 0.5g of the adsorbent with 25 ml of the dye solutions for 100 min with a pH of 5, 6, 7, 8 and 9 respectively. Variation of pH was accomplished by drop-wise addition of HCl or NaOH (0.1M in each case) with the aid of a dropping pipette until required pH is indicated by the pH meter.

## RESULTS AND DISCUSSION

### Effect of Dye Concentration on the Adsorption of the Dye

Fig. 1 shows the effect of dye concentration. It was observed from Fig. 1 that at an initial concentration of 35mg/L a removal capacity of

56.28% was recorded at equilibrium and by increasing the concentration to 45mg/L, the highest adsorption (62.30%) was recorded, thus an increase in concentration has led to an increase in removal capacity.

This phenomenon may be attributed to the driving forces that needed to be overcome for the resistance of mass transfer between the aqueous and solid phases as observed by [6].

However, upon further increase in dye concentration from 45 - 65mg/L, there was a drop in the removal capacity. The reduction may be either due to the saturation of the active binding sites on the adsorbent surface at higher concentrations [7] or due to increase in repulsive force between dye molecules in the solution thus reducing the removal efficiency [8].

### Effect of pH on the Adsorption of the Dye

The pH is the most important factors affecting adsorption as it contributes not only to surface charge of adsorbent and the degree of ionization of the material present in the solution but also to the dissociation of functional groups on the active sites of the adsorbent [3, 9, 10].

In Figure 2, the removal capacity was seen to be higher at acidic pH (5 -6.5) with the optimum colour removal capacity (63.07%) recorded at pH 6. In the basic region (pH above 7), removal capacity was observed to decrease with increasing pH with minimum colour removal capacity (56.03%) recorded at pH 9. It is worth noting that the hydroxyl apatite structure of bone char presents it with a negatively charged surface, however activation with  $CaCl_2$  essentially confers positive charge to its surface.

According to the explanation offered by [6], it can be deduced that decrease in pH of aqueous solution can lead to the decrease in negative charge density on the adsorbent surface. This view will yield positive results for adsorption of anionic dyes (reactive dye in this case). Therefore, the increase in dye adsorption observed at acidic pH may be related to the electrostatic interactions between the positively charged adsorbent surface and negatively charged reactive dye anions. On the other hand, at basic pH (above 7), the repulsive electrostatic forces between the dye anions and the negatively charged adsorbent surface (due to increased pH) may be responsible for the consequent decrease in adsorption observed.

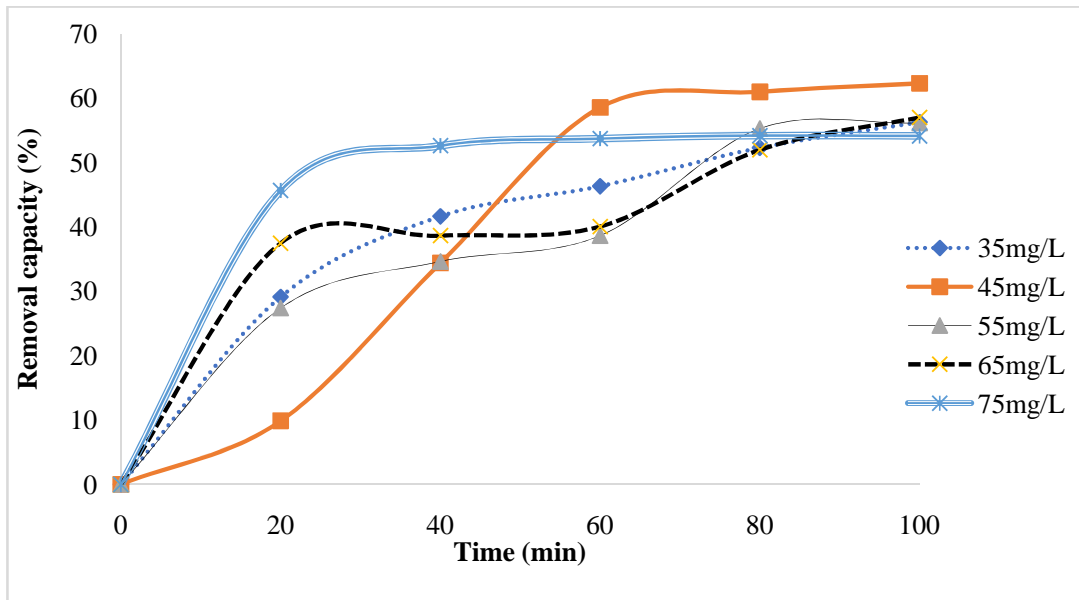


Figure 1. Effect of Initial Dye Concentration on the Adsorption of the Dye

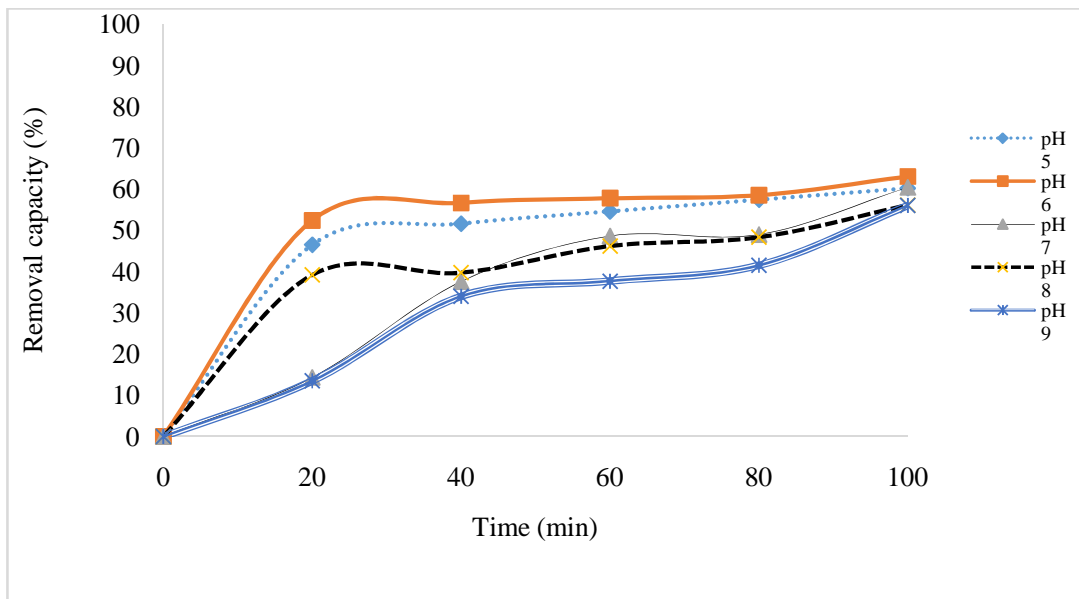


Figure 2. Effect of pH on the Adsorption the Dye

### Adsorption Isotherm

Adsorption isotherms provide qualitative information on the capacity of the adsorbent as well as the nature of the solute-surface interaction [11]. To describe the equilibrium adsorption process, the two theoretical models used in comparing the experimental results are Langmuir and Freundlich isotherms and all the parametric values calculated from their respective curves are shown in Table 1.

Table 1. The Values Obtained of Respective Isotherm Parameters for the Adsorption of the Dye

Isothermal Model	Parameters	Values
Langmuir Isotherm	$Q_m$ (mg/g)	94.34
	$K_L$ (L/mg)	105.99
	$R_L$	0.0003
	$R^2$	0.5823
Freundlich Isotherm	$K_F$ (mg/g)	4.915
	1/n	0.8025
	N	1.2461
	$R^2$	0.6369

Figures 3 and 4 respectively represent the Langmuir and Freundlich isothermal models for adsorption of the dye. The Langmuir constants  $K_L$  and  $R_L$  were obtained from the slope and intercept of the linear plot of  $1/Q_e$  versus  $1/C_e$  while the Freundlich constants  $K_F$  and  $n$  were also obtained from the intercept and the slope of the linear plot of  $\ln Q_e$  versus  $\ln C_e$ . The value of separation factor obtained ( $R_L=0.0003$ ) reflects that the experimental data agrees with the Langmuir model ( $0 < R_L < 1$ ) according to [12] and [13]. Similarly, the value of the adsorption intensity ( $n=1.2461$ ) obtained for the Freundlich model being greater than unity reflects that the experimental data agrees with the Freundlich model.

This is because the Freundlich constant 'n' gives an idea of the favorability of the adsorption process, so the value of n should be less than 10 and higher than unity for a favorable adsorption condition [11].

However, the correlation coefficient for Freundlich isotherm ( $R^2 = 0.6369$ ) being greater than that of Langmuir model ( $R^2 = 0.5823$ ) indicates that Freundlich model fits the adsorption process better. Therefore, the adsorption of Reactive Yellow FG can be referred to as a multilayer adsorption since the assumption of Freundlich's equation is based on heterogeneous distribution of active sites [3].

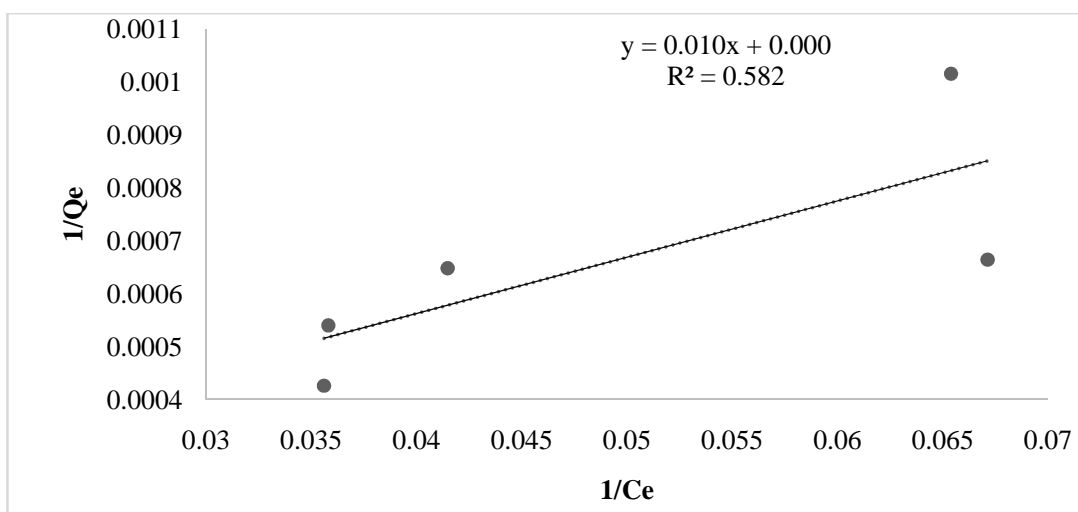


Figure 3. Langmuir Isotherm Model for the Adsorption of the Dye

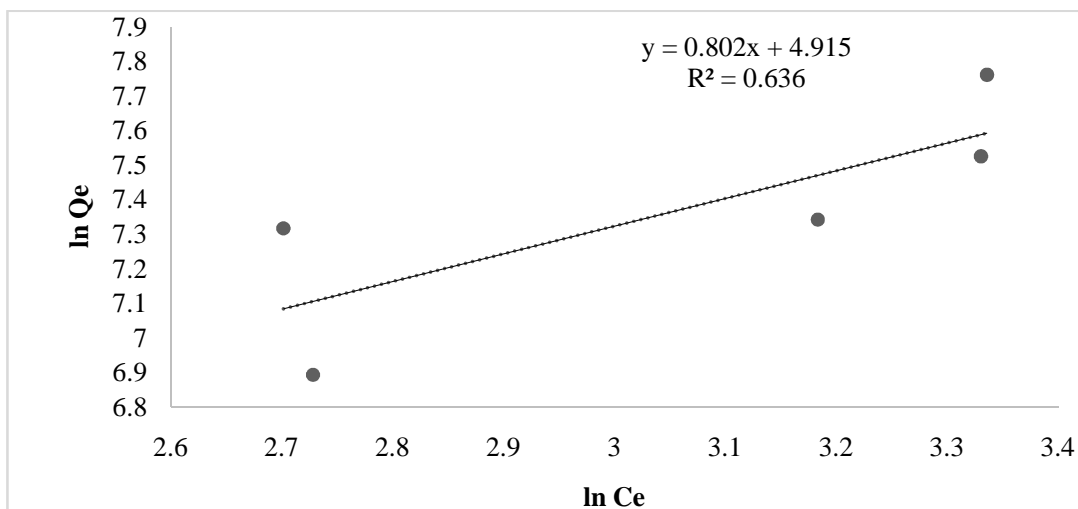


Figure 4. Freundlich Isotherm Model for the Adsorption of the Dye

### 3.7 CONCLUSION

The two variables investigated viz. dye concentration and solution pH have been found to have direct influence on the adsorption process

and an optimum adsorption of 62.30% was recorded at 45mg/L while pH 6 recorded an optimum adsorption of 63.07%.

Freundlich's model is best fit for the adsorption of the dye, consequently, the adsorption is said to be multilayer adsorption process.

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## **Evaluation of the Mechanical Properties of Chemically Modified Cow Hair Fibres Filled Recycled Low-Density Polyethylene Composites**

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### **ABSTRACT**

*Composites were prepared from recycled low-density polyethylene (RLDPE) and cow hair using melt mixing and compression moulding technique. The fibres were treated with 0.2M H<sub>2</sub>O<sub>2</sub> for improved adhesion. Composites of untreated and treated cow hair fibres were prepared with 0 to 50 wt% fibre loading at intervals of 10 wt% and their mechanical properties were evaluated. Results obtained show a remarkable increase in the tensile strength, tensile modulus, flexural strength and flexural modulus of the composites filled with H<sub>2</sub>O<sub>2</sub> treated cow hair fibres. At 30% filler loading of the treated fibres the tensile strength improved from 7.45 MPa to 9.62 MPa. The Tensile Modulus increased from 4.4 MPa to 33.58 MPa at 40 % filler loading. The Flexural strength and flexural modulus markedly improved from 13.29 MPa to 24 MPa and from 67.44 MPa to 292.26 MPa respectively. SEM micrographs of the tensile fractured surfaces revealed enhanced fibre-matrix interfacial adhesion with the modified hair fibres.*

**Keywords:** Recycled low-density Polyethylene, Cow hair fibres, Hydrogen peroxide, Mechanical properties, Morphology

### **INTRODUCTION**

As the concern of an over polluting environment is increasing day-by-day, the need of the present time is to develop such materials which are sustainable and at the same time energy efficient in nature so as to minimize and reduce further damage to an already damaged environment [1]. As a result of this, environment friendly and non-toxic materials are gaining popularity among researchers and industry. Intensive research in adopting natural fibres as reinforcement in polymeric composites with an objective to take advantage of nature's gift to mankind and reduce the huge resources pumped into the development of synthetic fibres, has intensely attracted the interests of contemporary materials' scientists and engineers [2].

Natural fibres have found wide applications and they are abundant in nature [3]. Examples of natural fibres are; cotton, coconut fibre, straws of wheat, hemp, sisal, silk, avian fibre, horse hair, alpaca hair, human hair, cow hair and more. Their availability, renewability, low density, and being inexpensive as well as satisfactory mechanical properties render them an eye-catching ecological substitute for glass, carbon and man-made fibres that have been conventionally used for the manufacturing of composites [4]. They are

subdivided based on their origins; from plants, animals, or minerals. The predominant ones exploited, are those of plant origins, due to their wide availability and renewability in short time with respect to others [5]. However, this present study concentrates its pivotal point on the animal fibre; specifically cow hair.

In most tanneries in Nigeria, animal hair are heaped in large quantity and usually disposed off by burning. The burning process creates environmental pollution. In the same vain, burning method is widely adopted in the removal of hair from the body of cattle slaughtered for meat in Nigeria. In the cause of burning off, the smoke generated also create environmental pollution.

In order to add value to the cow hair, this work employed it as filler for composite making.

A composite consists of two phases i.e. a continuous phase and a discontinuous phase [6]. The continuous phase is known as the matrix while the discontinuous phase is the reinforcement [7]. In this work Recycled Low Density Polyethylene (RLDPE) is used as the continuous phase and cow hair as the discontinuous phase.

In Nigeria, one of the major domestic applications of Low-Density Polyethylene (LDPE) is in its use for packaging and for making water sachets. These plastic wrap and water sachets are not biodegradable and hence create disposal problems. The usual way of eliminating them from the environment is to burn them off. Increase in burning is hampering efforts to keep the environment green.

In order to divert the raw burning of the two waste materials and minimise burning, composite material was made by bringing the waste-LDPE and Cow Hair Fibres (CHF) together.

However, the prominent shortcoming of natural fibres in polymeric composites is the poor compatibility between fibres and hydrophobic matrix interface [8, 9]. The cow hair fibres were treated with 0.2 M H<sub>2</sub>O<sub>2</sub> because of its effective ability in removing lipids, waxes, and oils from the surface of animal fibres. This will roughen the surface of the fibres and improve mechanical anchorage of the fibre and matrix.

## MATERIALS AND METHODS

### MATERIALS

Cow hair of white Fulani species of cattle was collected from Zaria abattoir, Waste LDPE was collected from waste bins of Ahmadu Bello University, Zaria, Nigeria. H<sub>2</sub>O<sub>2</sub> procured from Sigma Aldrich. Two-Roll Mill (U.S.A Model 5189), Hydraulic Hot Press (U.S.A Model 3851-0), Instron 1195 universal materials testing machine were used to characterise the composites.

### METHODS

#### Chemical Treatment of Cow Hair Fibres

The cow hair fibres were thoroughly washed with detergents and rinsed with distilled water and then oven dried at 50 °C. Fibres were immersed in a 2000 cm<sup>3</sup> beaker containing 0.2M H<sub>2</sub>O<sub>2</sub>. The beaker was placed in a shaker water bath maintained at 50 °C for 4 hours to effectively remove lipids from the surface of the fibres. After four hours the fibres were thoroughly rinsed with distilled water and oven dried at 50 °C.

#### Composite Preparation

Melt mixing and hot compression technique was used to prepare the composites. Two sets of composites were prepared with cow hair untreated (CHU) and cow hair treated with H<sub>2</sub>O<sub>2</sub> (CHHO). In each set same filler loading of 10, 20, 30, 40, and 50 wt% were used. A control sample made of

only waste low-density polyethylene was prepared.

## Composite Characterisation

### Tensile Strength Test

Tensile strength test was carried out according to ASTM D3039 standard for testing the tensile strength of composite materials using Instron 1195 universal testing machine. The dimensions of the samples were 100 mm x 20 mm x 3 mm, maintained at a cross head speed of 10 mm/min. Five samples were tested in each case and the average recorded.

### Flexural Strength

Flexural strength was measured under a three-point bending approach using Instron 1195 universal testing machine according to ASTM D790. The dimensions of the samples were 100 mm x 20 mm x 3 mm. The distance between the spans was 40 mm, and the strain rate was 5 mm/min. Five samples were tested in each case and the average recorded.

### Hardness Test

Hardness test was carried out with computer-controlled Vickers hardness tester in accordance with ASTM E384. Average of five samples were taken in each case.

### Scanning Electron Microscopy

A PHENOM ProX SEM with field emission gun and accelerating voltage of 15 KV was used to obtain SEM images for the tensile fractured composite samples. The samples were made conductive by coating with Osmium with the use of vacuum sputter coater and the fractured surfaces were viewed.

## RESULTS AND DISCUSSION

### Tensile Strength

Figure 1 shows that incorporating cow hair fibre (CHF) into Recycled Low-Density Polyethylene (RLDPE) without modifying the surface of the hair will reduce the tensile strength. This decrease in tensile strength is due to the poor adhesion between the untreated filler and the matrix. The untreated hair fibres have lipids and waxes on their surfaces capable of impairing proper adhesion with the matrix [10]. Treatment of the hair fibres with H<sub>2</sub>O<sub>2</sub> steadily increased the tensile strength from 6.39 MPa to 9.57 MPa at 30% fibre loading in contrast to the untreated hair fibres which generally exhibit low tensile strength. The increment however superseded the tensile strength of the RLDPE matrix at filler loading of 30 wt%



and 40 wt% only. The tensile strength of the RLDPE at these points improved by 29.13% and 28.46 % respectively. These improvements are likely due to the effective removal of lipids from the surface of the hair fibres thus creating stronger adhesion between the filler and the matrix via improved anchorage. This result agrees with the findings of Supri *et al.* [11]. The improved interfacial bond had a positive impact on the stress transfer, hence reducing the chance of interfacial de-bonding which will otherwise reduce the strength. Above the 40 wt% filler loading the tensile strength of the composites markedly decreased by 24.43 % of the RLDPE matrix. The major functions of a matrix in composites are to help hold the filler together and transfer stress onto the filler [12]. However, as the amount of filler increases and the amount of matrix decreases inadequate wetting begins to set in. At this point, the extent to which the matrix holds the fillers together decreases thus stress transfer from the matrix to the filler becomes weak [13].

#### **Tensile Modulus**

Tensile modulus of the neat RLDPE improved with the incorporation of both the untreated and treated filler at filler loadings of 10 wt% - 40 wt% beyond which the tensile modulus began to drop. At filler loading of 40 wt% where there is maximum improvement for both the untreated and treated filler, the untreated CHF improved the tensile modulus by 527.7% while the treated CHF improved the tensile modulus by 663.18%. This indicates that the rigidity of the RLDPE will be improved by about five times its value when incorporated with unmodified CHF and about seven times its value when incorporated with CHF treated with 0.2 M H<sub>2</sub>O<sub>2</sub>. Similar result was obtained by George *et al.* [14]. The presence of the fibres reduced ductility and increased the stiffness of the material.

#### **Elongation at break**

Results obtained show that as the rigidity of the composites increased, the elongation at break decreased. This is because of the bond strength between the fibre-matrix interface. The elongation at break of the neat RLDPE decreased significantly when both untreated and treated CHFs were incorporated into the matrix. There was however, a more rapid decrement with the treated hair fibres. The addition of hair fibres decreased the ductility of the matrix. Similar trend has been reported by other researchers [11, 13, 14 & 15]. They found out that the presence of filler decreased the flexibility of low-density polyethylene matrix.

#### **Flexural Strength**

Flexural strength measures the composite's level of resistance to bending forces. The higher this value, the more resistance the composite has for bending forces and vice versa. The flexural strength like the tensile strength is a function of the bonding strength of the fibre-matrix interface.

When CHFs were incorporated into the RLDPE its flexural strength improved for both the untreated and treated filler. The maximum increase by the untreated filler is at 10 wt% of the filler loading by 49.48 % of the control. At the 10 wt% of filler loading, the treated CHFs improved the flexural strength of the control by 67.19%. Beyond 10 wt% fibre loading with the untreated hair, the flexural strength began to steadily decrease and finally dropped below the flexural strength of the control. This decrease in flexural strength is due to weak adhesion as discussed in the case with the tensile strength. The treated fibres improved the flexural strength beyond 10 wt% filler loading with maximum improvement at 30 wt% filler loading by 80.59% of the control. This indicates that the stiffness of the RLDPE will be improved by about its own value when incorporated with 30 wt% of CHFs treated with 0.2 M H<sub>2</sub>O<sub>2</sub>.

#### **Flexural Modulus**

The flexural modulus of the RLDPE maximally increased at 10 wt% by the untreated CHFs from 67.44 MPa to 176.87 MPa corresponding to 162.26% improvement over the RLDPE. At 10 wt% fibre loading where the untreated hair was best with a value of 176.87 MPa the treated fillers had a value of 198.80 MPa which is 194.78% of the control. Unlike with the untreated fibre the flexural modulus steadily improved with increase in filler loading of the surface modified fibre. The maximum improvement being at 30 wt% of the filler loading. Here the flexural modulus was improved to 333.33% of the value of the control. The presence of the fibre decreased the molecular chain mobility of the low-density polyethylene matrix [15].

#### **Hardness**

The hardness of the composite material is seen to increase on a general note for all the fibre loading with both the untreated and treated CHFs. At 10 wt% untreated filler loading the hardness value increased from 7.50 HV to 12.17 Hv which is 62.27% improvement. Incorporating 10 wt% of the H<sub>2</sub>O<sub>2</sub> treated filler improved the hardness by 139.6%. The hardness value improved steadily in all cases with the highest been at 40 wt% of fibres treated with H<sub>2</sub>O<sub>2</sub>. The hardness value here

improved by 285.73%. This marked improvement is due to the corrosiveness of H<sub>2</sub>O<sub>2</sub> which must have removed lipids and waxes from the surface

of the hair fibres roughening the surface for improved adhesion of the fibre-matrix interface.

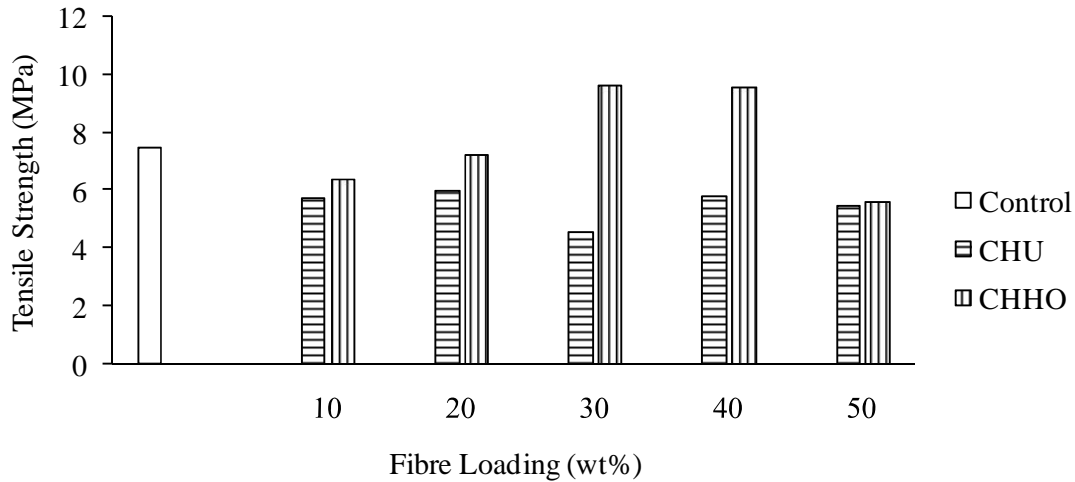


Figure 1: Effect of H<sub>2</sub>O<sub>2</sub> Treatment on the Tensile Strength of CHF/RLDPE Composites at Different Fibre Loadings.

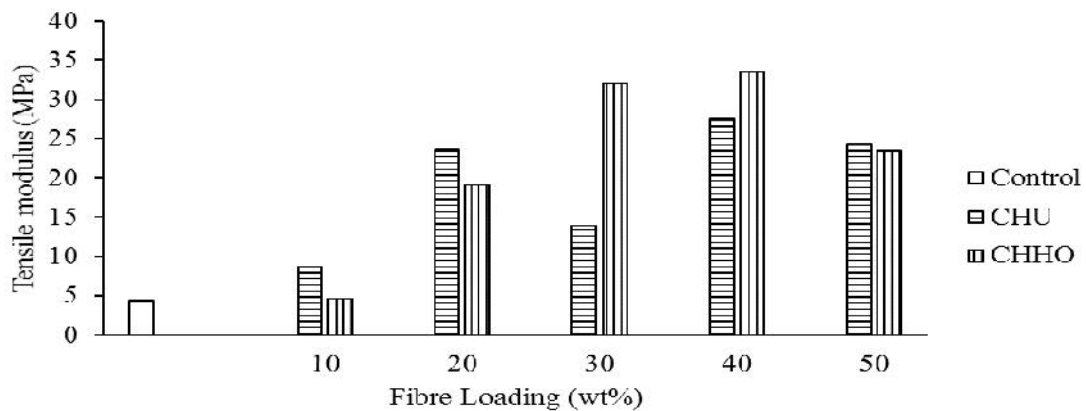


Figure 2: Effect of H<sub>2</sub>O<sub>2</sub> Treatment on the Tensile Modulus of CHF/RLDPE Composites at Different Fibre Loadings.

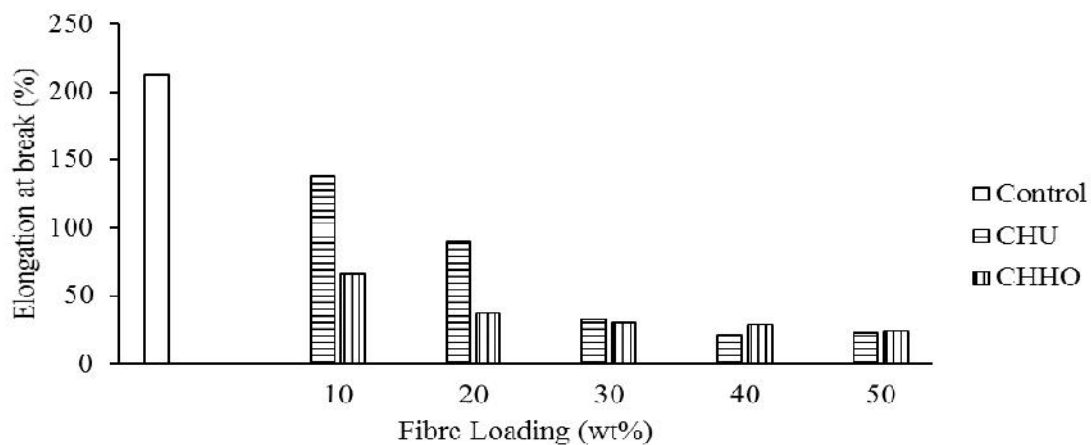


Figure 3: Effect of H<sub>2</sub>O<sub>2</sub> Treatment on the Elongation at Break of CHF/RLDPE Composites at Different Fibre Loadings

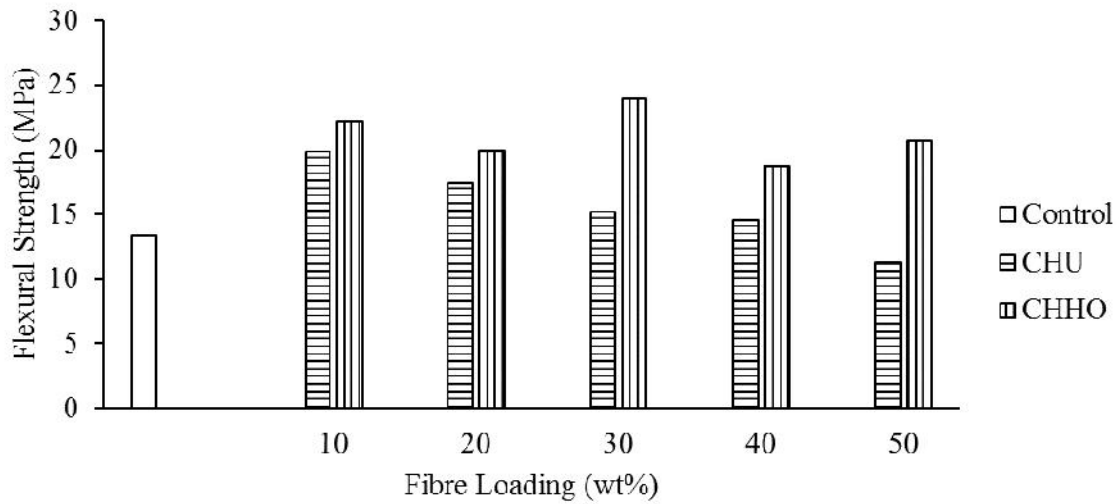


Figure 4: Effect of H<sub>2</sub>O<sub>2</sub> Treatment on the Flexural Strength of CHF/R LDPE Composites at Different Fibre Loadings.

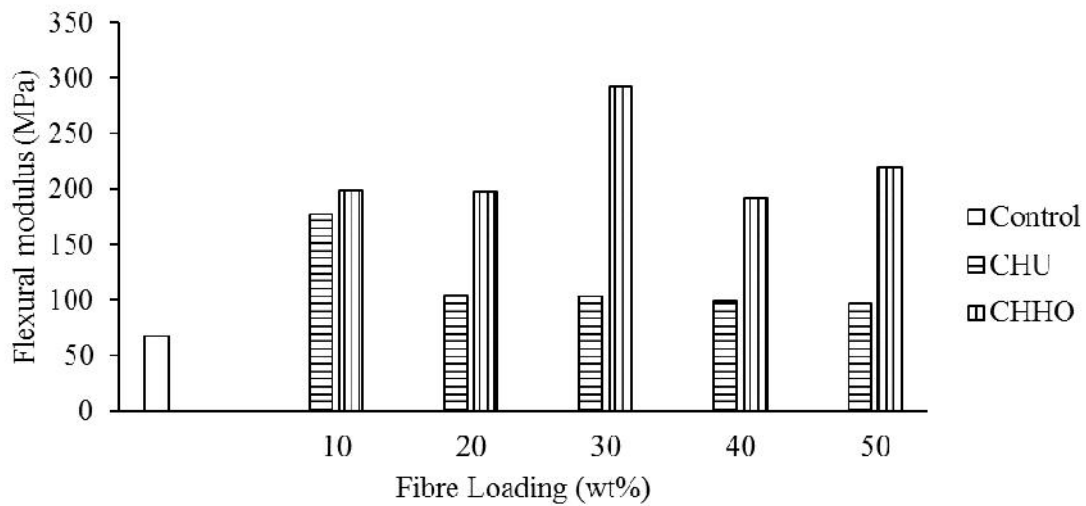


Figure 5: Effect of H<sub>2</sub>O<sub>2</sub> Treatment on the Flexural Modulus Of CHF/RLDPE Composites at Different Fibre Loadings.

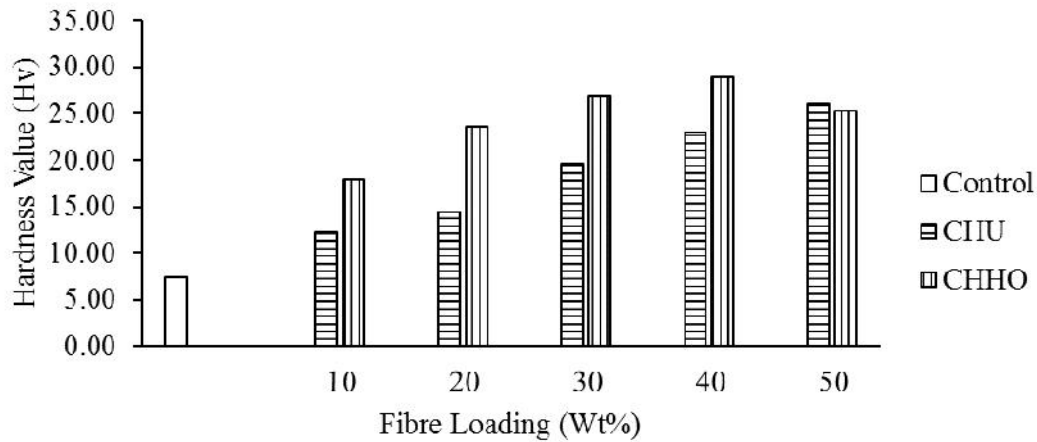
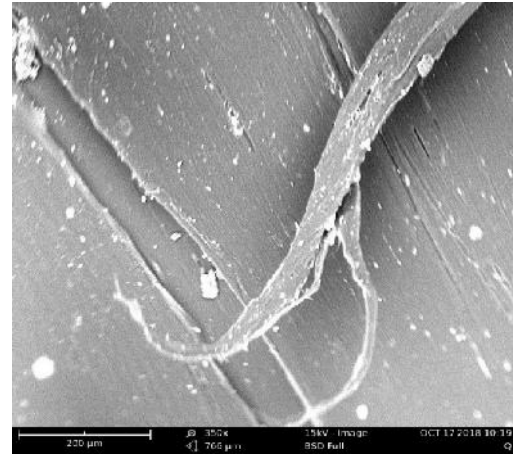


Figure 6: Effect of H<sub>2</sub>O<sub>2</sub> Treatment and Fibre Loading on the Hardness of Cow Hair Filled RLDPE Composites at Different Fibre Loadings.

## Morphological Study



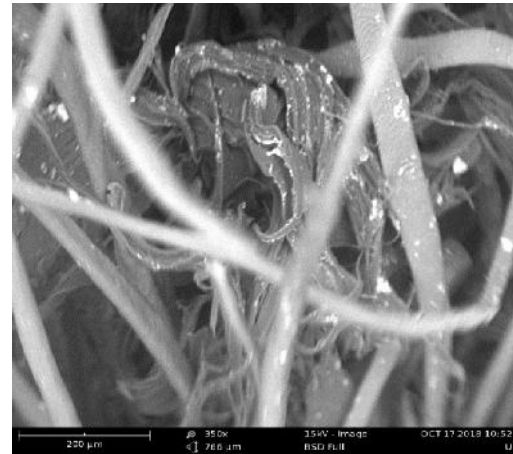
(a)



(b)



(c)



(d)

Plates: SEM micrographs of tensile fractured surface of; (a) untreated CHF/RLDP at 10 wt% filler loading (b) treated CHF/RLDPE at 10 wt% filler loading (c) Untreated CHF/RLDPE at 50 wt% filler loading (d) treated CHF/RLDPE at 50 wt% filler loading

SEM micrographs for all the 10 wt% filled composites show a ductile failure without exposing the fibres. This is expected because of the inherent flexibility of the matrix.

At 50 wt% untreated filler loading there is debonding of the fibre from the matrix and a whole lot of fibre misalignment. This is the reason why the mechanical properties of the composites of untreated fibres at 50 wt% fibre loading was weak.

Chemical treatment of the fibres improved adhesion between the filler and matrix as the degree of fibre misalignment is low compared with the untreated fibres in Plate (c). Fibre alignment factors play a crucial role in the overall properties of composites [15].

Even though chemical treatment removed lipids from the surface of the cow hair fibres and improved adhesion between the fibre and matrix the mechanical properties dropped at 50 wt% fibre loading. This is because of the debonding and fibre pull out as evidenced in Plate (c) and (d). The debonding is attributed to inadequate wetting of the fibres as the filler loading exceeded 40 wt%.

## CONCLUSION

Waste Low Density Polyethylene (WLDPE) can be used as matrix and incorporated with 30 wt% cow hair fibres treated with 0.2 M hydrogen peroxide ( $H_2O_2$ ) to produce useful composites with improved tensile properties. The composites will find applications in areas such as Celine boards, interior wall decorations, and table tops. This will help reduce the environmental pollution caused by Waste low-density polyethylene.

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## Dyeing Performance of Dyes Derived from 2-Amino Heterocycles on Leather

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### ABSTRACT

The dyeing properties of dyes synthesized from 2-amino-4-phenylthiazole (Dye 1) and 2-amino-4-methyl-5-carbathoxythiazole (Dye 2) on leather were studied. The effects of pH, temperature, dye concentration, dyeing time as well as rubbing and light fastness properties of the dyes were evaluated. Results obtained indicate that the dyes used were absorbed optimally at pH 3. It was found that increase in dyeing time increases percent exhaustion such that 60 % exhaustion in 20 min. was increased to 85-90 % in 40-60 min. The exhaustion of Dye 1 was 89.4 % at 50 °C while that of Dye 2 was 92 % at 65 °C. The dyeings showed very good fastness to rubbing and good fastness to light.

**Keywords:** 2-amino-4-phenylthiazole, 2-amino-4-methyl-5-carbathoxythiazole, monoazo, leather

### INTRODUCTION

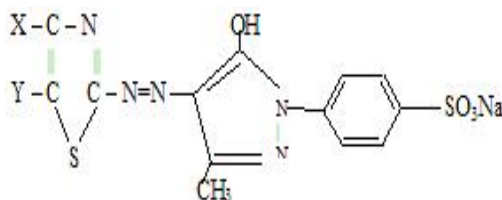
Acid dyes can be applied to nylon from acid dyebath in exactly the same manner in which they are applied to wool and silk fibres. The affinity of acid dyes for nylon is higher than their affinity for wool which accounts for the superior wet fastness and inferior migration properties on the former fibre [1]. Many innovations have been discovered in the past thirty-five years in the field of azo dye chemistry based on heterocyclic systems and studies in the synthesis of such derivatives have been reported. Azo dyes derived from heterocycles are not only important for their excellent properties as dyes for polymeric materials; they are also being used in areas such as reprographic technology, lasers, functional dye applications, photodynamic therapy and nonlinear optical systems. Although a variety of heterocyclic systems have been used for the synthesis of dyes, there remains much for the design and development of new chromophores [2-5].

Recent research has focused on structural variations of existing types for example variation in substituents on the side chains of the coupling components. In our previous work, we synthesized acid dyes derived from 2-amino-4-phenylthiazole and 2-amino-4-methyl-5-carbathoxythiazole and studied their dyeing performance on wool, silk and nylon [6]. To understand fully the dyeing characteristics of these dyes on another substrate, we investigated the dyeing performance of these dyes on leather.

### MATERIALS AND METHODS

#### Materials

Chrome retanned leather was obtained from National Institute for Leather and Science Technology, Zaria. The leather was cut into straps measuring 3x12cm and soaked in water for a period of 12 hours after which the samples were weighed separately for dyeing trials. The structure of the dyes used are shown in Figure 1.



For Dye 1: X = C<sub>6</sub>H<sub>5</sub>, Y = H, for Dye2: X = CH<sub>3</sub>,  
Y = COC<sub>2</sub>H<sub>5</sub>

Figure 1: Structure of Dye 1 and Dye2

### Methods

#### Dyeing trials

A stock solution containing 2% each of Dye 1 and Dye 2 respectively were prepared and used for dyeing trials. Dyeings on leather were carried out at various pH, temperature and time. Dye exhaustion was determined spectroscopically using Unicam spectrophotometer and percentage exhaustion was calculated by measuring the absorbance of the dye bath before and after dyeing using the relation:

$$E ha \quad (\%) = \frac{A_0 - A_1}{A_0} \times 100 \dots \dots (1)$$

where  $A_0$ = absorbance before dyeing and  $A_1$ = absorbance after dyeing

**Effect of pH on dyeing**

This was carried out by measuring a calculated volume of the prepared 2% stock solution based on the weight of wetted leather samples, varying the pH values from 3 to 9 at an interval of 1 pH value. The pH of the dye baths were adjusted using dilute solutions of NaOH and HCl as required, after which the volumes were made up to 50mls. The dye baths with pH values from 3-9 were placed in a steam bath, the leather samples were immersed and then the dyeing carried out for one hour at 55°C.

**Effect of temperature on dyeing**

To determine the effect of temperature, dyeings were carried out within the temperature range of 50-70°C at an interval of 5°C. The pH of the dye baths were adjusted to pH3 being the best pH value obtained where the leather samples were introduced.

**Effect of dye concentration**

Solutions containing 1, 2, 3, 4, 5 and 6% dye were separately prepared. Six leather samples were separately weighed and treated with volumes of dye solution respectively depending on the weight of wetted leather sample. The pH of each solution was adjusted to 3 after which the volume of the dye bath was made up to 50ml. The leather samples were immersed into their respective dye baths placed into a thermostatic water bath at 50°C and dyeing continued for one hour.

**Effect of time on dyeing**

Twelve leather samples were soaked in distilled water for twelve hours, after which they were removed, blotted with tissue paper and separately weighed. A calculated volume of dye stock solution based on the wetted leather sample was taken and pH of the dye bath adjusted to 3. The volume of the dye bath was made up to 50ml, dyeing commenced immediately with continuous agitation for one hour. The dyeing time was varied for 10-120 min. at an interval of 10 min.

**Dry and wet rub fastness**

The dry and wet rub fastness properties of the dyed leather samples were carried out using the rub fastness tester (model THE INST) at 500 revolutions. The leather samples after rubbing were assessed using the grey scale.

**Light fastness measurement**

Eight blue wool standards and specimens (1 cm by 4.5 cm) each were placed side by side on a hinged opaque cover and the central one third of each covered with a black cover. The assembly was exposed to sunlight for 72 hours after which the samples are compared with the original unexposed samples. The change in colour was assessed using the blue wool standards [7-9].

**RESULTS AND DISCUSSION**

**Effect of pH on dye exhaustion**

Table1 shows that the highest recorded percentage absorption of these dyes were 85% and 86% for Dye 1 and Dye 2 respectively at pH3. At pH 4, 84 % absorption of Dye2 was obtained. Conversely, at this pH, Dye1 has the lowest percentage exhaustion (42.5%) The overall performance of the dyes within the pH range used indicate that both dyes are absorbed maximally at pH 3. As the pH value of the dyebath was raised from pH 3 to pH 6, the surface charge of leather varied from being positive to being negative and as a consequence, the electrostatic forces between the negatively charged leather and negatively charged acid dyes changed from attractive forces to repulsive forces. This change in the electrostatic forces reduced the uptake of acid dyes by leather with increasing pH value [10].

**Table1: Effect of pH on dye exhaustion**

pH	Percentage exhaustion	
	Dye 1	Dye 2
3	85	86
4	42.5	84
5	67.5	64
6	52.9	56
7	66.0	51
8	64.0	58
9	45.7	54

**Effect of temperature on dye exhaustion**

The effect of temperature on dyeing performance of both dyes are shown in Table 2. The percentage dye exhaustion of Dye 1 was 89.4% at 50°C while that of Dye2 was 92% at 65°C. The lowest percentage dye exhaustion for Dye 1 and Dye 2 are respectively 82.4% at 70°C 79% at 50°C. Increase in dyeing temperature from 50 to 65 °C increases dye exhaustion for Dye 2 and this may be due to the fact that increase in dyebath temperature increases the kinetic energy of the dye molecules in solution. The rate of dye diffusion into the fibre is thus increased hence the rate of dyeing. As the dyeing temperature is increased, the movement of the macromolecules

increases and the structure of leather opens up more thus the penetration of dye became easier leading to an increased dye sorption. For Dye 1, increase in temperature decreases percent exhaustion. It could be deduced that the saturated dye sorption of leather occurred at 50 °C. Dye molecules penetrated the interior of leather and occupied almost all of the dye sites at that temperature hence no additional interactions between leather and dye molecules even when temperature is increased [10].

**Effect of time on dye exhaustion**

From the results obtained, 94.5% of Dye 1 was the highest absorbed by the substrate after a period of 50minutes while 100% of Dye 2 was absorbed after a period of 120 min. This means that for Dye 1 the extended dyeing time may probably lead to desorption of the dye into the dye bath which could explain the low percentage exhaustion with increase in time. For Dye 2, higher dyeing time generally favoured dye absorption by the substrate. This can be explained by the increase in percentage dye exhaustion with increase in dyeing time. The effect of time on dye exhaustion may be due to the fact that the dye is initially absorbed on the fibre surface and with time it slowly diffused into the innermost portion of the fibre to form ionic linkages with the amine end-groups. During dyeing, the concentration of dyes on the fibre surface increases with dyeing time while that in the dyebath decreases and equilibrium is reached at the fibre-dyebath interface when the concentration of dyes on the fibre does not change with time. Kert et al [11] confirmed the same phenomenon where percent exhaustion increased by prolonged dyeing time until equilibrium is reached and no change of sorption with time. Table 3 shows the effect of time on dye exhaustion.

**Effect of dye concentration on percent exhaustion**

Table 4 shows that the higher the concentration of each of the dyes, the lower the percentage dye exhaustion. This can be understood from the point that there is a maximum amount of dye that can be absorbed by the substrate; hence with higher concentration of the dyes there will naturally be more dyes left in solution. 100% of Dye 2 was absorbed at 1 and 2% dye concentration while 89.95 and 98.8% of Dye 1 was absorbed at dye concentrations of 1% and 6% respectively. Absorption of acid dyes is affected by dye structure, dyeing agents, liquor ratio, dyeing temperature, dyeing time as well as dye concentration [12-15].

**Table 2: Effect of temperature on dye exhaustion**

Temperature (°C)	Percentage exhaustion	
	Dye 1	Dye 2
50	89.4	79
55	86.4	88
60	84.0	85
65	82.4	92
70	82.4	88
75	-	81

**Table 3: Effect of time on dye exhaustion**

Time (min.)	Percentage exhaustion	
	Dye 1	Dye 2
10	68.75	72.5
20	84.09	72.0
30	85.42	89.0
40	72.24	91.1
50	94.58	92.2
60	83.91	92.2
70	86.66	90.8
80	85.37	90.0
90	88.63	93.0
100	90.48	96.25
110	80.95	100
120	81.13	100

**Table 4: Effect of dye concentration on percent exhaustion**

Dye concentration (%)	Percentage exhaustion	
	Dye 1	Dye 2
1	98.8	100
2	82.9	100
3	75	97.7
4	73.7	88.0
5	64.4	83.0
6	89.95	76

**Light fastness test**

Results in Table 5 showed that Dye 2 had better light fastness than Dye 1 at all pH values, temperature, concentration and dyeing time. There are many factors that affect colour fastness. Some of them are the depth of colour, the presence of foreign substances and the inherent photostability of the dye chromophore and the way in which this stability is affected by the chemical nature of the fabrics [7]. The introduction of the phenyl group to position 4 in the dye molecule lowers the photostability of the dye. This may be due to the fact that electron releasing groups lower light fastness because they release electrons to the azo group which is basically the site of photochemical interaction and cleavage.



**Table 5: Light fastness ratings**

Sample number	Light fastness rating	
	Dye 1	Dye 2
1	3/4	4/5
2	3	4/5
3	3	5
4	3	5
5	4/5	5
6	4/5	5

### CONCLUSION

The dyes were applied on leather fabric at 2% shade as acid dyes. These dyes gave very good brightness, levelness and depth on leather. The dyed leather fabrics have moderate to good light fastness. A good levelness and brightness indicate good penetration and excellent affinity of these dyes for leather.

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## **Production and Investigation of the Properties of Non-Disposable Diaper in Comparison with Disposable Diapers**

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### **ABSTRACT**

*This research work produced a reusable diaper and compared its properties, especially absorbency with selected commercial disposable diapers in Nigeria market. The absorbency of two reputable commercial disposable diapers A and B in Nigeria were compared with the reusable diaper C. Absorbency test was carried out in action and also in a saline solution. The result shows that all the diapers have good absorbency. Diaper A has the highest absorbency of see fig.1 which was closely followed by diaper B with 86.00% while diaper C has absorbency of see Fig.1. Though diaper C has see fig.1 percentage absorbance, it is relatively close to those of A and B, thus, compares favourably given that it is not a onetime use diaper. The dimensional stability of the reusable diaper C was found to be excellent with no obvious shrinkage after three washes. Again, from the economic view point, the reusable diaper is cheaper on the long run when the cost of its production was compared to the prevailing market price of all the commercial disposable diapers.*

**Key words:** Production, Investigation, Diaper, Disposable, Reusable, Properties.

### **INTRODUCTION**

Diapers are underwear in which the wearer can urinate or defecate into when the wearer is unable to use the toilet. It has the ability to retain the waste for a little while before discomfort sets in, diaper can be used by infant, children and incontinent adult. Cloth diapers are reusable diaper [1]. Diapers are underwear of polymeric materials been worn by infants, children and incontinent adults in which the wearer can defecate or urinate into it in the absence of toilet; more so, when the wearer cannot control waste disposal mechanism at will [2].

Inability to replace a used diaper regularly may lead to skin diseases in the area in which the diaper covers. Cloth diapers can be found in many different styles and fabrics [3], basically waterproof (plastic material), diaper cover (or shell) and an inner absorbent material [4]. Proponents of cloth diapering cite challenge of environmental friendliness and baby health [5] of disposable diaper as reasons for their usage. Disposable diapers are more harmful because they create enormous amount of trash [6]. They are being produced or treated with chemicals that enhance absorbency and are been disposed after usage [7,8]. Pants made of polymeric material are been used as cover diaper to minimize leakage of waste if over full [9]. The cost of disposable diapers, depend on how well a child or user is potty-trained.

The reusability of cloth diapers also means one can clean them and use afterwards. It can also be transferred to another user in turn, increases savings [10]. For decades, the disposable diaper has been in use. In recent time, these disposable diapers have become very expensive due to recession in the economy and not affordable by low income earners. The numerous disadvantages of disposable diaper viz-a-viz higher environmental cost which arise from significant consumption of natural raw materials, energy usage, health challenge, environmental pollution (majorly air and water) and their disposal after usage contributes a lot of hazard because they are not easily decomposed been made of synthetic material. This necessitated this research.

These issues raise concern and call for the production of alternatives that can take care of the problems generated by the use of disposable diapers. [11] and [12] reported negligent disposal of soiled disposable diapers which is associated with environmental health challenges [13]. The prevalence of disposable diapers in dumpsite attest to this assertion. According to [14] and [15], disposed diapers when buried will eventually mix with underground water and this is dangerous to human health. This research work produces a reusable diaper that may be a good alternative to disposable diaper by taking care of most of the problems identified with disposable diapers. In this research work, a cloth diaper that can compete with the disposable diaper in terms of

absorbency, cost and contain less chemical was produced, the absorbency of the diaper produced examined and a field test carried out in other to assess its comfortability.

## MATERIALS AND METHODS

### Materials

Chip board, Pre-fold, Velour, Polyester fabric, Water proof cover, Elastic, Plaster, Absorbent pad, Diaper, Saline solution (NaCl in water), Water (1ltr), Salt (0.9 gram) and (9 gram), Sewing Machine: butterfly product, (Made in China), Stop watch, Beaker, Cloth pin, Weighing balance.

### Methods

With the aid of a chip board paper, a pattern was drafted using a dimension according to the size wanted and during drafting, there was no seam allowance, after which the pattern was cut out. The pattern was laid on the material to be used, such as the pre-fold, velour, polyester material, water proof cover, suede cloth, after which it was traced out and cut. After cutting the material out, the pre-fold was first joined with the velour placing the absorbent pad at the center and then sewed them together. The suede cloth material was then sewed to cover the surface of the velour. The polyester material (decorative print) was then placed on the surface of the suede cloth with elastic and sewed on both sides (left and right). After which the fabric was turned in order for the decorative print to be at the back of the diaper. Finally, the plaster was added at both ends; at the top (the waist of the diaper) and joined all ends together by sewing to complete the making of the cloth diaper.

### Absorbency Test

Some quantity of saline solution was prepared using Sodium Chloride at 0.9 g per litre solution or 0.9 % solution per litre solution, and stimulated the minerals in baby's urine the weighted dry sample of reusable diaper was immersed (lying flat) into the saline solution in a container and allowed to stay for average of 10 minutes. The diaper was withdrawn from the solution and allowed to drip (placed vertical) for an average of 2 minutes to allow excess solution to drip-off and the final weight was taken. The best diaper for the performance attribute will be the one with the highest absorbent capacity.

The procedure above was repeated for all the samples. This was carried out five times and the averages were recorded.

## RESULTS AND DISCUSSION

### Test for Diaper Performance (Comfortability test)

The non-disposable diaper was tested on 3 babies during the day and over the night, to assess the degree of comfortability. The baby was seen to be comfortable during the day and all through the night. It was also observed that there was no form of rashes on the baby's skin after use all through the night and day.

### Dimensional stability test

Table 1: Dimension of diaper before and after use

MEASUREMENT	LENGTH OF DIAPERS (cm)	BREADTH OF DIAPER (cm)
Before 1 <sup>st</sup> use	28	27
After 1 <sup>st</sup> use	28	27
Before 2 <sup>nd</sup> use	28	27
After 2 <sup>nd</sup> use	28	27
Before 3 <sup>rd</sup> use	28	27
After 3 <sup>rd</sup> use	28	27

**Table 1:** Shows the result of diaper measurement before use for the first, second and third time and there was no change in dimension.

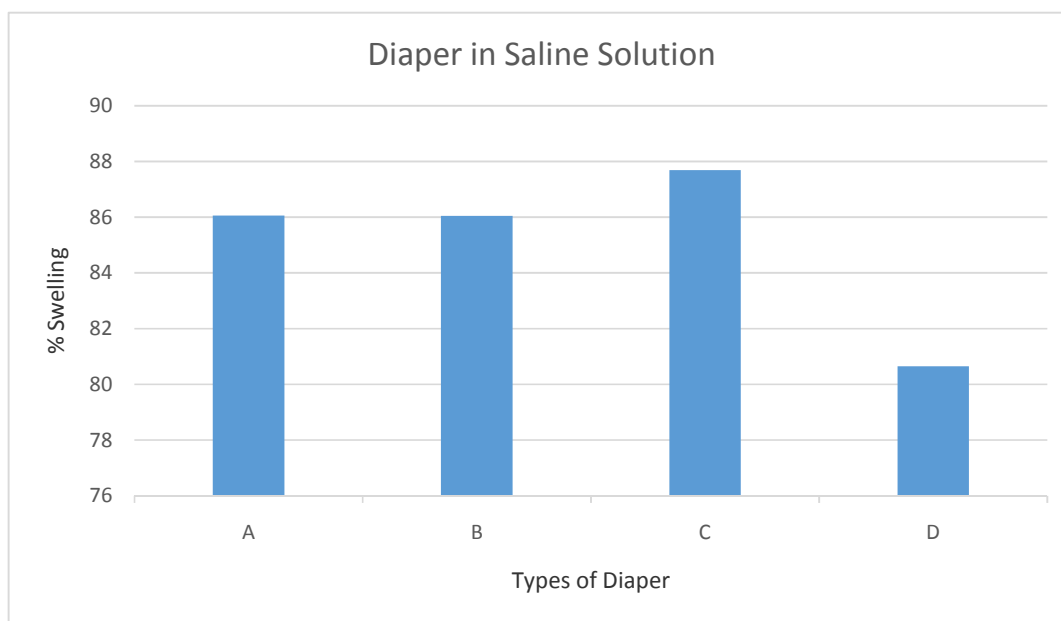
### Cleaning with detergent, antiseptic and bleaching agent

After the first usage of the diaper, it was washed with a detergent, no colour change was observed in the diaper. After the second use, the diaper was washed with water containing antiseptic (brand: Dettol), also no colour change was observed in the diaper after the second washing and the dimensional stability was the same. After the third use, the diaper was washed with water containing bleaching agent (H<sub>2</sub>O<sub>2</sub>), there was a slight change in colour but there was no change in the dimensional stability.

**Table 2** shows the result of the diaper with the highest amount of liquid absorbed. The sample C has a weight difference of 244.5g having absorbed the highest amount of saline solution. The diaper that absorbed least is the sample D with differential weight of 163.33g. This can be compared with the least absorbed disposable diaper sample A with differential weight of 185.53g. Standard procedures were followed by ensuring that the correct measurement of saline solution was used. Also, accurate weight measurements were taken.

**Table 2: Percentage swell of reusable (Sample D: Non-disposable diaper) and disposable diaper (Samples A, B and C: commercial disposable diaper)**

DIAPER TYPES	INITIAL WEIGHT (DRY WEIGHT) (g)	FINAL WEIGHT (WET WEIGHT) (g)	WEIGHT DIFFERENCE (FINAL -INITIAL) (g)	SWELLING (%)
A	30.07	215.6	185.53	86.05
B	34.74	249	214.26	86.04
C	34.30	278.8	244.5	87.69
D	39.17	202.5	163.33	80.65



**Figure 1:** Comparative analysis of the swelling rate of non-disposable diaper (Sample D) with selected commercial disposable diaper (Sample A, B, & C).

**Figure 1** shows that sample A and B has 86.05 and 86.04 percentage swelling respectively, the small difference in their % swelling can be attributed to similarity in their construction. Also, sample C and D has 87.69 and 80.65 percentage swelling respectively. As shown in **Figure 1**, sample D has the lowest swelling rate and this is good for comfortability because the higher the swelling rate the lower the comfortability of the diaper on the user.

### CONCLUSION

At the end of this research, it was discovered that, reusable cloth diaper has similar absorbency to that of the selected commercial disposable diaper. The tests conducted include the absorbency test carried out while in use and also using a saline solution. It was also observed that the disposable diaper (sample C) has more absorbent power than that of the cloth reusable (sample D). Absorbency test also shows that the cloth diaper has similar absorbency to that of disposable diaper (sample A). In order words, the cloth diaper has an

advantage over the disposable diaper which account for their similarity in absorption with some type of disposable diaper, also no chemicals were used in the production of cloth diaper unlike those of the disposable diapers therefore it causes no harm to human health.

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## Dyeing of Polyester with Pristine Multiwalled Carbon Nanotubes

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### ABSTRACT

*Pristine Multiwalled Carbon nanotubes (MWNT) were dispersed in two selected surfactants and their mixture using a probe sonicator at room temperature. The results presented in this article apply only to the short-length MWNT. The dispersions achieved generally ranged from very poor to average. In the average category, the solvents ranked o-Dichloro benzene > Dimethyl formamide > Tetrahydrofuran. The surfactants and one of the mixtures performed better than average with Sodium lauroamphoacetate (Miranol) > Cetyl pyridinium chloride (CPC) > Miranol/CPC mixture (50:50). The higher the aspect ratio, the more difficult they are to disperse; the shorter the nanotubes, the better and more uniform the dispersion obtained. Miranol was then applied (dyed) on Polyester fabrics and various chroma Characteristics obtained.*

**Keywords:** Dispersion; Solvent; Surfactant; Sonication; Mixture; Chroma

### INTRODUCTION

The great deal of interest devoted to the study of CNTs is due to their exceptionally unique collection of properties. Applied quantum chemistry, specifically, orbital hybridization best describes chemical bonding in CNTs. The chemical bonding is composed entirely of  $Sp^2$  bonds, similar to those of graphite. These bonds, which are stronger than  $SP^3$  bonds found in alkanes, diamond and other materials from saturated bonding, provide them with unique mechanical, thermal and electrical properties. These properties confer on them great potentials in general nanotechnology applications especially optics and electronics [1, 2, 3, 4].

CNTs produced by catalytic carbon vapour deposition (CVD) grow in wavy, entangled assemblies which hold them together mechanically [5]. Although they do not possess reactive groups such as  $-OH$ ,  $-NH_2$ ,  $-COOH$  on their surfaces or ends, strong electrostatic forces keep CNTs together: van der Waals forces acting on a high surface area, e.g.  $300 \text{ m}^2/\text{g}$  [6, 7]. In addition, the absence of reactive groups makes them relatively inert in much the same way as graphene. Consequently, in the pristine form, they do not classically dissolve, but form poor dispersions in solvents - a distribution of particles which lack the capacity to form a homogenous mixture composed of only one continuous phase.

The ability to disperse them will therefore depend on their purity, aspect ratio, method of preparation, solvent employed and the type and duration of mechanical agitation.

Achievement of a good dispersion is a prerequisite for improving the performance of any composite, but this has yet to be satisfactorily achieved in CNTs [6, 7, 8]. This challenge is partly responsible for the inability to obtain optimum materials' property enhancement.

The methods of dispersing CNTs can be classified into two main categories; the physical technique, which involves the use of high power direct mixing operations without the modification of the CNT structure. The most common of these are high shear mixing, triple roll milling, ultra-sonication and melt mixing.

Apart from mild ultra-sonication, all these techniques are rather aggressive, causing breakages and rupture of the CNT structure, leading to shorter tubes with lower aspect ratios, and a significantly lower capacity for property-enhancement.

The chemical technique, which involves CNT surface modification, can further be grouped into two: Functionalization and use of surfactants. Functionalization involves the chemical modification of the CNT by introducing reactive

species or groups, such as  $-OH$ ,  $-NH_2$ ,  $-COOH$  onto its structure using strong acids such as sulphuric, hydrochloric and nitric acids under rather harsh conditions. These reactive groups confer solubility and reactivity, but the CNTs would have been robbed of their inherent unique properties.

The second technique does not involve covalent chemical reaction and is therefore, not a permanent surface modification. This non-covalent surface modification employs surfactants and compatibilization polymers. These solvate the tubes, which then disperse more easily [5, 7].

On its part, polyester is relatively inert, and in fabric form, in most cases, disperse dyes are used to dye them, popularly by high temperature (exhaust) dyeing or carrier dyeing. In order not to tamper with the inherent qualities of the CNTs by chemicals normally employed in carrier dyeing, exhaust dyeing was selected as a suitable method by which CNTs could be applied onto the polyester fabric. In this study, an attempt was made to find better dispersants for CNTs and then assessing them by applying on polyester fabric.

## EXPERIMENTAL

### Materials

Long-length MWNTs prepared by catalytic carbon vapour deposition using Fe/Co catalysts (average length – 500 nm) and Short-length MWNTs (( 95% purity) were obtained from US Research Nanomaterials Inc., Houston, Texas (US 4353); (length : 0.5 - 2.0 nm; the chemicals/reagents used were as listed in Tables 2.1. The Ahiba nuance dyer was supplied by Datacolor International, U.S.A., and the probe by Misonix Inc., U.S.A.; The digital spectrophotometer (Spectralite III i7) was obtained from X-rite, U.S.A. The Plain woven 100 % Polyester (Dacron; 171 g/m<sup>2</sup>) was obtained from the Laboratory stores of TECS, College of Textiles, NCSU, USA. Its details are shown in Table 1.1.

### Methods

#### Dispersion of MWNTs

The pristine *short-length* CNTs: 0.050g was placed in a 250mls stoppered flat-bottomed flask containing 100mls of the selected 1% surfactant in de-ionised water. This was then stirred for 30 mins using a probe sonicator operating at 550 watts at a frequency of 20 kHz at room temperature.

## Application of CNTs on the fabric

### High temperature (exhaust) dyeing method

(a) **Baths with normal pH:** The Ahiba Nuance top speed dyeing machine was used to dye the samples using standard high temperature (exhaust) dyeing techniques normally employed for 100% Polyester fabrics. It has 16 detachable, tight-lidded sample containers, uses infra-red (IR) heating with programmable dyeing temperature and rate of heating/cooling. Baths were prepared containing 1 (v/v) % of surfactant and 0.05 (w/v) % CNTs. Using a liquid-to-goods ratio (LR) of 50:1, 1 g of fabric was entered into individual sample containers at room temperature, and the machine set to increase at 20 °C/min up to a maximum of 130 °C. The agitation rate was 15 rpm. Dyeing was carried out at this temperature for 1 hr and then cooled to room temperature at a rate of 3°C/min. The same procedure was used to dye fabric pieces in the surfactant only (henceforth referred to as 'blank' or control)

(b) **Baths with adjusted pH:** The procedure in 3.3.1(a) was repeated, but with the pH of the baths adjusted first to 5, and then to 4 using 10 % acetic acid (to decrease the pH) and 10 % sodium carbonate (to increase it).

(c) **Baths with adjusted pH and electrolyte (sodium chloride, NaCl):** The procedure in 3.3.1(b) was repeated in the presence of NaCl (1 w/v) %

(c) **Cold-padding:** A bath was prepared containing 1 (v/v) % surfactant and 0.05 (w/v) % CNTs at a LR 20:1. with bath pH adjusted in the presence of the electrolyte. Fabric samples (1 g) were impregnated in this bath for 30 mins with occasional stirring, and the excess squeezed out at 20 % expression using a mini pad mangle. The results are shown in Fig. 1.1.

MWCNT application at different percent (%) shades One of the best surfactants used (Miranol) was selected and 1 g pieces of the fabric were dyed at 0.1 %, 0.5 %, 1 %, 2.5 %, 3.5 % and 5 % shades on weight of fabric (owf). The results are as shown in Fig. 2.1.

### Reflectance spectroscopy

The equipment used is a digital spectrophotometer, Spectralite III-i7 model. It has the capacity to measure opacity/transparency, reflectance, transmittance/absorbance, gloss measurements, optical brightness as well as fluorescence of samples. It uses tri-beam

technology which allows the simultaneous measurements with the specular component included (SCI) and specular component excluded (SCE). It can be used to measure these characteristics in textiles, plastics, coated materials, liquids, optically brightened materials, etc. Essentially, it measures chromatic information based on the CIE L\* a\* b\* colour scales. Since the test materials are textured, the equipment was set to take measurements with the specular component included (SCI).

The equipment was calibrated using a 6 mm aperture and the standard white and black discs under standard laboratory conditions. One after the other, the samples were mounted in front of the reflectance aperture and closed. The sample identification data was entered, the type of test and number of replications were selected in the CPU software and the test run.

To eliminate the reflectance of the substrate as a factor, the same function was determined for the 'blank' or control (which was 'blank' dyed) and the value for the control subtracted from that of the dyeing. The results are as indicated in Fig. 2.1 and Table 3.1.

**RESULTS AND DISCUSSION**

In dispersing the CNTs, only surfactant solutions were used so as not to interfere with their chemical constitution.

When CNTs were adequately dispersed, a black ink-like solution was obtained. This appearance suggests it can be used in dyeing, printing, painting or any such-like activities. Since the corresponding substrate is a fabric and making it electrically conductive is the goal, dyeing it with CNTs appears to be one of the best application methods of choice.

The polyester fabric absorbed the CNTs better with decrease in pH (optimally at pH 4) but better yet in the presence of an electrolyte (2 % owf). Exhaust dyeing gave poor, unlevelled results. It is noteworthy that higher temperatures are detrimental to the procedure as simple cold-padding at room temperature (27 °C) gave the best results.

The chromatic information was based on CIE L\*a\*b\* colour scales. They include L\* (lightness) which is on a 0-100 scale, 0 being completely black and 100 completely white; a\* is redness

(positive, 0°) to greenness (negative,180°); b\* is yellowness (positive, 90°) to blueness (negative, 270°) ; C\* (chroma, the colour saturation) from 0 (very dark) to 20 (very light); h° (hue angle, measure of the colour range between 0°-360°; going anti-clockwise on a flat plane from the cartesian co-ordinate abscissa, 0° = red, 90° = yellow, 180° = green, and 270° = blue).

For all the fabric samples, the higher the concentration of CNTs (owf), the lower the values of L\* and C\*, and the darker the shade, and vice-versa (Table 3). It also gave the Kubelka-Munk (K/S) values for determining additive reflectance for coloured objects [9, 10, 11].For all our samples, the K/S values increased with increase in concentration of CNTs (Fig. 3.1).

**Table1.1: Fabric details**

	<b>Warp</b>	<b>Weft</b>
<b>Sett (threads/cm)</b>	40.00	34.00
<b>Yarn linear density (tex)</b>	65.00	70.00
<b>Yarn twist (turns/cm)</b>	11.60	12.50
<b>Crimp (%)</b>	7.75	12.40

**Table 2.1: Surfactants used**

<b>Surfactant</b>	<b>Class of Surfactant</b>
<b>Cetyl Pyridinium Chloride (CPC)</b>	Cationic
<b>Sodium Lauroamphoacetate (Miranol)</b>	Zwitterionic
<b>CPC+Miranol (50:50)</b>	Cationic+ Zwitterionic

**Table 3.1: Chroma Characteristics of the fabric samples**

<b>Shade (%)</b>	<b>C*</b>	<b>L</b>
<b>0.1</b>	2.24	61.38
<b>0.5</b>	2.19	45.21
<b>1.0</b>	1.90	35.56
<b>2.5</b>	1.18	25.43
<b>3.5</b>	0.59	20.95
<b>5.0</b>	0.36	19.99





Fig. 1.1: Cold padding in acidic surfactant + electrolyte.

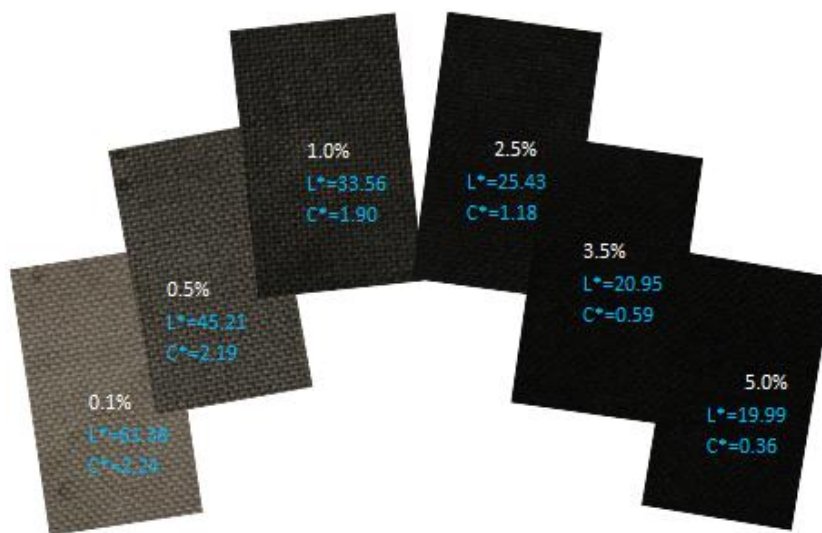


Fig. 2.1: CNT concentrations and their chroma characteristics.

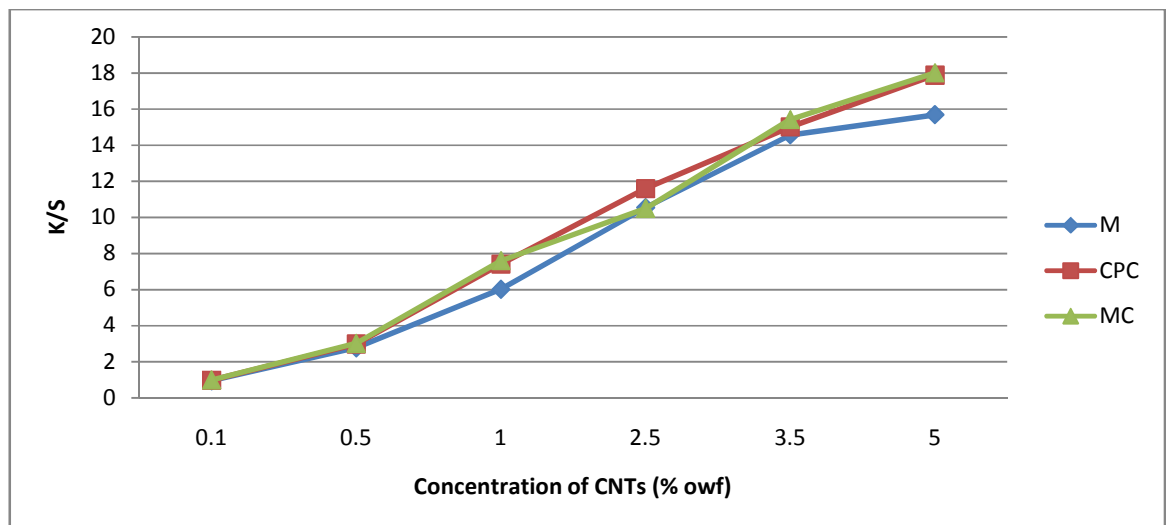


Fig. 3.1: K/S versus concentration for Miranol (M), CPC and their mixtures (MC).

## CONCLUSION

Although CNT dispersions are dye-like solutions, they are not classical dyes and, therefore, should not be applied onto polyester fabrics as such. The 'exhaust' style, a typical, almost traditional method of dyeing polyester, did not yield good results. Miranol, CPC and their mixtures gave good dispersions for short periods of sonication. They were best applied on polyester fabrics by cold padding, especially short, drip-dry cycles in acidic dyebaths in the presence of an electrolyte. After curing, washing with water has very little noticeable effect on the dyed fabric.

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## **Effect of Filler Particle Size on the Mechanical Properties of Waste Polypropylene/ Date Seed Particulate Composites**

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### **ABSTRACT**

*This study was aimed at investigating the effects of filler particle size on mechanical properties of waste polypropylene (wPP) / date seed particles (DSP) composites. Filler particle size of 63, 125, 250, 500 and 750  $\mu\text{m}$  were used at 20 % filler loading. The composites obtained were subjected to mechanical tests (tensile and flexural test, impact test and hardness HV) and the morphological analysis was done using scanning electron microscopy (SEM). The tensile, flexural and impact strength and elongation at break were observed to decrease with increase in filler particle size, while tensile modulus, flexural modulus and hardness (HV) increased with increase in filler particle size. The scanning electron microscopy (SEM) of the tensile fractured surfaces revealed a better particles distribution at 63  $\mu\text{m}$  than 250  $\mu\text{m}$  and 750  $\mu\text{m}$  where agglomerations and interfacial gaps were observed.*

**Keywords:** Composites, waste polypropylene, date seed, filler particle size and mechanical properties

### **INTRODUCTION**

Government regulations and a growing environmental awareness throughout the world have triggered a paradigm shift towards designing materials that are compatible with the environment. The use of biofibres derived from renewable resources as reinforcing fibres in both thermoplastic and thermoset matrices provide positive environmental benefits with respect to ultimate disposability and raw material utilization [1]. Recent legislations associated with environmental impacts of post-consumer plastic wastes have driven substantial attention towards developing viable recycling techniques. The advantages of natural fibres over traditional reinforcing materials such as glass fibre, talc and mica are: acceptable specific strength properties, low cost, low density, high toughness, good thermal properties, reduced tool wear, reduced thermal and respiratory irritation, ease of separation, enhanced energy recovery and biodegradability [2]. Lignocellulosic-plastic composites have been reviewed by Kowell, *et al.*, [3]. The quest for an economically feasible light weight composite that will compete favourably with the conventional materials such as metals in terms of physico-mechanical properties for structural engineering and in building application led to the use of ligno-cellulosic materials as reinforcing fillers in the production of composites[4]. The date (*Phoenix dactylifera L.*) has been an important crop in arid and semiarid

regions of the world. It has always played an important part in the economic and social lives of the people of these regions. The fruit of the date palm is well known as a staple food. Pits of date palm (seeds) are a waste product of many industries after technological transformation of the date fruits or their biological transformation. In some date-processing countries, such as Tunisia, date seeds are discarded or used as fodder for domestic farm animals [5]. In Nigeria date palm is very popular among the people of Northern Nigeria. It is one of the major sources of income to farmers.

Despite the abundance of date seeds, their use as fillers in composites has not been widely reported. Using date seed as bio-filler can help add value to it instead of farm base animal's feed or disposal. Therefore the aim of this research was to produce waste polypropylene / date seed particulate composites (wPP / DSP) from the light fraction of municipal plastic wastes (post-consumer).

### **Materials and Methods**

#### **Materials**

The matrix waste polypropylene (materials used as packaging from dump sites). The filler (date seeds) used in this study were collected from Zaria city market (y'an dabino) Kaduna State, Nigeria.

## Methods

The matrix (waste polypropylene) collected were cleaned to remove impurities such as oil and soil dirt from the containers, air dried and then shred to smaller pieces (flakes form) and kept in the laboratory for future work.

Date seeds were cleaned to remove impurities such as soil dirt, sundried and then ground to particles using jaw crusher and ball mill machines (Retsch Masch. Nr 70992 GmbH & CO. and Kera b.v. Soeter berg Overveld 057748 Holland) respectively. The date seed particles were then sieved using Impact Lab. Seive ISO 3310-1:2000, bs 410-1:2000) to obtain particle sizes of 63, 125, 250, 500 and 750  $\mu\text{m}$ . It was finally dried in an oven at 70°C for 24hours.

The fabrication of the composites was carried out by compounding and compression moulding techniques using two roll mill and compression moulding machines. The filler loading was 20/80 % filler/matrix at all the particle size investigated. A mould of dimension 150×120×5 mm made of steel was used. Control sample was produced using unfilled waste polypropylene. The matrix and filler were mixed for 5 min in a two roll mill for producing homogenous composites. The mixture was placed in a mould in which mould releasing agent was applied. The press cycle consist of three phases, i.e first phase involved the manual pressing to reduce the height, second phase involved in shifting the composite to the compression moulding machine and finally for cooling under pressure to facilitate the setting of thermoplastic resin. The maximum pressing temperature, pressure, time and cold pressing or pressure holding time were 160°C, 5 N/mm<sup>2</sup>, 15min and 5min respectively [6]. After cooling, the resultant composites were removed from the mould for further cooling at room temperature. The composites panels were then trimmed and put into an oven for conditioning at 70°C for 48 hr before testing.

### Tensile Test

The test was conducted according to ASTM D638 standard with a gauge length of 40mm. The samples were cut in dimension of 100 x 10 x 5mm and the test was carried out using YG026D. Multifunctional Electronic Fabric Strength Machine at a cross head speed of 10 mm/min. The test sample was mounted and proper gripping was observed. The tensile parameters were determined and recorded.

### Flexural Test

Three point bending test was performed in accordance with ASTM D790M Test Method I,

Procedure A to measure flexural properties of the composite samples. The samples measured approximately 100 x 20 x 5mm in length, width and thickness respectively. The span length was 80mm apart at 0.5mm / min strain rate

### Impact Test

The impact strength of the composite samples was carried out according to ASTM E23 (Notched) using Norwood charpy impact tester (Model no. 6957, capacity of 15 joules). The specimen size was 100 x 10 x 5 mm. Each sample was placed on the vice and clamped firmly. The pendulum hammer was raised to the required height and then released and strike the sample at once. Then, the impact energy absorbed by the specimen was recorded.

### Hardness Test

Indentation test for composite samples was carried out using Vickers hardness tester (HV) according to ISO 6507. Each sample was loaded on the machine while in the compressed moulds form of 30 x 10 x 5 mm and the indenter (steel ball indenter) brought down to make contact with each sample at three different places. The average of the three readings were calculated and recorded.

### Morphological Studies

The Scanning Electron Microscopy (SEM) of the composites were studied using Scanning Electron Microscope (SEM) Joel JSM 7600F at an accelerating voltage of 5.0Kv and magnification of 500.

## RESULTS AND DISCUSSION

### Tensile Strength

Figure 1 shows the graph of tensile strength of the composites at different filler particle size 63, 125, 250, 500 and 750  $\mu\text{m}$  of DSP. It is shown that increase in filler particle size from 63 to 750  $\mu\text{m}$  led to a decrease in tensile strength. The highest tensile strength was seen with 63  $\mu\text{m}$  at 23.15 MPa and the lowest was seen with 750  $\mu\text{m}$  at 20.26 MPa. This decrease in tensile strength with increase in filler particle size was due to lower surface area provided by the larger particle size led to irregular distribution of the filler particles reducing efficient stress transfer from the polymer matrix to the filler particles. Similar trends were reported by [7,8,9,10].

### Tensile Modulus

The results of tensile modulus are presented in Figure 2. It can be seen that there was an increase in tensile modulus with increase in filler particle

size. The highest tensile modulus was seen with 750  $\mu\text{m}$  at 1.27 GPa and the lowest was seen with 63  $\mu\text{m}$  at 1.12 GPa. This increase in tensile modulus with increase in filler particle size may be due to irregular distribution of filler particles within the matrix structure limiting or reducing polymer molecular chain mobility. This trend was also reported by Genevieve and Isaac [11].

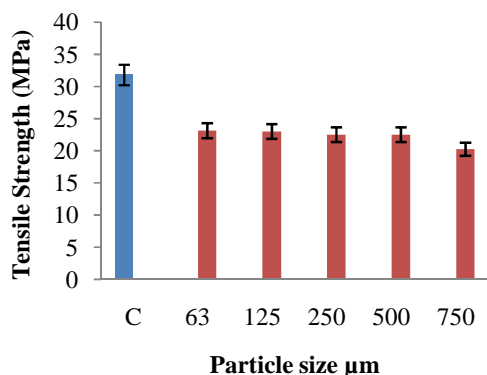


Figure 1: Effect of particle size ( $\mu\text{m}$ ) on Tensile Strength of wPP / DSP composites

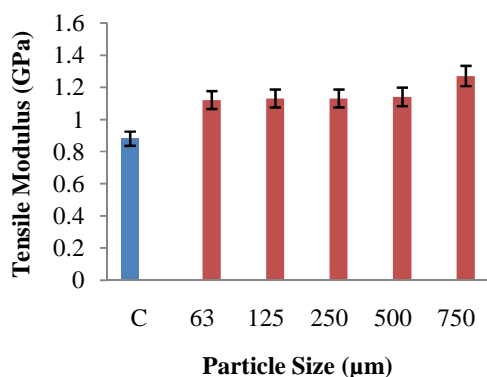


Figure 2: Effect of particle size ( $\mu\text{m}$ ) on Tensile Modulus of wPP / DSP composites

### Elongation at Break

Elongation at break results in Figure 3 can be seen to decrease with increase in filler particle size. Highest elongation at break was seen with 63  $\mu\text{m}$  at 139.62 (%) while lowest was seen with 750  $\mu\text{m}$  at 73.0 (%). This could be attributed to the fact that lower particle size has larger surface area enabling the particles to orient themselves in a form of layer absorbed and share the transmitted stress with the matrix. Also, it could be due to weak matrix / filler interfacial adhesion as a result of lower surface area provided by larger particles compared with smaller particle size. Genevieve *et al.*, [11] reported that the elongation at break of snail shell powder filled polypropylene composites decreased with increases in the filler

particle size. According to Mehdi *et al.*, [12], the particle size of filler greatly affect the elongation at break of the resultant composites by decreasing with increase in particle size from 3 to 48  $\mu\text{m}$ . This may be due to the increased porosity, discontinuity in the microstructure and less formability.

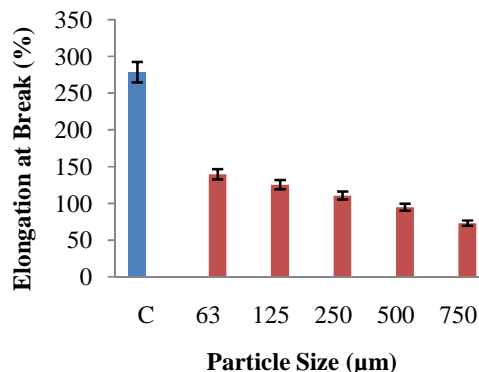


Figure 3: Effect of particle size ( $\mu\text{m}$ ) on Elongation at break of wPP / DSP composites

### Flexural Strength

Flexural strength results in Figure 4 showed a decline with increase in filler particle size. The highest flexural strength was obtained with 63  $\mu\text{m}$  at 100.0 MPa while lowest was seen with 750  $\mu\text{m}$  at 38.0 MPa. This is attributed to the difficulties in achieving homogeneous dispersion associated with larger particles led to inefficient stress transfer from matrix to the filler particles. This difficulty in achieving homogeneous dispersion caused irregular distribution of filler particles within the polymer matrix structure resulted in the formation of agglomeration as can be seen in the SEM images. These agglomerated particles could be stress concentrator points and could affect the final performance of the composites, counterparts were also observed in other studies [13, 14].

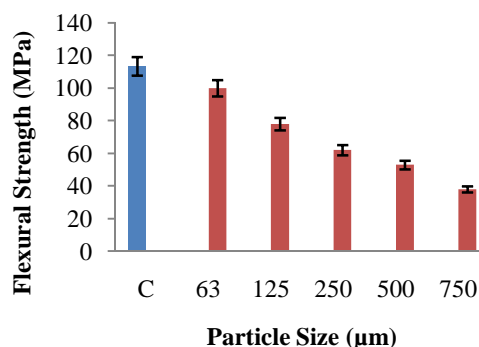


Figure 4: Effect of particle size ( $\mu\text{m}$ ) on Flexural Strength of wPP / DSP composites

### Flexural Modulus

The results of flexural modulus are presented in Figure 5, similar to the tensile modulus results, showed an increase with increase in filler particle size. The highest flexural modulus was seen with 750  $\mu\text{m}$  at 1.52GPa and the lowest is seen with 63  $\mu\text{m}$  at 1.22GPa. The increase in flexural modulus could be attributed to lower contact area provided by larger particle size filler that led to uneven distribution of filler particles which eventually leads to restrain or limiting polymer chain mobility thereby increasing its stiffness. These results also correspond with previously reported work by Zaini *et al.*, [15]. It was reported that flexural modulus increased with increase in particle size for composites made with 230 to 60 mesh oil palm wood flour / polypropylene composites. These results also correspond with previously reported work by Stark and Berger [16].

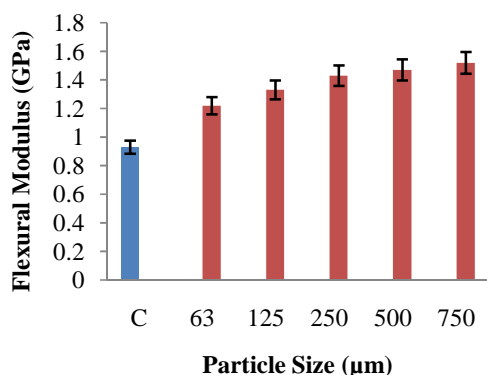


Figure 5: Effect of particle size ( $\mu\text{m}$ ) on Flexural Modulus of wPP / DSP composites

### Impact Strength

The impact strength results shown in Figure 6 indicate a decrease with increase in filler particle size. 63  $\mu\text{m}$  had an impact strength value of 0.7  $\text{J}/\text{m}^2$  while 750  $\mu\text{m}$  had impact strength of 0.42  $\text{J}/\text{m}^2$ . 63  $\mu\text{m}$  with higher impact strength value was as a result of lower particle's ability to orient themselves in a form of layer within the polymer matrix structure due to their high contact and surface area. This could also be attributed to better matrix / filler interaction when lower particle size filler was used. Larger particle size fillers having lower impact strength value indicated their incapability to support stress transfer from matrix to the filler. Gajender and Narula [17] made an observation where impact strength was observed to be dependent on the particle size, and the decrease was higher in the presence of macro filler as compared to micro filler.

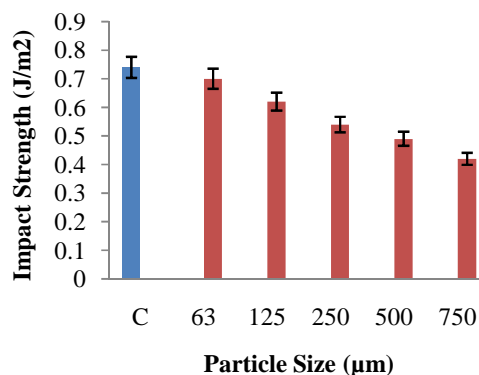


Figure 6: Effect of particle size ( $\mu\text{m}$ ) on Impact strength of wPP / DSP composites

### Hardness

The hardness test result is presented in Figure 7. As shown lower hardness value with 63  $\mu\text{m}$  at 10.8 vickers (HV) Hardness, while higher hardness value was observed with 500  $\mu\text{m}$  at 25.4 vickers (HV) Hardness. This increase could be due to presence of more filler particles at the surface of the composites than in the core, thereby generating greater resistance to indentation and consequently resulting in high hardness values of the composites. Decrease in hardness value was observed at 750  $\mu\text{m}$  with 16.0 vickers (HV) Hardness which may be attributed to void formation during processing. This observation is in agreement with the finding made by [18, 19]. The increase in hardness of the materials with increase in filler particle size may be as a result of irregular distribution that caused the formation of agglomeration seen in SEM micrographs.

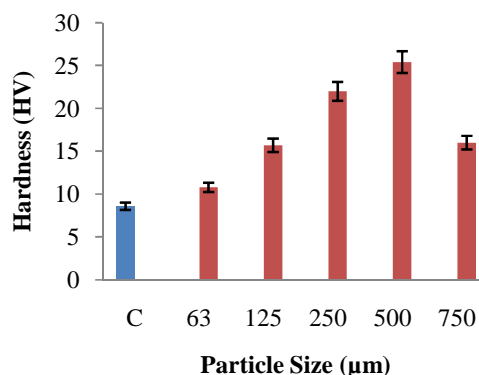


Figure 7: Effect of particle size ( $\mu\text{m}$ ) on Hardness of wPP / DSP composites

### Morphological Studies

The scanning electron microscopy (SEM) results as seen in Figure 8, 9 and 10 shows the fractured surface of the 63  $\mu\text{m}$  (a) shows a better dispersion

of filler particles within the matrix with no clusters seen compared to 250  $\mu\text{m}$ (b) and 750  $\mu\text{m}$ (c). There are agglomerated fillers seen in the 250  $\mu\text{m}$ (b), this is due to inhomogeneity in the compounded materials and can account for some of the lower values in the results of tensile, elongation at break, flexural and impact test results.

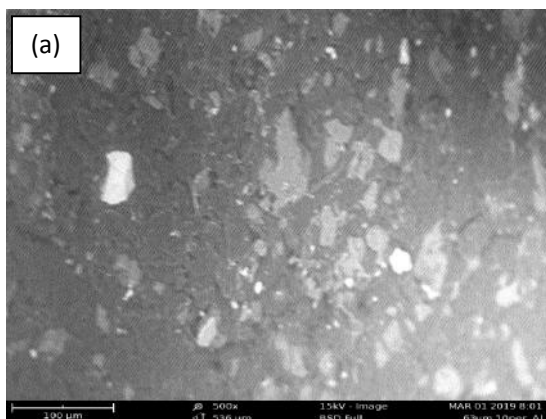


Figure 8: SEM micrograph of fracture surface of tensile strength specimen at 63  $\mu\text{m}$

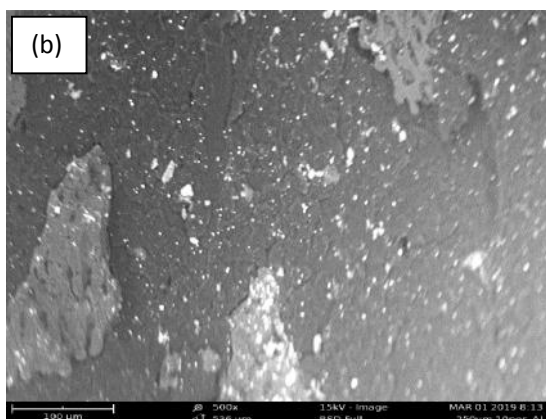


Figure 9: SEM micrograph of fracture surface of tensile strength specimen at 250  $\mu\text{m}$



Figure 10: SEM micrograph of fracture surface of tensile strength specimen at 750  $\mu\text{m}$

Similarly in 750  $\mu\text{m}$ (c) agglomerations were observed, this can be the reason for the low mechanical properties such as tensile, elongation at break, flexural and impact compared to 63  $\mu\text{m}$  (a). These agglomerated filler could be a stress concentrator point and can account for the reduction in tensile strength, elongation at break, flexural and impact strength. Dina *et al.*, [20] reported that there are a lot of fillers agglomerations and dispersions in the 50% recyclates and can account for some of the lower values in the results of tensile, flexural and impact test result. Similar report was made by [21].

## CONCLUSION

It can be concluded that tensile strength, elongation at break, flexural and impact strength on wPP / DSP composites decreased with increase in filler particle size. 63  $\mu\text{m}$  with higher tensile strength, elongation at break, flexural and impact strength at 23.15 (MPa), 139.62 (%), 100.0 (MPa) and 0.7 ( $\text{J} / \text{m}^2$ ) than 750  $\mu\text{m}$  at 20.26 (MPa), 73 (%), 38.0 (MPa), and 0.42 ( $\text{J} / \text{m}^2$ ) respectively. 750  $\mu\text{m}$  had the highest tensile and flexural modulus at 1.27 and 1.52 (GPa) than 63  $\mu\text{m}$  at 1.12 and 1.22 (GPa) respectively. While 500  $\mu\text{m}$  had the highest hardness values at 25.4 than 63  $\mu\text{m}$  at 10.8 vickers (HV) hardness.

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