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## Synthesis and Dyeing Properties of Dichloro-S-Triazinyl Reactive Dyes Bearing a Benzaldehyde Reactive Moiety on Nylon Fabrics

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**Abstract** Eight (8) novel dichloro-s-triazinyl reactive dyes were synthesized by diazotization and coupling reaction of aniline, p-aminobenzaldehyde, and 4-amino-3-nitrobenzaldehyde with cyanurated Gamma and Bronner's acid. The dyes were characterized using UV-Visible and IR spectroscopic techniques, and then applied by exhaustion dyeing on nylon 6.6 fabrics. The percentage dye exhaustion of 71% - 89%, percentage fixation of 57% - 79% and fixation efficiency of 40% - 71% was achieved after application. The dyed fabric samples showed moderate to very good fastness properties to washing, light and perspiration.

**Keywords** Reactive dyes, Gamma acid, Bronner's acid, Nylon 6.6, and Dyeing

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### 1. Introduction

Before the advent of reactive dyes, direct and vat dyes were already in existence and being used for application especially on cellulosic fibres. However, none of these classes of dyes enter into any true form of chemical combination with the cellulose, which usually resulted in the poor fastness properties to washing and other wet processes of direct dyes. Vat dyes on their part, though possess outstanding fastness properties, but are however expensive to produce and troublesome to apply. The desire to overcome these challenges led to the discovery of reactive dyes [1].

Reactive dyes are coloured compounds which contain one or more reactive groups capable of forming covalent bonds between a carbon and phosphorus atom of the dye ion or molecule and oxygen, nitrogen or sulphur atom of the hydroxyl, amino or mercapto group of the substrate. Such covalent bonds are formed with the hydroxyl group of the cellulose fibre, with the amino, hydroxyl or mercapto group of the protein fibre and with the amino group of polyamides [2].

The success of reactive dyes can be attributed to their unique ability to undergo stable covalent bond formation with polymeric substrates, predominantly cellulose, but also polyamides such as nylon [3].

Building on these successes, attention is now being shifted from the use of reactive dyes with only one reactive group to those with more than one reactive group (bifunctional reactive dyes) because of the benefits associated with them.

Bifunctional reactive dyes are coloured compounds that possess more than one reactive moiety per molecule or group, capable of forming covalent bonds between dye ions or molecules and the substrate [4].

Reactive dyes containing cyanuric chloride molecule play an important role in the synthesized dyes. It is a key component in the dyestuff having two reactive groups in their structure, which gives good fixation yields, excellent



wet fastness, brilliant shades and simple application techniques in textile printings. Dye manufacturers have long recognised the need to improve fixation levels, and early attempts to address this issue resulted in the emergence of dyes having more than one reactive moiety per molecule, to statistically improve the chances of dye-fibre bond formation, and since the late 1970s, bifunctional dyes research has largely moved away from simple homobifunctional dyes and towards heterobifunctional molecules, which as the name suggests, contain reactive groups of different types [3]. It can also be easily understood that dyes with two reactive groups give a good fixation yield than dyes with one reactive group for if one of the two dye-fibre bonds is hydrolyzed one is still left for fixation [5-6]. Excellent solubility, higher degree of fixation, good levelling and good to excellent fastness properties is therefore achievable with bifunctional reactive dyes.

In general, acid dyes have attracted much attention to nylon substrates due to their interaction mechanism and easy method for application. However, to achieve satisfactory levels of wash fastness, recourse is required to an after treatment with a commercial syntan and other fixing systems. While an after treatment of the dyed nylon substrates can improve wash fastness, this treatment can impart a change in shade of ground colour but also it is temporary in nature. Consequent upon this, application of reactive dyes to nylon substrates has attracted interests to solve those problems [4]. Reactive dyes react chemically with amino groups within the nylon fibres to form a covalent bond. Theoretically, by virtue of the covalent nature of the dye-fibre bond, reactive dyeing on nylon fibres can display excellent wash fastness without any recourse to an after treatment. In particular, reactive dyes containing heterobifunctional groups can provide great opportunity for efficient dye-fibre reaction due to the sulphatoethylsulphone and monochlorotriazinyl reactive systems [7]. Although this mode of attachment renders the dyeing of nylon straightforward, at least in principle, the wet fastness is usually less than ideal, and there is often a delicate balance to be struck between level dyeing performance and wet fastness. Loss of colour and staining of adjacent fabrics arises from the facile partial dissociation of ion-ion links between dye and nylon. In order to render reactive dyes for nylon attractive to potential customers it is necessary that any such products exhibit an attractive balance of technical and commercial properties. Desirable technical features include high fixation, good build-up and fastness properties [4].

Dichloro-s-triazinyl reactive dyes are reactive dyes that contain two replaceable (reactive) chlorine groups in their structure, which make them more reactive and usually applied at lower temperatures compared to monochlorotriazinyl reactive dyes. With the incorporation of benzaldehyde (reactive) moiety, substantivity of the dyes is expected to increase, and hence, improve the degree of exhaustion, and fixation of the dyes. The objectives of the current investigation were therefore, to synthesize and evaluate tinctorially strong, and bright reactive dyes with two chloro and benzaldehyde reactive moieties per molecule to improve dye fixation levels, it is also hoped that this will allow the dyeing at low temperature instead of at the boil. With increase in the percentage exhaustion of the dye at lower temperature, the amount of dye that will remain unfixated will be reduced, and thereby reducing the environmental pollution caused by the unabsorbed dyes.

## 2. Materials and Methods

Cyanuric chloride, acetone, sodium carbonate, Gamma acid, Bronner's acid, 4-acetamide benzaldehyde, acetic anhydride, hydrochloric acid, ethanol, p-aminobenzaldehyde, nitric acid, and sulphuric acid were purchased from Sigma-Aldrich and of analytical grades and were used directly without further purification. FTIR-84005 Fourier Transform Infrared Spectrometer used in National Research Institute for Chemical Technology (NARICT), Zaria-Nigeria. Jenway UV/Visible spectrophotometer (6405) was used in Multiuser Laboratory, Department of Chemistry, Ahmadu Bello University, Zaria-Nigeria. A&D electronic weighing balance, Gallenkamp thermostatic water bath (made in England) were used in the Department of Polymer and Textile Engineering, Ahmadu Bello University, Zaria-Nigeria.

### 2.1. Synthesis of 4-Amino-3-nitrobenzaldehyde

About 10g (0.06 moles) 4-acetamido benzaldehyde was added to 25 ml of acetic anhydride and heated to 105 °C and vigorously stirred until it completely dissolved. The solution was then cooled rapidly with an ice bath to 30 °C so as



to give fine precipitates. 4 ml nitric acid and 10 ml acetic anhydride were then mixed carefully, and the mixture was then added drop wise to the already stirred suspension of 4-acetamido benzaldehyde at such a rate that the temperature did not rise above 35 °C, after the addition of about  $\frac{1}{3}$  of the acid, colourless crystals of 4-acetamido benzaldehydediacetate separated [8]. Further addition of the acid made the crystals to dissolve, and towards the end of the addition, the temperature was allowed to rise to about 50 °C. The mixture was then stirred with the temperature still maintained at 50 °C for 15 minutes, and then poured into iced water (200 ml). The precipitate of 4-acetamido-3-nitrobenzaldehyde diacetate was filtered off, washed thoroughly with ethanol, and then with water and dried. Recrystallization from ethanol gave the diacetate pale yellow leaflets. About 6 g of the diacetate was then added to 20 ml of concentrated HCl and heated on a water bath for 15 minutes, after cooling, 30 ml of water was added and 4-amino-3-nitrobenzaldehyde was precipitated. The precipitate was filtered off, washed with water, and dried. The intermediate was then purified by recrystallization from water to give orange needles of 4-amino-3-nitrobenzaldehyde.

## 2.2 Preparation of cyanurated Gamma and Bronner's Acids

The cyanuric chloride (1 g, 0.005 mole) was stirred in 25 ml acetone at a temperature below 5 °C for a period of 1 hour. A neutral solution of 2-amino-8-naphthol -6-sulphonic acid (Gamma acid; 1.2 g, 0.005 mole) in aqueous solution of Na<sub>2</sub>CO<sub>3</sub> (10% w/v) was added in small amount over a period of 1 hour. The pH was maintained neutral by simultaneous addition of Na<sub>2</sub>CO<sub>3</sub> solution (1% w/v). The reaction mixture was stirred at 0-5 °C for further 4 hours in order to obtain a clear solution; the resulting product was then used directly. The same procedure was used for cyanuration of Bronner's acid (1.1g, 0.005 mole).

## 2.3 Procedure for Diazotization

### Diazotization of P-aminobenzaldehyde

p-aminobenzaldehyde, (0.6 g, 0.005 mole) was added to 10 ml of 40% aqueous solution of sulphuric acid to form a mixture which was then heated to 80 °C, with agitation for 15 minutes, thereafter, 100 g iced water was incorporated to cool the resulting mixture. The cooled mixture was further incorporated with a drop wise addition of sodium nitrite, (0.35 g, 0.005 mole) over a period of 30 minutes while maintaining the solution at a temperature lower than 5 °C; this was then treated with urea, (2 g, 0.05 mole) to destroy excess nitrous. 200 ml of iced water was then added to obtain aqueous solution of diazonium salt of p-aminobenzaldehyde.

### Diazotization of 4-amino-3-nitrobenzaldehyde

4-amino-3-nitrobenzaldehyde, (1 g, 0.005 mole) was added to a mixture of acetic acid (24 ml), and water (6 ml). The mixture was heated, and then cooled with stirring to about 12 °C. Sodium nitrite (0.35 g, 0.005 mole) was then dissolved in 10 ml water, which was then added in one portion, and 10 ml concentrated HCl also added immediately. The mixture was then maintained at 15-20 °C over a period of 15 minutes; thereafter, a minimum amount of urea was added to destroy excess nitrous acid. The solution was then diluted in an iced water (200 ml) to give a clear solution of the diazonium salt.

### Diazotization of Aniline

10 ml of aniline was dissolved in 25 ml (2.5 equivalent) of HCl and rapidly cooled to a temperature of 0-5 °C in an ice bath, after which, 10 ml (1 equivalent) of sodium nitrite (2.5 g, 0.036 mole) was added over a period of 30 minutes.

## 2.4 General Method of Coupling

A previously prepared solution of p-aminobenzaldehyde (0.6 g, 0.005 mole) in 5 ml water was added drop wise to an ice cooled well stirred solution of cyanurated Gamma acid (1.2 g, 0.005 mole) over a period of 10-15 minutes. The pH was maintained at 7.5-8.5 during the addition. Stirring was carried out for 4 hours, maintaining the temperature at 5 °C. The reaction was then heated to 60 °C, and NaCl (5 g) was added to the mixture and stirred for 1 hour. A solid precipitate was formed, which was filtered, and washed with minimum amount of NaCl (5% w/v).



The solid was dried at a temperature of 80-90 °C and extracted with DMF. The dye (denoted as 1A) was precipitated by diluting the DMF-extract with excess chloroform, it was filtered, washed with chloroform and dried at 60 °C.

Dye 1B was prepared using the same coupling conditions and procedure employed in synthesizing 1A above, by using cyanurated Bronner's acid.

The same coupling conditions and procedure employed in the synthesis of dye 1A was used with 4-amino-3-nitrobenzaldehyde as the diazo component using cyanurated Gamma acid, and Bronner's acid respectively to produce dyes 2A and 2B. Dyes 3A and 3B were obtained using diazotized aniline, and finally, the use of p-aminobenzaldehyde with non-cyanurated coupling components of Gamma acid and Bronner's acid respectively gave dyes 4A and 4B.

## 2.5 Dyeing

The synthesized dyes were applied to nylon 6.6 fabrics by using the procedures reported by Tandama and Bello [9]. The sample (Nylon 6.6) was dyed under acidic conditions.

## 2.6 Percentage Dye Exhaustion (% E), Fixation (% F), and Fixation Efficiency (% T)

The dye % exhaustion of nylon fabrics (%E) is calculated according to the following equation [10].

$$\text{Percentage dye bath exhaustion (\% E)} = \frac{O.D_1 - O.D_2}{O.D_1} \times 100$$

where O.D<sub>1</sub> = optical density before dyeing

O.D<sub>2</sub> = optical density after dyeing

The fixation was obtained thus;

$$\text{Percentage Fixation (\% F)} = \frac{\Delta D - O.D_3}{O.D_1} \times 100$$

where ΔD = difference in optical density before and after dyeing

O.D<sub>3</sub> = optical density after stripping

The overall efficiency value (T) is related to the original amount of dye applied, a simple relationship exist between % T, % F and % E given by:

$$\% T = \frac{\% F \times \% E}{100}$$

## 2.7 Determination of Fastness Properties

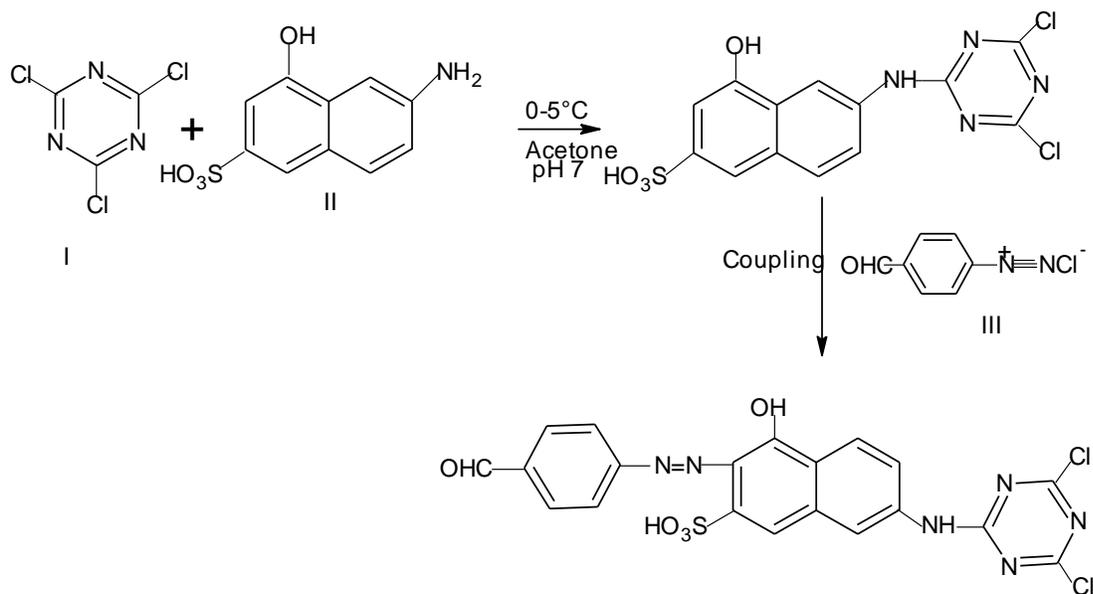
The dyed samples were tested according to the American Association of Textile Chemists and Colourists (AATCC) standard methods [11].

**Table 1:** Conditions used for application of the synthesized dyes

Material	Nylon 6.6
Weight of fabric	1g
Glauber Salt (20% w/v)	4ml
Soda ash (10% w/v)	-
Acetic acid (10%v/v)	1ml
NaCl	-
Liquor Ratio (LR)	50:1
Total Vol. of dye bath	80ml
pH	3.0
Dyeing Temperature	30-40°C
Duration of dyeing	60minutes



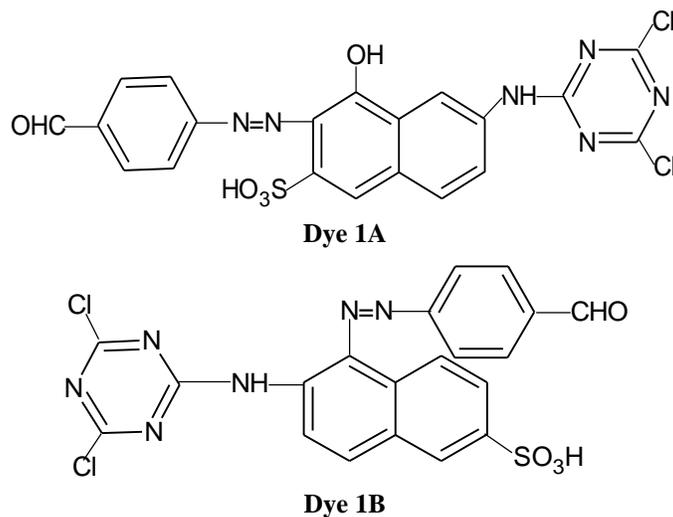
### 3. Results and Discussion

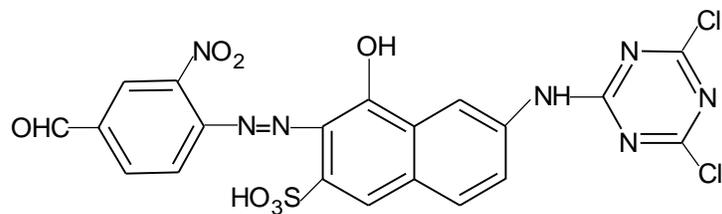


*Scheme 1: Synthesis of dye and intermediate*

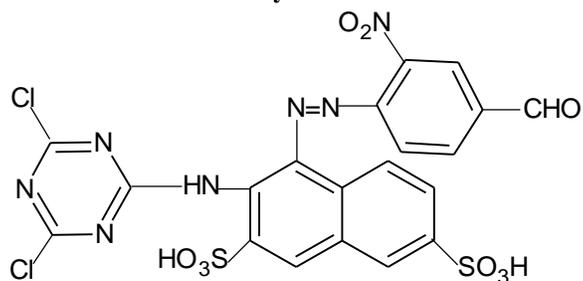
Where; **I** = Cyanuric Chloride, **II** = Gamma acid, and **III** = Diazotized p-aminobenzaldehyde.

The dye obtained from the scheme above is denoted 1A; by replacing Gamma acid (**II**) with Bronner's acid, dye 1B was formed. Dyes 2A and 2B were formed respectively from Gamma and Bronner's acids by replacing the diazo component (**III**) with 4-amino-3-nitrobenzaldehyde; when the diazo component was changed to aniline, dyes 3A and 3B were formed. By using non-cyanurated components (i.e non-cyanurated **II**) with p-aminobenzaldehyde, dyes 4A and 4B were formed.

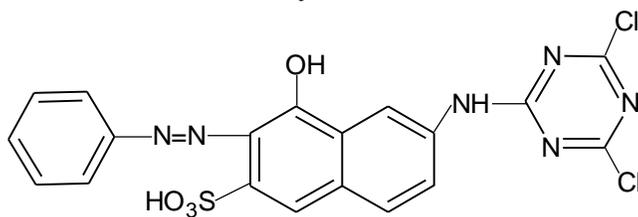




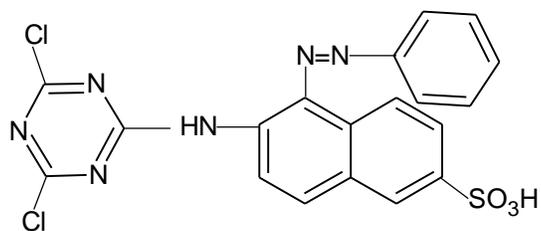
Dye 2A



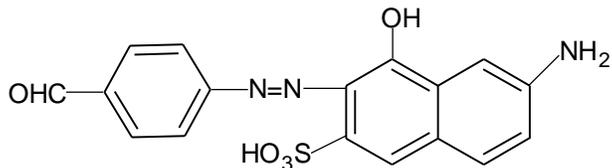
Dye 2B



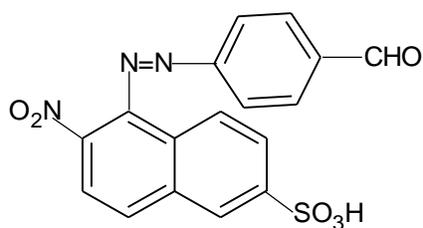
Dye 3A



Dye 3B



Dye 4A



Dye 4B

Figure 1: Structures of dyes 1A – 4B



### Infrared Spectral Results

The IR absorption spectra of the dyes were acquired using FTIR- 8400S Fourier Transform Infrared Spectrophotometer.

**Table 2:** FTIR Results of the synthesized dyes

Dye	OH	CHO	NO <sub>2</sub>	SO <sub>3</sub> H	N=N	C-H	C=C	C-N	C-Cl	N-H
1A	3413.15	1715.74	-	646.17	1396.51	2890.43	1486.20	1172.76	807.24	3405.44
1B	3405.03	1714.77	-	650.03	1396.51	2764.75	1485.24	1193.98	808.20	1609.65
2A	3418.94	1633.76	1492.92	647.14	1492.95	2771.80	1406.15	1188.19	544.91	770.59
2B	3423.76	1414.83	1571.07	661.61	1571.07	-	1414.83	1184.33	-	-
3A	3422.80	-	-	647.14	1398.44	2765.05	1489.10	1168.90	799.52	799.52
3B	3423.76	-	-	657.75	1487.17	2766.01	1400.37	1181.44	542.02	795.66
4C	3426.66	1627.01	-	629.78	1365.65	-	1487.17	-	-	809.17
4D	3424.73	1638.58	-	-	1493.92	2824.84	1638.58	-	-	-

The FTIR results of the synthesized dyes are summarised in Table 2.

The IR spectra in general show characteristic broad band peaks at 3400 – 3505cm<sup>-1</sup> indicating the presence of O-H and N-H stretching vibrations of amino and hydroxyl groups contained in the synthesized dyes [12] as well as C-Cl and C-N stretching vibrations of cyanuric chloride at 542- 800cm<sup>-1</sup> and 1080 – 1360cm<sup>-1</sup> respectively [13].

Dye 1A which was obtained by using diazotized p-aminobenzaldehyde as the diazo component and cyanurated gamma acid as the coupling component, gave the following characteristic peaks; O-H peak of hydroxyl group at 3413.15cm<sup>-1</sup>, CHO group at 1715.74 cm<sup>-1</sup>, SO<sub>3</sub>H peak at 646.17 cm<sup>-1</sup>, 1396.15 cm<sup>-1</sup> due to N=N, C-H stretch at 2890.43 cm<sup>-1</sup>, C=C peak at 1486.20 cm<sup>-1</sup>, C-N had a peak at 1172.76 cm<sup>-1</sup>, C-Cl at 807.24 cm<sup>-1</sup>, and N-H stretch at 3405.44 cm<sup>-1</sup>. When cyanurated Bronner's acid was used as the coupling component, this gave rise to dye 1B with the following peaks; 3405.03 cm<sup>-1</sup> peak due to O-H group, CHO peak at 1714.77 cm<sup>-1</sup>, SO<sub>3</sub>H peak at 650.03 cm<sup>-1</sup>, N=N at 1396.51 cm<sup>-1</sup>, C-H stretch at 2764.75 cm<sup>-1</sup>, C=C at 1485.24 cm<sup>-1</sup>, C-N peak at 1176.62 cm<sup>-1</sup>, C-Cl at 808.20 cm<sup>-1</sup>, and N-H bending mode peak at 1609.65 cm<sup>-1</sup>.

In the second series of dyes (i.e 2A & 2B), 4-amino-3-nitrobenzaldehyde was used as the diazo component. When cyanurated Gamma acid was used as the coupling component, dye 2A was obtained with characteristic peaks as follows; O-H peak at 3418.94 cm<sup>-1</sup>, CHO at 1633.76 cm<sup>-1</sup>, N=O peak at 1492.92 cm<sup>-1</sup>, SO<sub>3</sub>H peak at 647.14 cm<sup>-1</sup>, N=N at 1492.95 cm<sup>-1</sup>, C-H stretch at 2771.80 cm<sup>-1</sup>, C=C at 1406.15 cm<sup>-1</sup>, C-N at 1182.40 cm<sup>-1</sup>, C-Cl peak at 544.91 cm<sup>-1</sup>, and N-H wagging mode at 770.59 cm<sup>-1</sup>. Dye 2B was obtained using cyanurated Bronner acid, the characteristic peak due to O-H was at 3423.76 cm<sup>-1</sup>, CHO at 1414.80 cm<sup>-1</sup>, N=O at 1571.07 cm<sup>-1</sup>, SO at 661.61 cm<sup>-1</sup>, N=N peak at 1571.07 cm<sup>-1</sup>, and C-N at 1188.19 cm<sup>-1</sup>. The N=O stretching vibrations represents a mark difference in the IR spectra of the synthesized dyes in this series.

Dye 3A was obtained using aniline as diazo component alongside cyanurated gamma acid coupling component with O-H stretch at 3422.80 cm<sup>-1</sup>, SO<sub>3</sub>H at 647.14 cm<sup>-1</sup>, N=N at 1398.44 cm<sup>-1</sup>, C-H stretch at 2765.05 cm<sup>-1</sup>, C=C at 1489.10 cm<sup>-1</sup>, 1174.00 cm<sup>-1</sup> peak due to C-N, and C-Cl peak was at 799.52 cm<sup>-1</sup>. Dye 3B was obtained from cyanurated Bronner acid with corresponding peaks as follows; O-H at 3423.76 cm<sup>-1</sup>, SO<sub>3</sub>H at 657.75 cm<sup>-1</sup>, N=N at 1487.17 cm<sup>-1</sup>, C-H at 2766.01 cm<sup>-1</sup>, C=C at 1400.37 cm<sup>-1</sup>, C-N at 1168.90 cm<sup>-1</sup>, C-Cl at 542.02 cm<sup>-1</sup>, and N-H at 795.66 cm<sup>-1</sup>.

The last series of dyes (4A & 4B) were obtained using p-aminobenzaldehyde as diazo component, with non-cyanurated coupling components. Dye 4A was obtained using non-cyanurated Gamma acid producing the following peaks; O-H peak at 3426.66 cm<sup>-1</sup>, CHO peak at 1627.01 cm<sup>-1</sup>, SO<sub>3</sub>H at 629.78 cm<sup>-1</sup>, N=N peak at 1365.65 cm<sup>-1</sup>, C=C



at  $1487.17\text{ cm}^{-1}$ , C-N at  $1174.69\text{ cm}^{-1}$  and N-H at  $809.17\text{ cm}^{-1}$ . While, dye 4B gave absorption peaks at  $3424.73\text{ cm}^{-1}$  due to O-H group, CHO at  $1638.58\text{ cm}^{-1}$ , N=N peak at  $1493.92\text{ cm}^{-1}$ , C-H at  $2824.84\text{ cm}^{-1}$ , and C-N at  $1186.26\text{ cm}^{-1}$ . The dyes in the second series (2A & 2B) all gave absorption peaks due to the nitro group ( $\text{NO}_2$ ) stretch between  $1483.31\text{ cm}^{-1}$  to  $1571.07\text{ cm}^{-1}$ , this is because of the introduction of the nitro ( $\text{NO}_2$ ) withdrawing group in the diazo component. Also, all the dyes in the third series (3A & 3B) gave no peak due to the aldehyde (CHO) group because; aniline was used as the diazo component. Finally, all the dyes gave absorption peaks due to the azo chromophoric group (N=N stretching vibration) in the range of  $1365.65\text{ cm}^{-1}$  to  $1571.07\text{ cm}^{-1}$  (Ovie, 2014).

### Percentage Dye Exhaustion (% E), Fixation (% F), and Fixation Efficiency (% T)

The percentage exhaustion, fixation, and fixation efficiencies are shown in Figures 2 below:

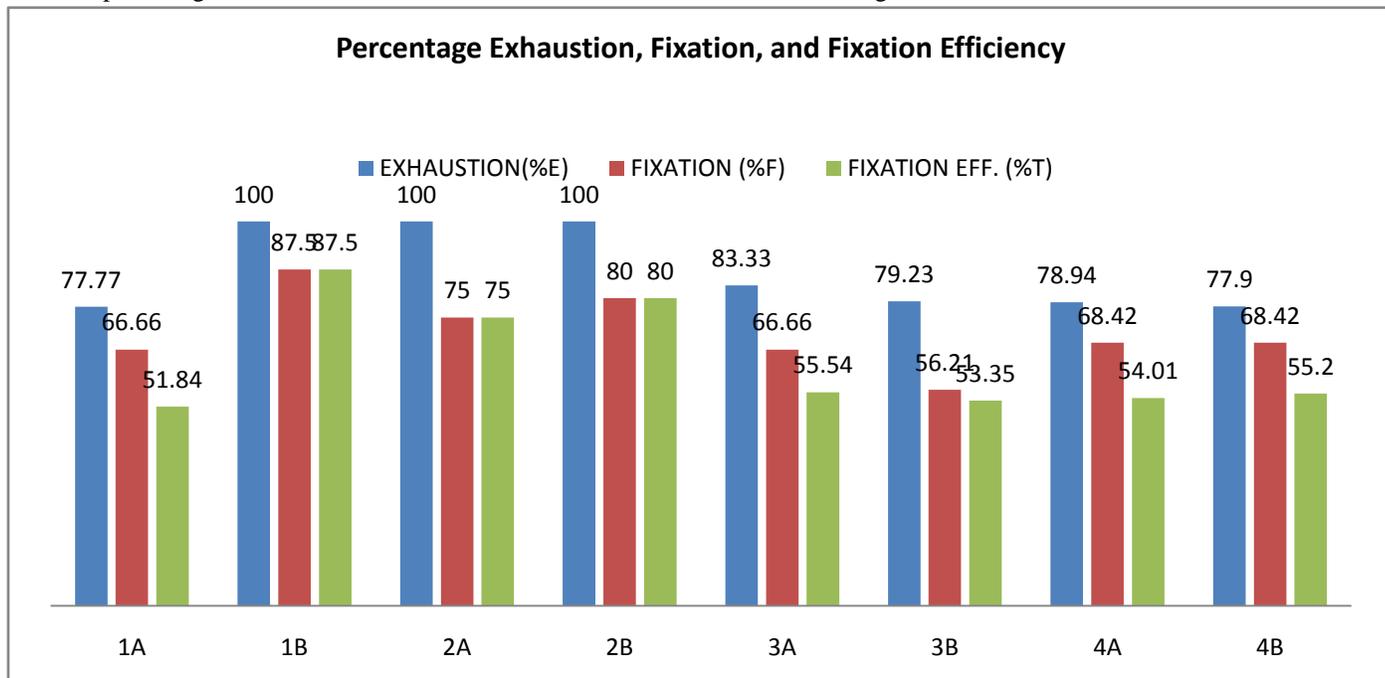


Figure 2: Percentage dye exhaustion, fixation, and fixation efficiency of the synthesized dyes nylon 6.6 fabrics

### Percentage Dye Exhaustion

The percentage dye exhaustion of the synthesized dyes on nylon 6.6 as shown above indicates that all the synthesized dyes possessed very good to excellent exhaustion on nylon 6.6 fabrics. The exhaustion ranges from 77% to 100%. Worthy of note is the fact that 100% dye exhaustion was achieved on nylon 6.6 with dyes 1B, 2A, and 2B. The very good exhaustion of these dyes indicates good affinity and solubility with the fabric [14].

All the dyes in the first, second, and the fourth series contains the benzaldehyde reactive moiety, the results of exhaustion studies of these dyes gave higher percentages when compared with the third series (when aniline was used as the diazo component). Percentage exhaustion of 77-100% was achieved with dyes bearing benzaldehyde reactive moiety; not only that, dyes 4A and 4B were made from non-cyanurated coupling components with percentage exhaustion on nylon 6.6 of up to 78%. This result has confirmed the benzaldehyde reactive moiety as a reactive group, since such an excellent exhaustion was achieved in the absence of cyanuric chloride (the most popular reactive group in reactive dyes).

Dyes 2A and 2B were made using 4-amino-3-nitrobenzaldehyde as the diazo component, this therefore, led to the introduction of an electron withdrawing nitro ( $\text{NO}_2$ ) group; the most powerful of all the common electron withdrawing groups [15]. The percentage exhaustion of dyes containing the nitro withdrawing group was



remarkable. This is because 100% exhaustion was achieved on nylon samples. This shows that, the introduction of an electron withdrawing nitro (NO<sub>2</sub>) has a positive influence on the dye exhaustion.

### Percentage Dye Fixation

The percentage fixation (% F) is the ratio of the amount of dye absorbed which is covalently bounded to the fibre. Figure 2 above shows percentage fixation range of 56% - 87% for the dyed samples. Dyes 1B, 2A, and 2B that had 100% exhaustion were able to achieve 87.5%, 75%, and 80% fixation respectively. The higher fixation values obtained with the new dyes is typical of a reactive dye containing two (2) different reactive groups. This is in agreement with Shore [16] who said the use of reactive dyes containing two reactive groups rather than its analogue with only one reactive group per molecule increases the fixation for a typical 60% to approximately 80% on the average in exhaust dyeing.

### Fixation Efficiency

Fixation Efficiency (% T) is a measure of the original amount of dye applied on a substrate. The fixation efficiency of 51% to 87.5% was obtained on nylon 6.6 samples. The fixation efficiency shows the effectiveness and efficiency of the dye in the dyeing process. It gives a comparison between the exhaustion and fixation of the dye on fabrics (nylon).

**Table 3:** Molar Extinction Coefficient and Fastness properties of the synthesized dyes

Dye	Colour	$\lambda_{\max}/\text{nm}$	log $\epsilon$	Wash fastness		Light fastness	Perspiration fastness			
				CS	SA		Acidic		Alkaline	
						CS	SA	CS	SA	
1A	Brown	688	4.44	4-5	5	3	3-4	3	3-4	3
1B	Yellow	419	4.61	3-4	4	3	3-4	3	3-4	3
2A	Green	658	4.18	5	5	3	4	3-4	4	3-4
2B	Purple	520	4.80	4-5	5	4	4	3	3-4	3
3A	Orange	480	4.07	5	5	4	3-4	3	3-4	3
3B	Yellow	363	4.64	5	5	4	3-4	3	4	3
4A	Pink	395	4.88	5	5	3	4	3-4	4	3-4
4B	Purple	528	4.65	5	5	4	4	3-4	4	3-4

**Key:** CS= Change in Shade SA= Staining of Adjacent fabric

**Wash & Perspiration:** 1= Poor, 2= Fair, 3= Good, 4= Very good, and 5= Excellent.

**Light Fastness:** 1= Poor, 2= Slight, 3= Moderate, 4= Fair, 5= Good, 6= Very good, 7= Outstanding, 8= Excellent.

Wash fastness is a measure of the resistance of dyed material to laundry treatments such as washing. The results of wash fastness rating of all dyed samples as contained in Table 3 was good, with all samples showing only moderate to very slight loss in depth, and in few cases showing no change in shade. The staining of adjacent fabrics for all dyed samples was also very slight. It is a clear indication of high resistance of the materials dyed with the new reactive dyes to laundry treatments. This excellent wash fastness property is attributed to good and appropriate diffusion of the dye molecules into the fabric [17].

Light fastness is a measure of the resistance of colour of dyed materials to fading under the influence of light. The fastness assessment was done using natural day light, and the evaluation of results was based on the eight standard wool fabrics with rating of 1-8. The resistance of dyed nylon 6.6 fabrics using the synthesized dyes as recorded in Table 3 show that, the dyes have light fastness rating ranging from 3 to 4 (i.e, significant fading to fairly fading) when they were exposed to natural day light.

Perspiration test is a measure of the resistance of dyed substrates to body fluids, especially in such areas where these fluid emissions are prominent. In this study, the test was done on dyed nylon 6.6 fabric samples in both acidic and alkaline media, and the results assessed using grey scale for change in shade as well as the degree of staining of adjacent undyed fabric in a similar fashion to wash fastness assessment. The results in Table 3 showed that the dyed



samples had good to very good fastness properties in both the acidic and alkaline media. This result further demonstrated the excellent wet fastness that is typical of reactive dyes. These high wet fastness levels are attributed to stable nature of covalent bond existing between the dye and the polymeric substrates, and it is in agreement with Peters & Freeman [3].

#### 4. Conclusions

Eight (8) dichloro-*s*-triazinyl reactive dyes incorporated with a benzaldehyde reactive moiety have been synthesized; the dyes are grouped into four (4) series, i.e. dyes 1A & 1B, 2A & 2B, 3A & 3B, and 4A & 4B. The first three (3) series (1A-3B) were synthesized using cyanurated coupling components, while 4A & 4B were done using non-cyanurated coupling components. The spectral results of the new dyes showed that the molar extinction coefficients ( $\log \epsilon$  ranging between 4.07 to 4.88). The IR spectra also showed characteristic peaks expected of the functional groups of the various dyes.

In dyeing of nylon 6.6 fabrics with these new reactive dyes, excellent dye exhaustion was recorded. The percentage fixation and fixation efficiency of the dyes were equally very good. Washing and perspiration fastness properties of the dyes on nylon 6.6 fabrics were generally very good; with only slight staining of adjacent undyed fabric. The light fastness results showed fair fading on the dyed fabric samples.

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