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ORIGINAL RESEARCH ARTICLE

Synthesis, Characterization, and Dyeing Performance of Novel Naphthalimide-Derived Bifunctional Reactive Dyes on Cotton and Chrome-Tanned Leather

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ABSTRACT

Various bifunctional reactive dyes were synthesized based on a naphthalimide derivative with a tetrazotised cyanurated coupling component, such as H-acid, R-acid, J-acid, γ-acid, and Tobias acid. The synthesized dyes were purified by recrystallization. The structures of the synthesized dyes were confirmed using UV–vis, FT-IR, MS, and 1H NMR spectroscopic techniques. Spectrophotometric investigations of the dyes in solvents of different polarities were performed to obtain absorption maxima, molar extinction coefficients, and salvochromic effects. These dyes give a percentage yield of 76-88%, a molecular weight of 1262.85-1454.98 g/mol, and a melting point of 300-306 ℃. The dyes produced brown, purple, orange, pink, and yellow colours on cotton fabrics and chrome-tanned leather. The differences in dye colour could be attributed to both the chromophore and the position of the substituent in different coupling components. The dyes showed satisfactory performance, with percentage exhaustion ranging from 70-89% and fixation ranging from 64-93% on the substrate. The fastness properties to washing rating very good to excellent (4-5), to light fastness rating good to very good (5-6), and to perspiration to both the alkaline and acidic rating, very good to excellent (4-5) media of the dyes on chrome tanned leather and cotton fabrics showed good to excellent performance. The study was confined to cotton under laboratory conditions, and further work is needed to test its behavior on other cellulosic materials and to assess its environmental performance. Overall, the findings substantiate the potential for naphthalimide-derived from bifunctional reactive dyestuffs as a versatile class of fiber-selective colorants for cellulose textile applications.

ARTICLE HISTORY

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Synthesis, Nitration, Oxidation, Amination, Purification, Fastness properties

INTRODUCTION

Synthetic organic colorants underpin the aesthetic and functional performance of thousands of products across many industrial sectors, such as textiles, paper, plastics, leather goods, coatings, cosmetics, and food. Ever since the introduction of the first synthetic dyes in the mid-nineteenth century, synthetic dyes have largely replaced natural colorants due to their superior performance, color palette, improved fastness properties, and economies of scale enabled by chemical synthesis. It is also possible to customize the molecular structure, resulting in dyes with targeted affinities for other substrates and performance profiles (Benkhaya et al., 2020; Al-Tohamy et al., 2022).

This synthetic flexibility permits the production of a vast structural variety, which may be tailored to yield almost any color, including the entire visible spectrum. Moreover, numerous azo dyes have high molar extinction coefficients, equivalent to high tinctorial power, and moderate to good fastness to light, washing, and perspiration, indicating that azo reactive dyes can be used in a wide variety of demanding applications (El-Sayed et al., 2024).

Reactive dyes are extensively used in the textile industry not only to colour cotton, but also wool and silk, because of their wide range of colour shades, high wet-fastness properties, and ease of application. New developments in azo dye synthesis have opened up the prospects of naphthalimide derivatives in the textile industry. As reviewed by Dodangeh et al. (2021), Gharanjig et al. (2017) originally synthesized monoazo disperse dyes based on N-ester-1,8-naphthalimide for dyeing polyester-cotton blends, which showed good thermal stability and dyeing fastness. This highlights the relevance of naphthalimide-based azo dyes for improving textile performance.

Ameuru et al. (2018) also demonstrated the industrial value of naphthalimide derivatives and their use in dyeing polyester fabrics at high temperatures without the addition of a dispersing agent, while preserving good fastness

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characteristics. Al-Majidi and Al-Khuzaie (2019) also prepared new azo compounds bearing a 1,8-naphthalimide group by condensing 1,8-naphthalic anhydride with p-phenylenediamine or benzidine. These were also diazotized and condensed with substituted phenols to yield azo products that can serve as acid-base indicators, demonstrating the chemical versatility of these structures. Reported literature on naphthalimide-based monoazo dyes has so far been confined largely to polyester substrates, where the dyes have performed well in terms of visible absorption but merely satisfactory fastness properties. The structural differences and the dyeing conditions resulted in wash and perspiration fastness between grades 4 and 5, and light fastness between grades 3 and 6 (Pendo et al., 2023).

In comparison, there are not many studies on nylon and other polyamides. The limited experiments conducted on naphthalimide-based acid dyes on these substrates exhibit exhaustion values of about 65-70% under acidic exhaustion dyeing conditions, with wash and perspiration resistance typically Grade 4, and light fastness typically Grade 3-4 (Shaki et al., 2015; Hosseinnezhad et al., 2017). Moreover, colorimetric shade differences in nylon 6.6 have not been systematically and comprehensively reported, leaving a gap in knowledge of the behavior of these types of acid dyes on polyamide fibers. This deficiency underscores the need for targeted research on the shade tunability and fiber-dye interactions, as well as the fastness performance of such systems under standard test conditions (Epolito et al., 2005).

Reactive dyes are coloured compounds which contain one or two groups capable of forming a covalent bond between a carbon atom or phosphorous atom of the dye ion or molecule and an oxygen atom, nitrogen or sulphur atom of a hydroxyl, an amino or a mercapto group respectively, of the substrate (Zollinger, 1991) have reported fibre-reactive dyes for silk, wool, and cotton. Mono azo reactive dyes and bis azo reactive dyes have been established as a major group for fixation to cellulose. Bisazo reactive dyes have been widely considered due to their higher fixation yields on various fibres. The purpose of the present investigation is to obtain reactive dyes with a higher degree of reactivity. We report here the synthesis and study of the dyeing properties of bifunctional reactive dyes based on naphthalimide derivatives. Several researchers have reported on the synthesis, characterization of bifunctional reactive dyes on cotton fabric, but there is no existing literature with regard to the synthesis of bifunctional reactive dyes derived from naphthalimide derivatives, which is a new heterocyclic intermediate. The presence of the naphthalimide moieties in the dye structure will improve the fastness properties of the dyes in cotton fabric and bring about deep shade, brilliant hues of high bathochromic effects due to donor donor-acceptor character of the amino and carbonyl groups present in the naphthalimide moiety. This investigation aimed to synthesize, characterize, and apply bifunctional reactive dyes based on naphthalimide derivatives.

MATERIALS AND METHODS

The following are some of the major equipment that were used in the course of the research includes; UV-Vis measurement was performed using a PerkinElmer Lambda 25 spectrophotometer in a 1 cm quartz cuvette with distilled water as the reference, and measurements were taken between 200-800 nm range, FT-IR Spectrophotometer (Agilent Technology Mode Cary 630), Nuclear Magnetic Resonance (NMR) (Agilent Technology Mode 400 MHZ) GC-MS analyses were also carried out using an Agilent Technologies 7590B GC coupled to a 5977A MSD instrument operated in electron ionization (EI, 70 eV) mode;, Most commercial products used in the synthesis of bifuctional reactives dye were purchased from sigma-Aldrich and Weifang Senya chemical company limited, China. All the chemicals and reagents used in the present study, such as: Acenaphthene (purity ≥ 98%), Sodium dichromate (purity ≥ 98%), Hydrazine hydrate (purity ≥ 98%), Stannous chloride (purity ≥ 99%), 8–amino–1–naphthol–3,6–disulfonic acid (H2SO4). 2-naphthol-3-6 disulforic acid (purity ≥ 99%) (R-acid), 2-amino-1-naphthalene sulfuric acid (purity ≥ 99%), 4-amino butanoic acid, 2-amino-1-naphthalene sulfuric acid (purity ≥ 99%), Tridichloro-1,3,5-triazine (purity ≥ 99%).

Synthesis of 5-nitroacenaphthene

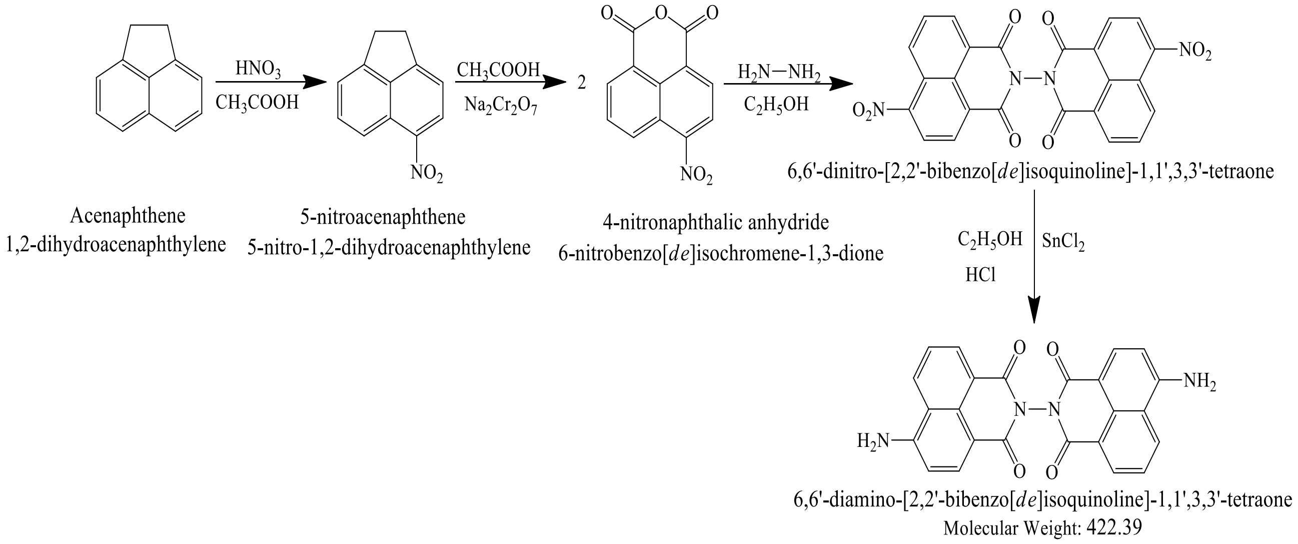
Acenaphthene (15.4 g, 0.1 mol) was dissolved in hot acetic acid (57.2 cm^3), cooled with vigorous stirring to separate the crystalline acenaphthene as finely as possible. Nitric acid (7.03 cm3) was added dropwise for 1 hour at 22-27 °C, then stirred for 1 hour more at 25 °C, and gradually warmed to 68 °C for 30 min then cooled, and the crystals were filtered, washed with acetic acid - water (1: 1) followed by distilled water to give needle like crystals (Ameuru *et al.,* 2018).

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

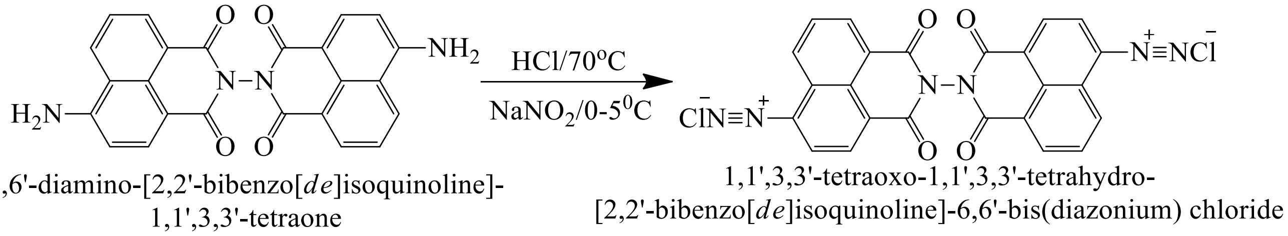
5-Nitroacenaphthene (24.87 g, 0.125 mol) was dissolved in hot acetic acid (248.75 cm^3). Sodium dichromate (Na2Cr2O7· 2H2O) (158.5 g) was added for 3 hours at 65-70 °C. This was gradually warmed to 98-100 °C for 30 min and then refluxed for 5 hours. The content was washed with 0.6 L of hot water, cooled, filtered, and the solid was washed with dilute HCl. It was further boiled for 30 min. with 200 cm^3 of 5% Na_2CO_3 solution for 30 min., filtered, and the filtrate was acidified, and the separated crystals were dried at 120 °C for 4 hours to obtain 4-nitro-1, 8-naphthalic anhydride, which was recrystallized from HNO_3 to afford the off-white needle-like crystals (Ameuru *et al.,* 2020)

Synthesis of 6, 6-dinitro-[2-2'bibenzo[de] isoquinoline]-1, 1'3-3'-tetraone (a)

A suspension of 4-nitro-1'8-naphthalic anhydride (0.02 mol, 4.86 g) and hydrazine hydrate (0.03 mol, 4.71g) was stirred under reflux for 7 hrs in ethanol (102 cm^3). The cooled suspension was filtered and recrystallized from ethanol, yielding a light brown product.



Scheme 1 Synthesis of 6, 6'-diamino-1H, 1'H, 3H, 3'H-[bibenzo[de]isoquinoline]-1, 1'3, 3'-tetraone,



Scheme 2 Synthesis of 1,1.3.3'-tetraoxo-1.1'.3.3'-tetrahydro-[2,2'-bibenzo[de]isoquinoline]-6,6-bis(diazonium)chloride

The intermediate (0.54 g, 0.005 mol) is suspended in distilled water (60 cm³), and hydrochloric acid (0.36 g) is added dropwise to the well-stirred suspension. The mixture is gradually heated to 70 ⁰C until a clear solution is obtained. The form solution is gradually cool to below 5 ⁰C in an ice bath, then already cooled (0-5 °C) $NaNO_2$ (0.6g in 4 cm³ of H_2O) is added over a period of 30 mins with continuous stirring. The stirring is continued for one (1) hour, maintaining the temperature of 0-5 °C with a positive test for nitrous with starch iodide paper. After completing the destruction of the excess of nitrous acid with the required amount of sulphamic acid, the clear tetrazonium solution at 0-5 ⁰C obtained is use for the next coupling as shown in scheme A. (Ameuru et al., 2020)

Cyanuric chloride (1.85g 0.01 mol) is stir in acetone (25cm³) at a temperature below 5 ⁰C for a period of an hour. A neutral solution of the coupling component (3. 19g, 0.01 mol) in aqueous sodium carbonate solution (10% W/v) is then added in small lots for an hour. The pH is maintained neutral by simultaneous addition of sodium carbonate solution (1% W/v). The reaction mass is then stirred at 0-5 °C for a further 4 hours. The cyanurated coupling component solution is use for subsequent coupling reaction in various acids such as H, Gamma, R, J, and Tobias acid respectively (Oforghor, 2021)

The synthesis of dye A_1-D_5 was done by adding a freshly prepared solution of tetrazotized ion 6, 6'-diamino-1H, 1'H, 3H, 3'H-[bibenzo[de]isoquinoline]-1, 1'3, 3'-tetraone. The pH 7.5-8.5 was maintained by simultaneous addition of sodium carbonate solutions (Na_2SO) (10% w/v), yielding a purple solution. The stirring was continued for 4 hrs at a constant temperature of 0-5°C, followed by 1hr. The solid dye precipitates out and is filtered, with a little amount of acetone, and dried at room temperature (Oforghor, 2021)

Purification of the synthesized Dyes

The synthesized dyes were purified by recrystallization from ethanol by adopting the method used by Alaa and Tarek (2006).

Determination of Percentage Yield of the Synthesized Dyes

The percentage yield of the synthesized dyes was determined using equation 1.1 (Gregory, 2005)

$$\%\text{Yield} = \frac{MP}{MMP} \div \frac{MR}{MMR} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 1.1$$

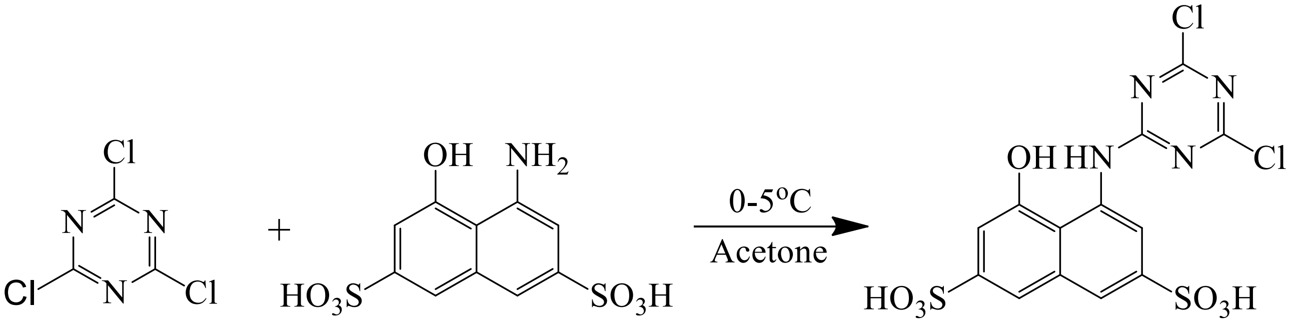
where;

MP is the mass of the product

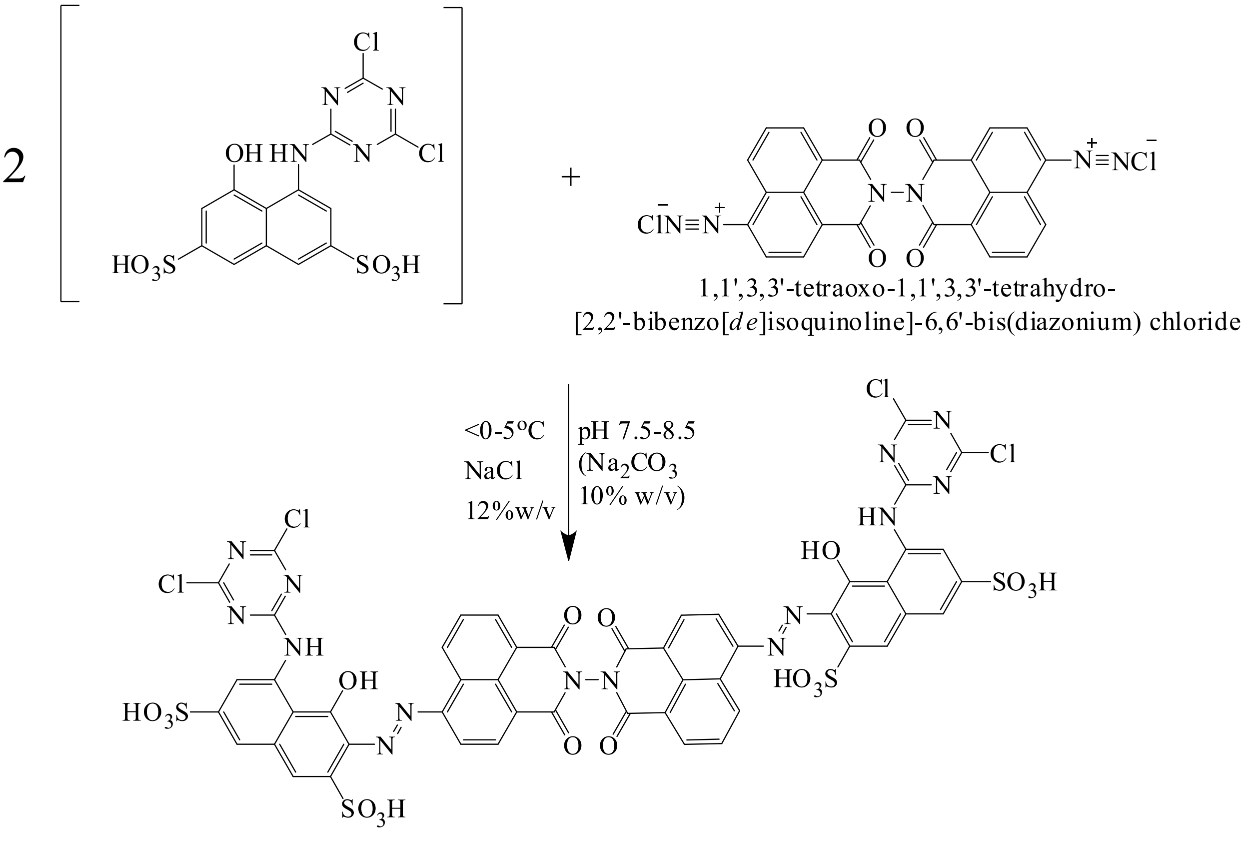
MMP is the molar mass of the product

MR is the mass of the reactant

MMR is the molar mass of the reactant

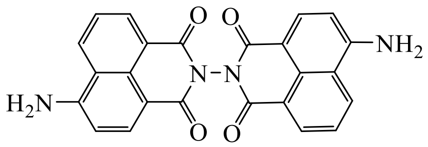


Scheme 3 Procedure for cyanuration of coupling components.



Scheme 4 Synthesis of 3, 3'-((1E, 1'E)-(1, 1', 3, 3'-tetraoxo-1, 1', 3, 3'-tetrahydro-[2, 2'-bibenzo[de]isoquinoline]-6, 6'-diyl)bis(diazene-2, 1-diyl))bis(7-((4, 6-dichloro-1, 3, 5-triazin-2-yl)amino)-4-hydroxynaphthalene-2-sulfonic acid)

Table 1.1: Structures of Synthesized intermediate, molecular formula their IUPAC Name

INTERMEDIATE No.	NAME	STRUCTURE	MOLECULAR FORMULA
A	6, 6'-diamino-1H, 1'H, 3H, 3'H-[2, 2'-bibenzo[de]isoquinoline]-1, 1', 3, 3'-tetraone		$C_{24}H_{14}N_4O_4$

Determination Melting Point of the Dyes

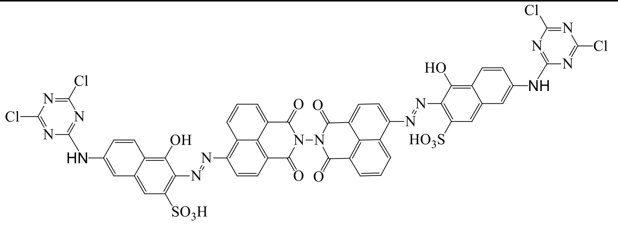
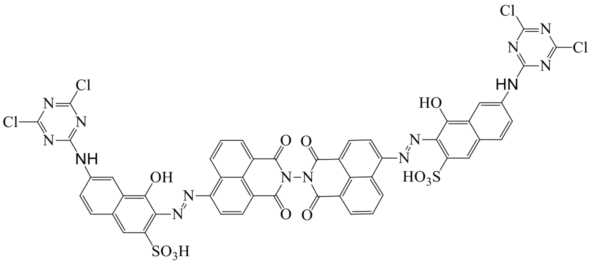
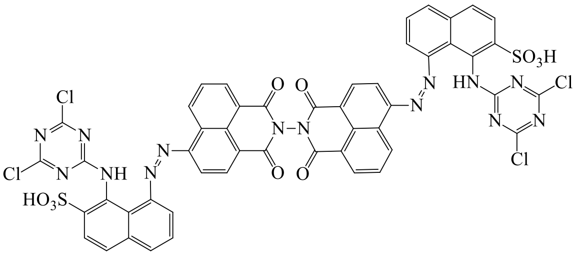
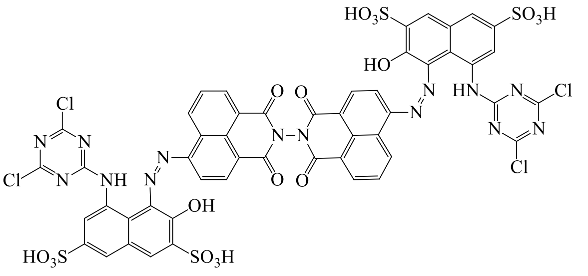
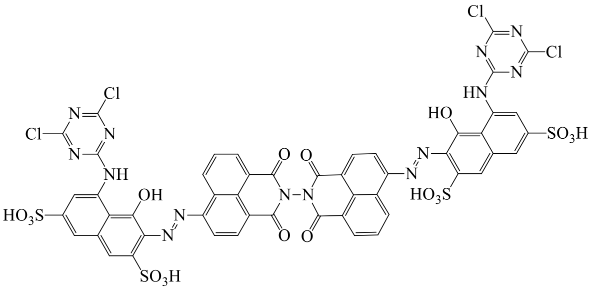
Melting points of the synthesized compounds were determined on a Gallenkamp melting point apparatus (model CD10127). A small amount of each dye was placed in a capillary tube and inserted into the apparatus. The melting point of each dye was obtained by consistently focusing on the apparatus as it gradually heated the dye in the tube.

Determination of Molar Extinction Coefficient

The molar extinction coefficient (Ɛ), which is a constant for each molecule at any given wavelength, represents the absorbance per liter. This was calculated using the relationship,

Log_{10} (I_o/I) absorbance A or optical density

Table 1.2: Structures of Synthesized reactive dyes, molecular formula, and their IUPAC Name

DYE No.		STRUCTURE	MOLECULAR FORMULA
A_1	3, 3'-((1E, 1'E)-(1, 1', 3, 3'-tetraoxo-1, 1', 3, 3'-tetrahydro-[2, 2'-bibenzo[de]isoquinoline]-6, 6'-diyl)bis(diazene-2, 1-diyl))bis(7-((4, 6-dichloro-1, 3, 5-triazin-2-yl)amino)-4-hydroxynaphthalene-2-sulfonic acid)		$C_{50}H_{24}Cl_4N_{14}O_{12}S_2$
A_2	3, 3'-((1E, 1'E)-(1, 1', 3, 3'-tetraoxo-1, 1', 3, 3'-tetrahydro-[2, 2'-bibenzo[de]isoquinoline]-6, 6'-diyl)bis(diazene-2, 1-diyl))bis(6-((4, 6-dichloro-1, 3, 5-triazin-2-yl)amino)-4-hydroxynaphthalene-2-sulfonic acid)		$C_{50}H_{24}Cl_4N_{14}O_{12}S_2$
A_3	8, 8'-((1Z, 1'Z)-(1, 1', 3, 3'-tetraoxo-1, 1', 3, 3'-tetrahydro-[2, 2'-bibenzo[de]isoquinoline]-6, 6'-diyl)bis(diazene-2, 1-diyl))bis(1-((4, 6-dichloro-1, 3, 5-triazin-2-yl)amino)naphthalene-2-sulfonic acid)		$C_{50}H_{24}Cl_4N_{14}O_{10}S_2$
A_4	4, 4'-((1Z, 1'Z)-(1, 1', 3, 3'-tetraoxo-1, 1', 3, 3'-tetrahydro-[2, 2'-bibenzo[de]isoquinoline]-6, 6'-diyl)bis(diazene-2, 1-diyl))bis(5-((4, 6-dichloro-1, 3, 5-triazin-2-yl)amino)-3-hydroxynaphthalene-2, 7-disulfonic acid)		$C_{50}H_{24}Cl_4N_{14}O_{18}S_4$
A_5	3, 3'-((1E, 1'E)-(1, 1', 3, 3'-tetraoxo-1, 1', 3, 3'-tetrahydro-[2, 2'-bibenzo[de]isoquinoline]-6, 6'-diyl)bis(diazene-2, 1-diyl))bis(5-((4, 6-dichloro-1, 3, 5-triazin-2-yl)amino)-4-hydroxynaphthalene-2, 7-disulfonic acid)		$C_{50}H_{24}Cl_4N_{14}O_{18}S_4$

$$A = \varepsilon CL \ldots\ldots\ldots\ldots 1.2$$

Ɛ = Molar Extinction Coefficient

A = Absorbance at λ_{max}

C = Concentration of dye in Mol/dm^3

L = Path length in cm

Infra-Red Spectra of the Dyes

FTIR spectra were measured on a Shimadzu FTIR-84005 spectrometer, in a wavelength range of 4000-400 cm -1 using KBr pellets; a polystyrene film standard was used to calibrate the instrument.

Table 1.3: Physical properties of the synthesized dye intermediates.

Name of Intermediate	Colour of Crystals	Melting point (°C)	Percentage yield (%)	Molecular weight (g/mol)	Molecular formula
A	Dark brown	278-280	96	422. 0	$C_{24}H_{14}N_4O_4$

Table 1.4: Physical properties of the synthesized bifunctional reactive dyes derive from naphthalimide derivatives.

Dyes No	Molecular formulae	Theoretical molecular weight (g/mol)	Melting point (℃)	Percentage yield (%)	Colour of the crystals
A_1	$C_{65}H_{28}CL_4N_{14}O_{18}5_4$	1454. 98	304-306	82	Blue
A_2	$C_{65}H_{28}CL_4N_{14}O_{18}S_4$	1454. 98	300-302	74	Red
A_3	$C_{56}H_{28}CL_4N_{14}O_{18}5_2$	1294. 85	300-302	88	Brown
A_4	$C_{56}H_{28}CL_4N_{14}O_{12}5_2$	1294. 85	308-310	80	Black
A_5	$C_{56}H_{28}CL_4N_{14}O_{10}5_2$	1262. 85	300 302	76	Orange

Table 1.5: Wavelength of Maximum Absorption and Molar Extinction Coefficient of the Dyes

Dye No	ε_{max} in DMF × 10^4 (L/mol^{-1} cm^{-1})	Distil H_2O λ_{max} (nm)	DMF λ_{max} (nm)	Ethanol λ_{max} (nm) (a)	Ethanol + HCl λ_{max} (nm) (b)	Change in λ_{max} (nm) (b-a)
A_1	2. 77	511	525	509	514	+5
A_2	2. 80	498	521	519	515	-4
A_3	3. 28	494	511	510	494	-16
A_4	2. 26	515	422	509	494	-15
A_5	8. 21	449	511	510	511	+1

Table 1.6: Infrared Spectra of the Synthesized Intermediates Based on Naphthalimide Derivatives

A-3067(N. H Str Vibr), 2784(C-H Str Vibr), 2583 (C-H Str Vibr Aliphatic), 2106 (C≡ C Str Vibr Ar), 1622 (C=O Str Vibr), 1670 (-N-C=O Str Vibr),

Table 1.7: FT-IR Analysis of the Synthesized Bifunctional Reactive Dyes

Dye No	FT-IR (cm^{-1})
A_1	3204 (OH Str Vibr) 3070 (C-H Str Vibr) 2121 (C-H Str Vibr) 1778-1707 (C=0 Str Vibr)1599-1299 (C-N Str Vibr) 1648 (N-H bend Vibr) 1372 (N=0 Str Vibr) 709-743 (C-Cl Str Vibr) 877-862 (SO_3H Str Vibr)
A_2	3432 (OH Str Vibr) 2840-2985 (C-H Str Vibr) 1797 (N=N Str Vibr) 1648 (N-H bend Vibr) 1439-1417 (C-N Str Vibr) 1041 (S=0 Str Vibr) 862 (SO_3H Str Vibr) 709-766 (C-CL Str Vibr) 1372 (N-O Str Vibr)
A_3	3592 (OH Str Vibr) 3439 (NH Str Vibr) 2840-2996 (C-H Str Vibr) 2508-2162 (-SCN Str Vibr) 1748 (C=0 Str Vibr) 1648 (N-H bend Vibr) 1588 (N=N Str Vibr) 1369 (N=0 Str Vibr) 1417-1439 (C-N Str Vibr) 728-762 (C-CL Str Vibr) 829 (SO_3H Str Vibr) 992 (SO_3H Str Vibr)
A_4	3435 (OH Str Vibr) 2870-2985 (C-H Str Vibr) 1819 (C-O Str Vibr) 1704-1745 (-N-C Str Vibr) 1588-1652 (-N=N Str Vibr) 724-762 (C-CL Str Vibr) 829-940 (SO_3H Str Vibr)
A_5	3432 (OH Str Vibr) 3238 (NH Str Vibr) 2840 -2985 (C-H Str Vibr) 1704-1748 (C-O Str Vibr) 724-762 (C-CL Str Vibr) 829-992 (SO_3H Str Vibr) 1588-1648 (-N=-N Str Vibr)

Table 1.8: Gas chromatography mass spectroscopy of synthesized intermediate based on naphthalimide derivatives

Intermediate code	Empirical formula	Experimental value of m/z fragment	Corresponding positive charge fragment	theoretical value
A	$C_{24}H_{14}N_4O_4$	43. 0, 75. 0, 105. 0 177. 0, 207. 0 422. 0	$C_2H_5NO_2^+$ $C_6H_5N_2^{+,}$ $C_{12}H_5NO^+$ $C_{12}H_7N_4^+$ $C_{17}H_3N_3O_4^+$ M^+	422.40

About 1.5 mg of finely ground synthesized sample was intimately mixed with about 150 mg of powdered potassium potassium bromide (KBr) in a mortar with a pestle. The finely ground mixtures were introduced between the two bolts (A and A1), and the upper screw A was tightened until the powder was compressed into a thin

disc. After compression, the sample bolts A and A1 were removed, and a steel cylinder with a pellet inside was placed in the path of the IR spectrometer beam, and a blank KBr pellet was placed in the path of the reference beam. The sample was run.

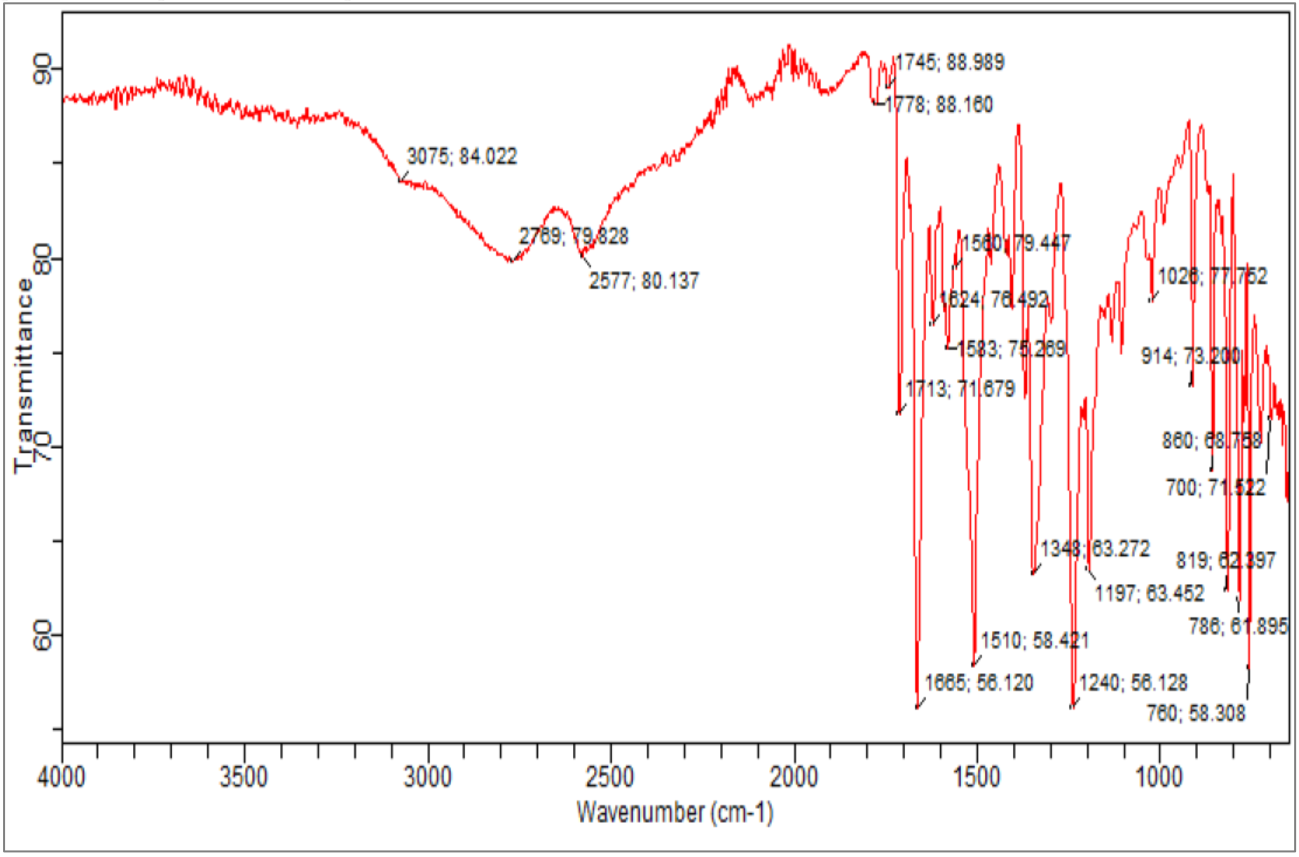


Figure A: TT-IR Spectra of Intermediate A

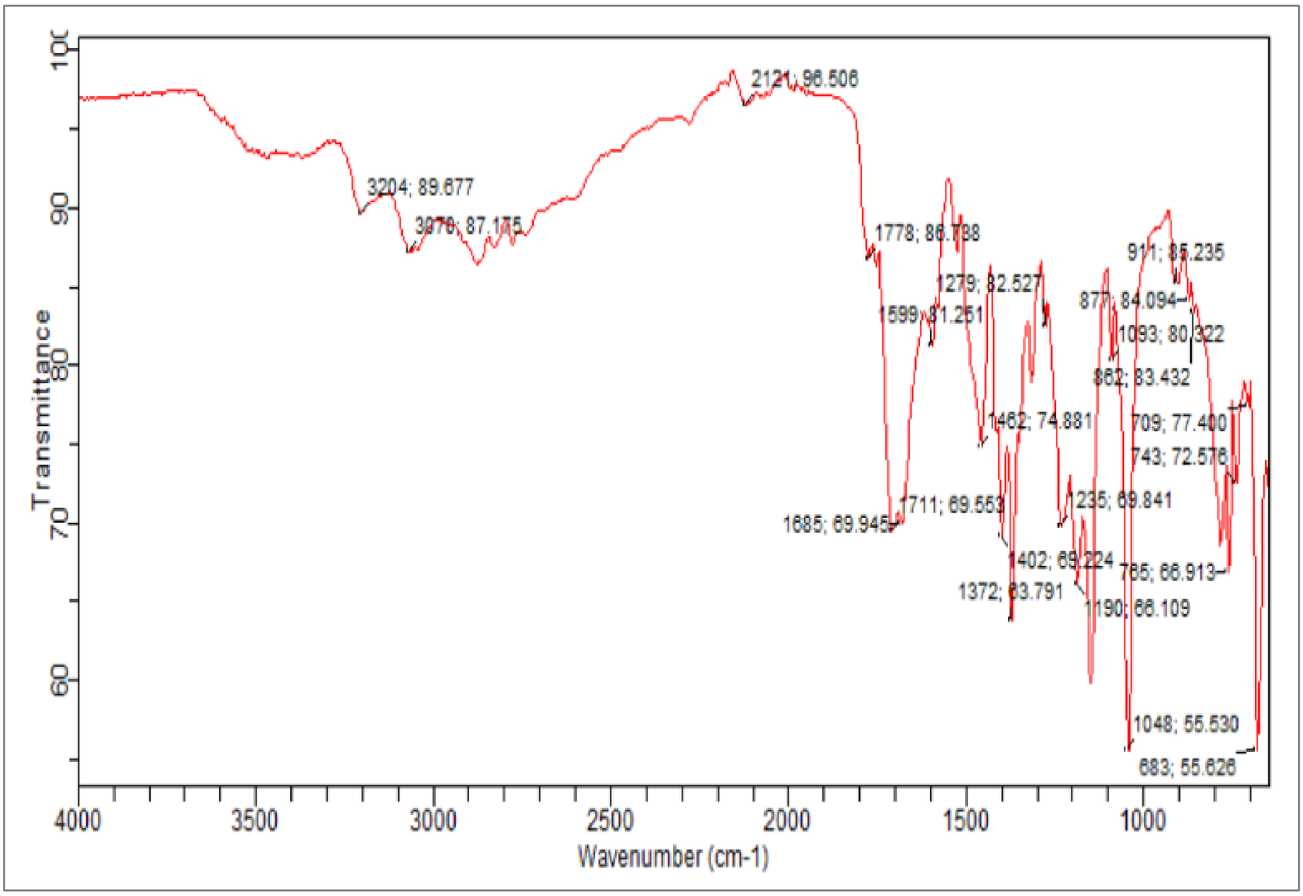


Figure A: 1. FT-IR Spectra of dye A_1

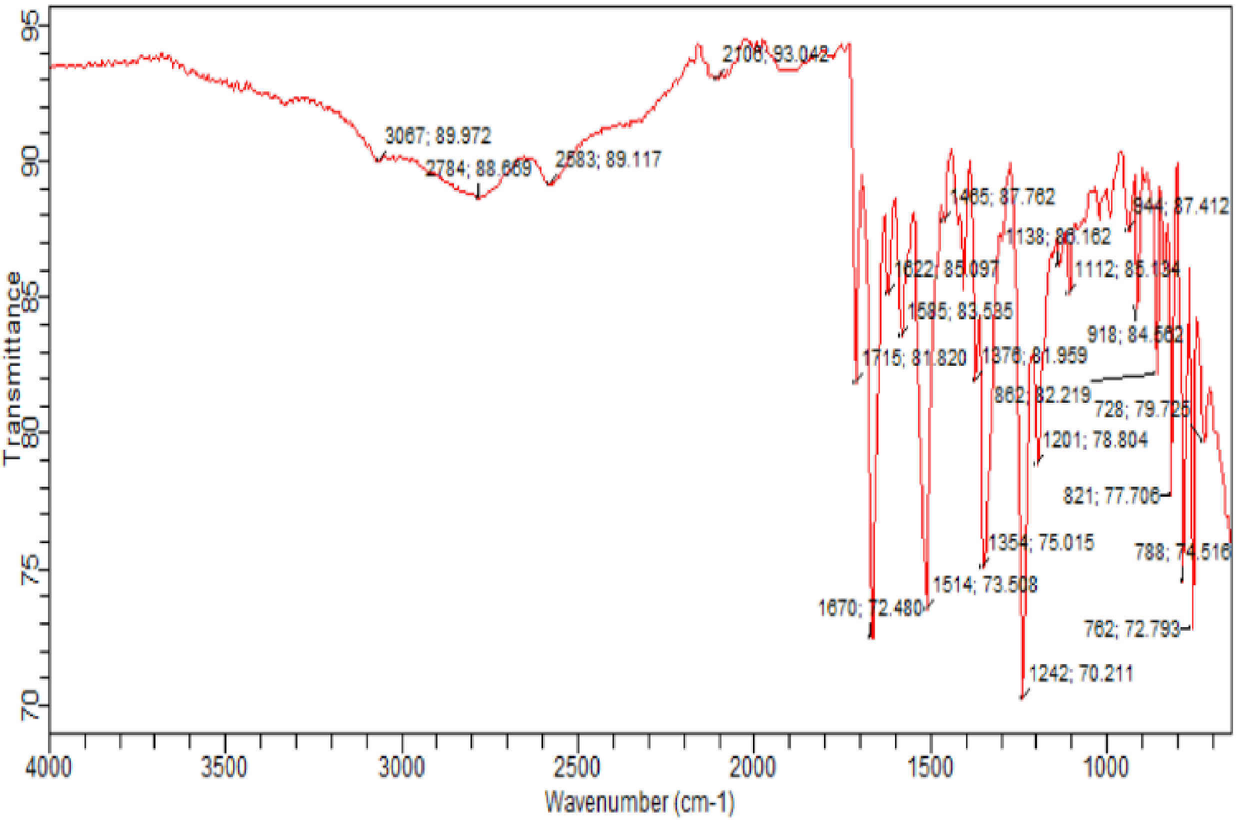


Figure A. 2. FT-IR Spectra of dye: A_2

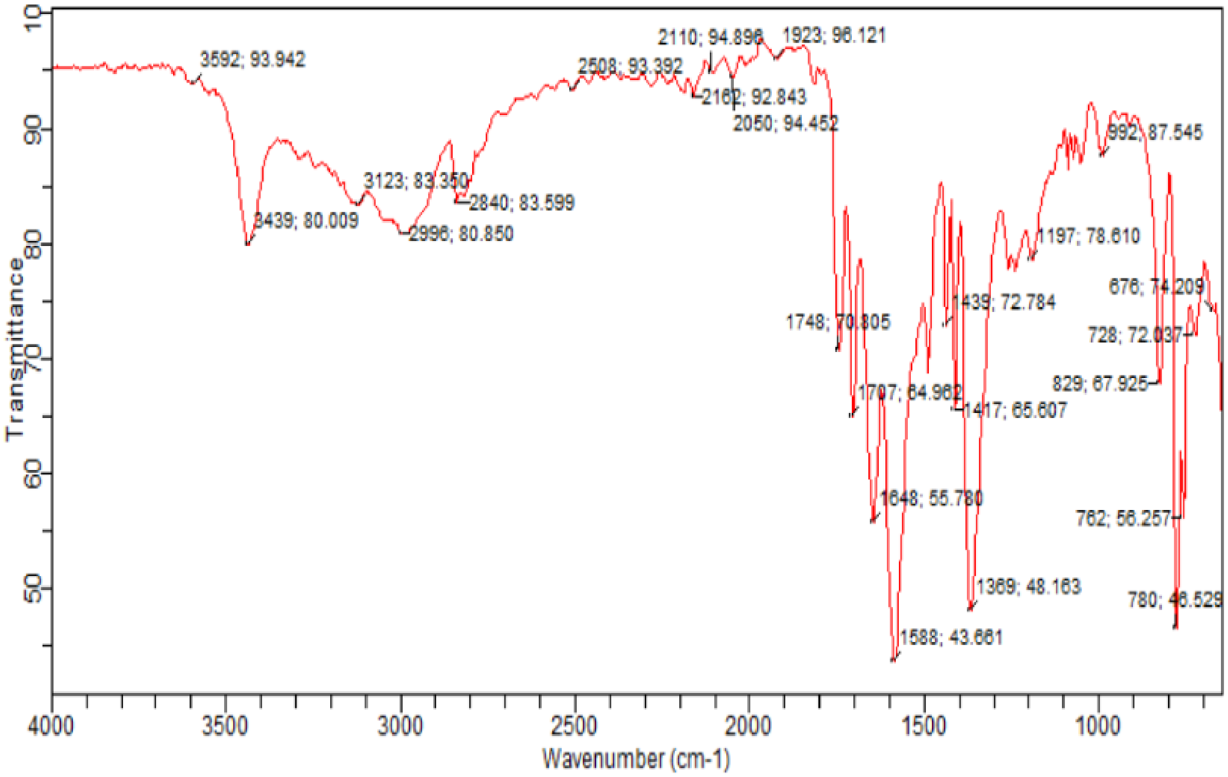


Figure A. 3. FT-IR Spectra of dye A_3

UV-Visible Absorption Measurements

UV-Vis measurements were performed using a PerkinElmer Lambda 25 spectrophotometer in a 1 cm quartz cuvette with distilled water as the reference, and measurements were taken over the 200-800 nm range. The wavelength of maximum absorption of each dye was determined in distilled water, ethanol, Ethanol ± HCl, and

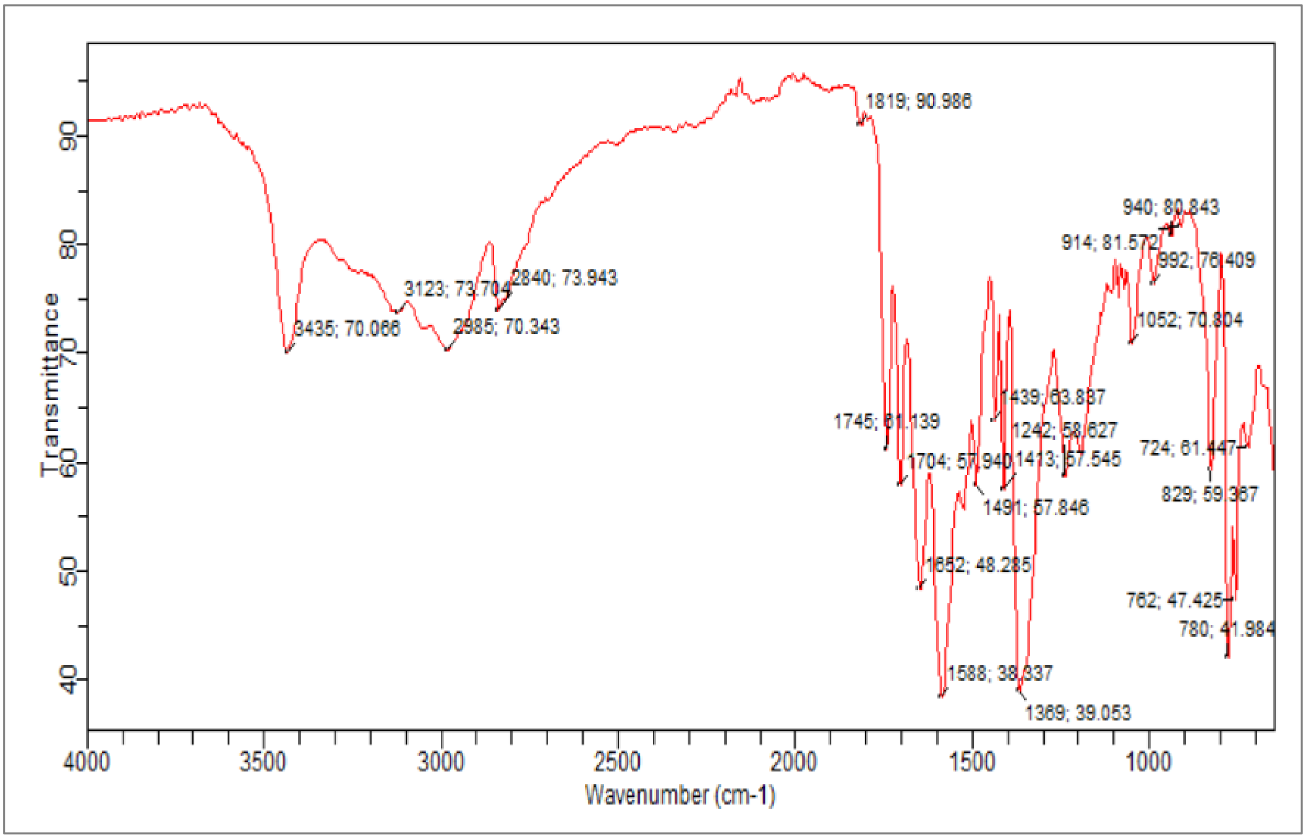


Figure A. 4. FT-IR Spectra of dye A_4

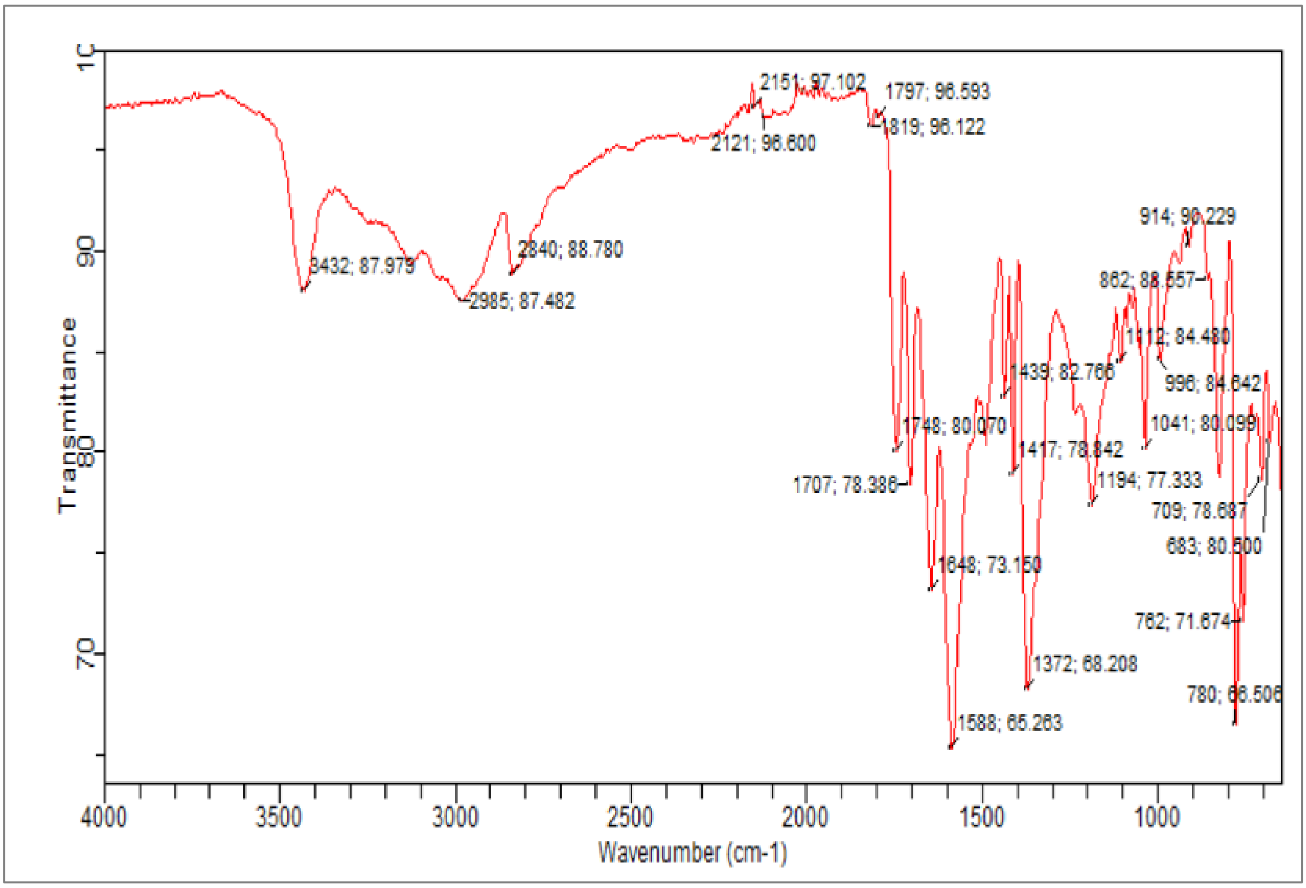


Figure A. 5. FT-IR Spectra of dye A_5

dimethylformamide (DMF). 0.001g of each dye was dissolved in 5 cm^3 of each of the solvents. The spectra of each dye solution were determined and recorded using UV-Visible scanning.

Dyeing of Cotton Fabric

Dyeing Of Cotton with synthesized dyes was carried out using 2 g of fabric, liquor to goods ratio 1:10 0.100 g L1

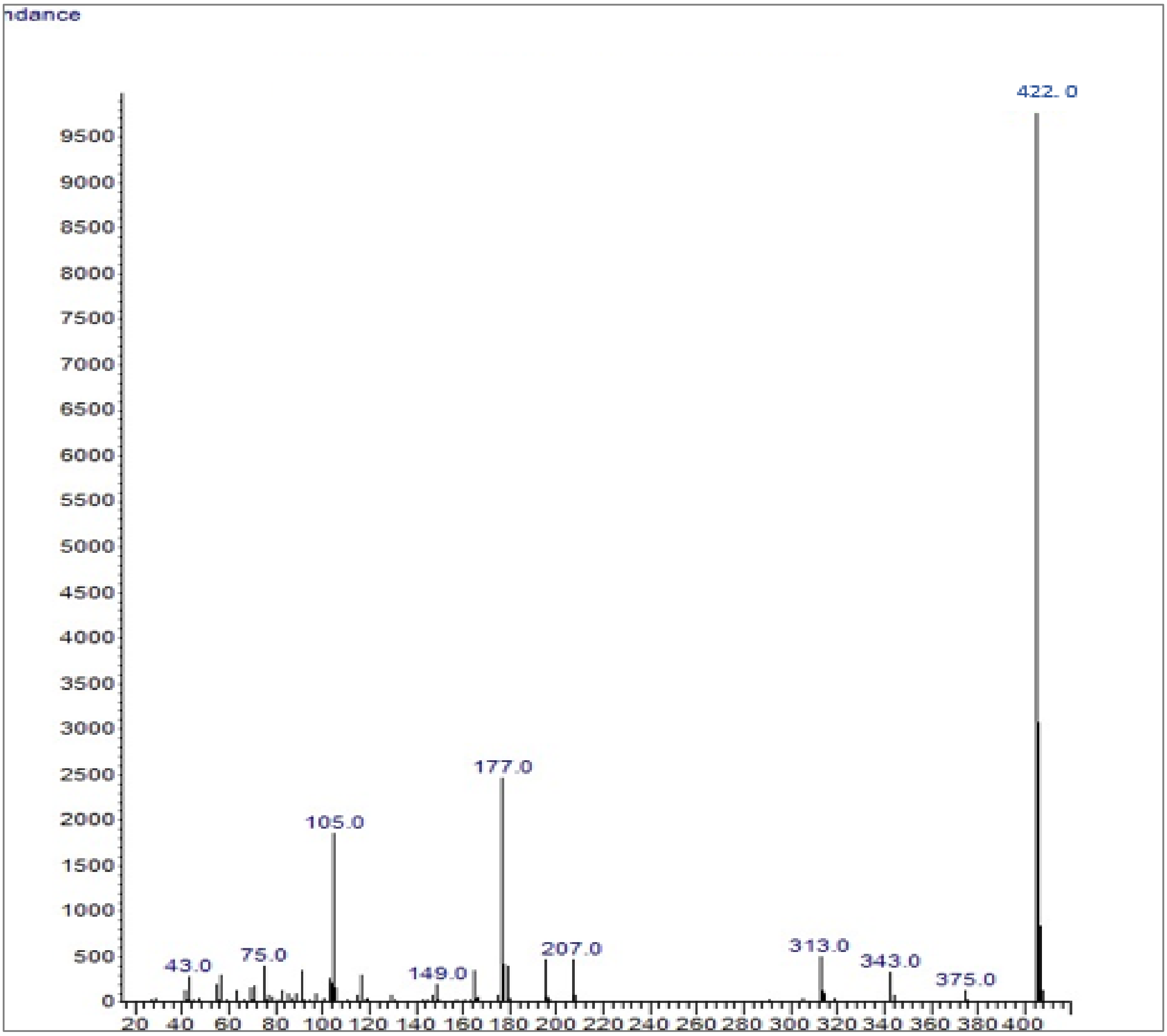


Figure A6: GC-MS Spectra of Intermediate A

Na_2SO_4 (Sodium sulphate), and 10 g L1 Na_2CO_3 (Sodium Carbonate) under the fixation of 60 ℃ isothermal process was used. After dyeing, the shopping was carried out using 2 g L-1 non-ionic surfactant at 90–95 ℃ for 20–30 min, and the sample was lastly cleaned and dehydrated.

Cotton fabric was dyed by the exhaust method using an Ahiba Datacolor IR dyeing machine (Model 2000). Dyebaths were prepared at a concentration of 3% on weight of fabric (owf) using an acetic acid–sodium acetate buffer adjusted to pH 4.5. One gram of cotton fabric was introduced into a dyebath (liquor ratio 50:1) at 40 °C, raised at 1.5 °C/min to 100 °C, and held for 60 minutes with agitation. After dyeing, all the samples were hot rinsed and treated with 1 g/l of soaping agent, followed by cold rinsing and air-drying (Clark, 2011; Tanning of hides and skins 1989).

Determination of percentage dye exhaustion

Dye uptake was determined by measuring the absorbance of diluted dye bath samples at the maximum absorbance wavelength. The bath was sampled before and after dyeing. A 1 mL aliquot was taken from the bath and diluted to 20 mL with 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 3.4 (Kamel, et al.,2014; Sekar and Deulgaonkar, 2007).

$$\% E = \frac{A_o - A_1}{A_o} \times 100\text{----------------------------}3.4$$

Where A_o and A_1 are the absorbance at λ_{max} of the dye bath prior to dyeing and after dyeing, respectively.

Determination of dyed fixation and fixation efficiency

The term fixation ratio refers to the ratio of the amount of dye absorbed (which is covalently bonded to the fibre) to the hydrolyzed dye on the surface of the surface.

$$\text{Percentage fixation} = \frac{OD_1 - OD_2 - OD_3}{OD_{1-OD_2}} \times 100\text{----------------}3.5$$

Where OD_1 = optical density of the dye before dyeing

OD_2 = optical density of the dye after dyeing

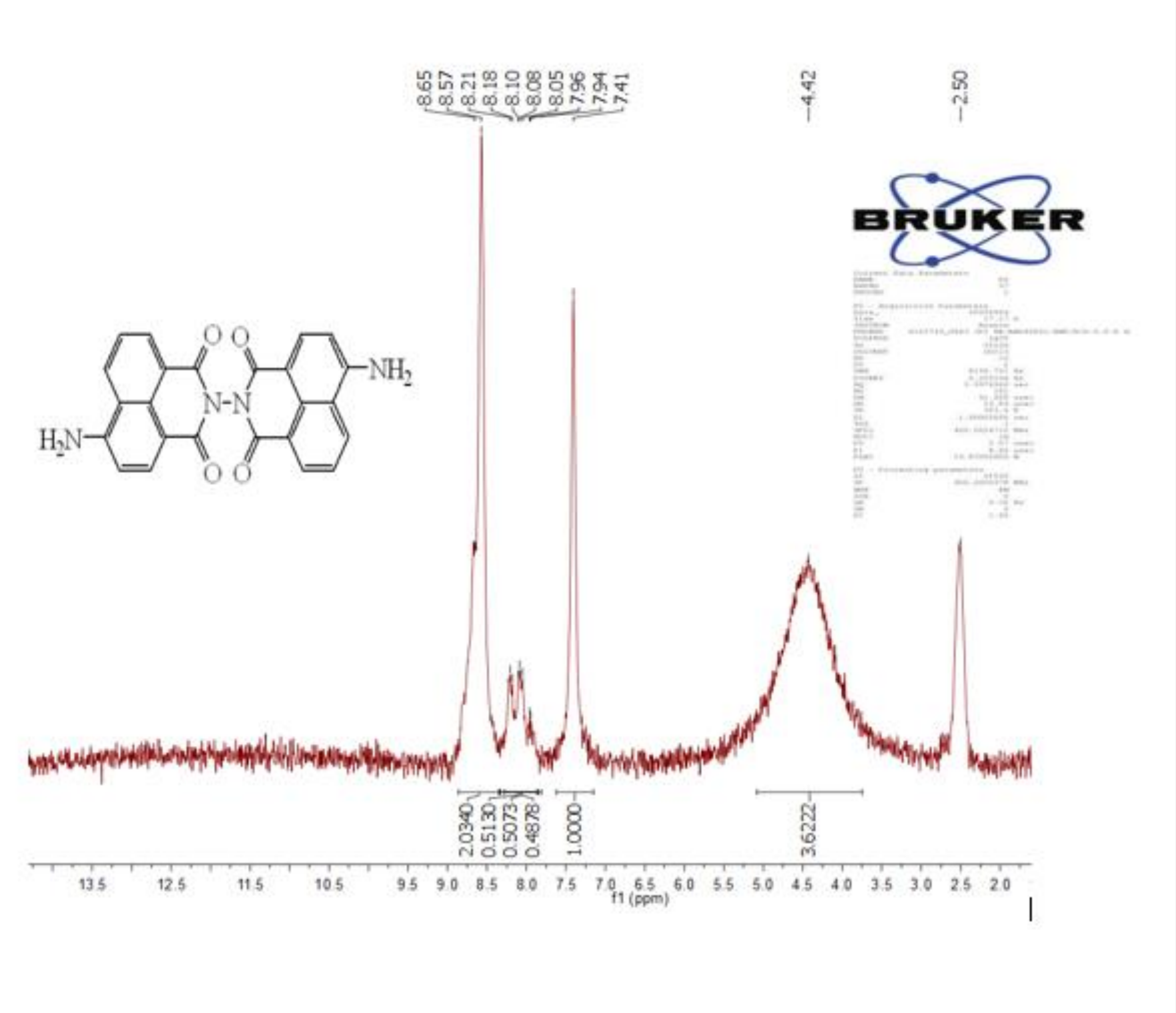


Figure B. ^{1}HNMR spectra of Intermediate A

OD_3 = optical density after stripping

Each dyed fabric was stripped in a 25% (w/w) pyridine in water solution (100 °C, IR 10:1) for 30 minutes, and a portion of the solution was taken to the spectrophotometer to give OD3, the optical density after stripping.

Optical overall fixation efficiency value (T) is related to the original amount of dye applied. A simple relationship exists between % T % F, and % E (dye bath exhaustion) given by:

Fastness to Washing

Using ISO Number 3 test methods, the washing conditions were adopted as shown below for the determination of wash fastness of a dyed sample.

Soap	5 g/l
Soda ash	2 g/l
Liquor ratio	50:1
Temperature	60 + 2 °C (140 + 4°F)
Time	30 min.

The dyed fabric (10 cm x 4 cm) was sandwiched between undyed nylon and undyed cotton fabrics and stitched. The composite sample was washed together using the above specification, and the change in colour and staining of the adjacent fabric were assessed using a grey scale (Choi et al., 2008)

Fastness to Light

Light fastness was determined by exposing the dyed nylon and eight wool blue standards on a Xenotest 150 (Original Hanau, chamber temperature 25-30°C, black panel temperature 60°C, relative humidity 50- 60%, and dark glass (UV) filter system) for 48 hours (Alaa and Tarek, 2006).

Fastness to Perspiration

The perspiration fastness was carried out in both acidic and alkaline solutions using the standard ISO-E04

(I) Alkaline solution

Sodium chloride (5 g/l), disodium hydrogen orthophosphate ($Na_2HPO_4{\cdot}2H_2O$) (2.5 g/l), Histidine mono-hydrochloride monohydrate, ($C_6H_9O_2N_3$. HCl. H_2O) (0. 5 g/l). The solution was brought to pH 8 using 0.1N NaOH solution.

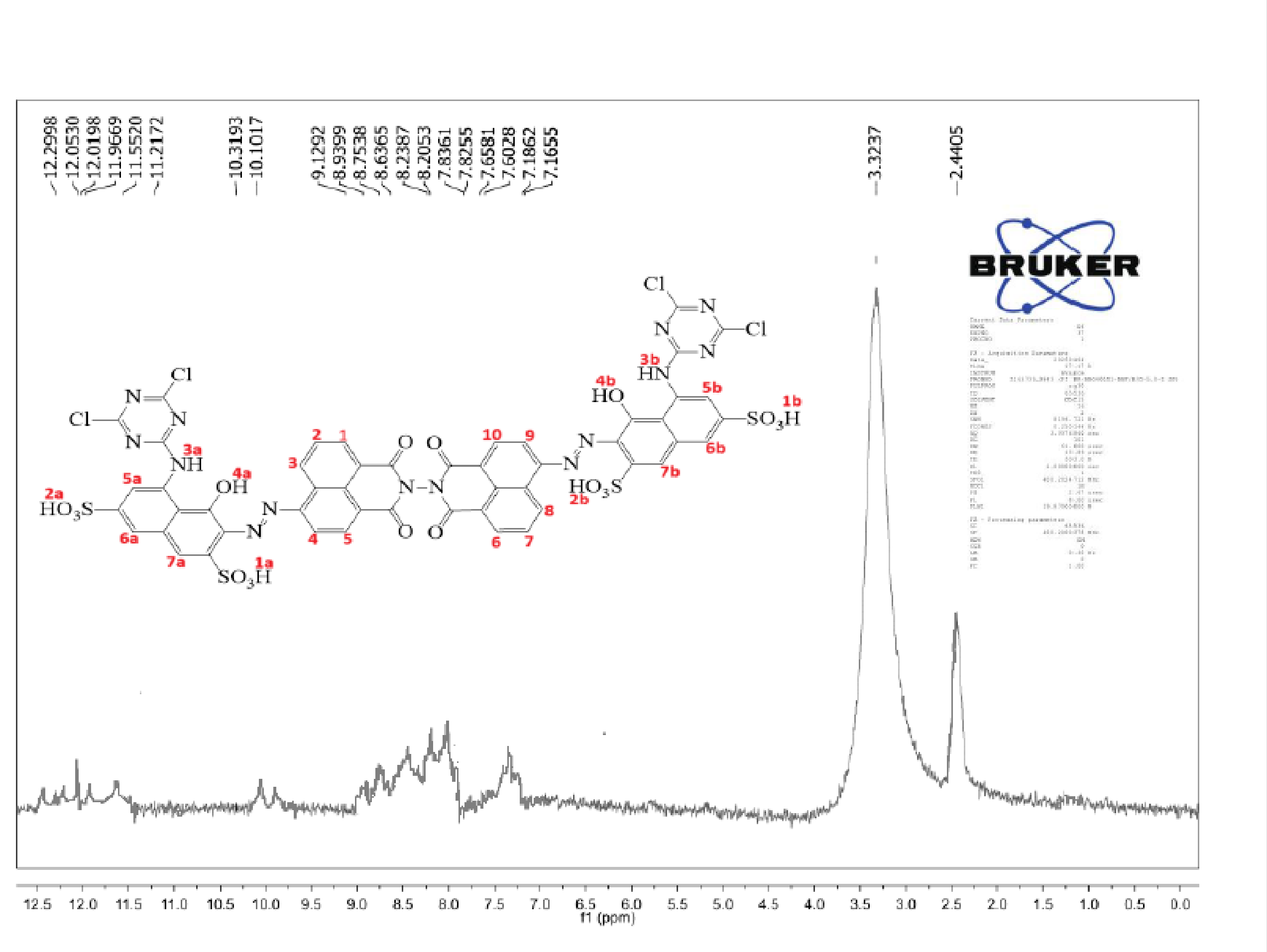


Figure B. ^{1}HNMR spectra of Dye A$_1$

(II) Acidic solution

Sodium chloride (5 g/l), sodium dihydrogen orthophosphate (NaH_2PO_4. $2H_2O$) (2. 2 g/l), Histidine mono-hydrochloride-monohydrate, ($C_6H_9O_2N_3HCl$. H_2O) (0.5 g/l). The solution was brought to pH 5.5 using 0.1N acetic acid solution.

The composite specimen was thoroughly wetted out in the alkaline solution (pH 8.5) at a liquor ratio of 50:1 and kept standing for 30 minutes at room temperature. The specimen was placed between two plates of the perspirometer at a pressure of 12.5 kPa, which was achieved by placing a 4.5 kg weight on the arranged samples in the perspirometer. The perspirometer was set up in the oven at 37 ^{0}C and maintained for 4 hours. The procedure was repeated for the other composite specimens at pH 5.5 for acid, and they were placed in another perspirometer. The samples were removed, opened up, and allowed to dry at room temperature. Both specimens were then assessed for colour change of the test fabric and staining of the adjacent fabric (Koh et al., 2003)

RESULTS AND DISCUSSION

Table 1.9: Exhaustion and fixation of the synthesized bifunctional reactive dyes on cotton fabric and chrome-tanned leather

Dye	% Exhaustion On Cotton /CTL	% Fixation On Cotton	Shade On Fabric
A$_1$	78. 50, 63.10	89. 10	Pink
A$_2$	84. 00, 68.50	90. 00	Purple
A$_3$	87. 20, 75.30	90. 53	Pink
A$_4$	74. 90	68. 30	Ash
A$_5$	81. 30	72. 41	Yellow

CTL=Chrome Tanned Leather

Displayed the structures of synthesized intermediates using acenaphthene as a starting material, and several reaction steps like nitration, oxidation, amination, and reduction were involved to obtain the intermediates, with the colour crystals dark brown, melting point ranging 278-280^{0}C percentage yield ranging from 58-98% and the molecular weight range from 422. 40 (g/mol).

Physical properties of the synthesized bifunctional reactive dyes A$_1$-A$_5$ derive from

Naphthalimide derivatives

The dichlorotriazine dyes A$_1$-A$_5$ have moderate to higher molecular weights ranging from 1186. 76 -2097. 87 (g/mol), which translates to a higher melting point range

Tables 1.10: The fastness properties of the synthesized bifunctional reactive dyes on cotton fabric

Dye No.	Light Fastness	Wash Fastness (CC)	Perspiration Fastness – Acid (CC)	Perspiration Fastness – Alkaline (CC)
A1	5	4	5	5
A2	5–6	4	4	5
A3	5	4	5	4
A4	4	4	5	4
A5	4	5	3–4	5

CC= Colour Change

Table 1.11: Light, wash and perspiration fastness properties of chrome tanned dyed leather.

Dye No.	Light Fastness	Wash Fastness (CC, CTL)	Perspiration Fastness – Acid (CC, CTL)	Perspiration Fastness – Alkaline (CC, CTL)
A1	5	4–5	3–4	4–5
A2	5–6	4	4–5	5
A3	5	4	4	4

CC= Colour Change CTL=Chrome Tanned Leather

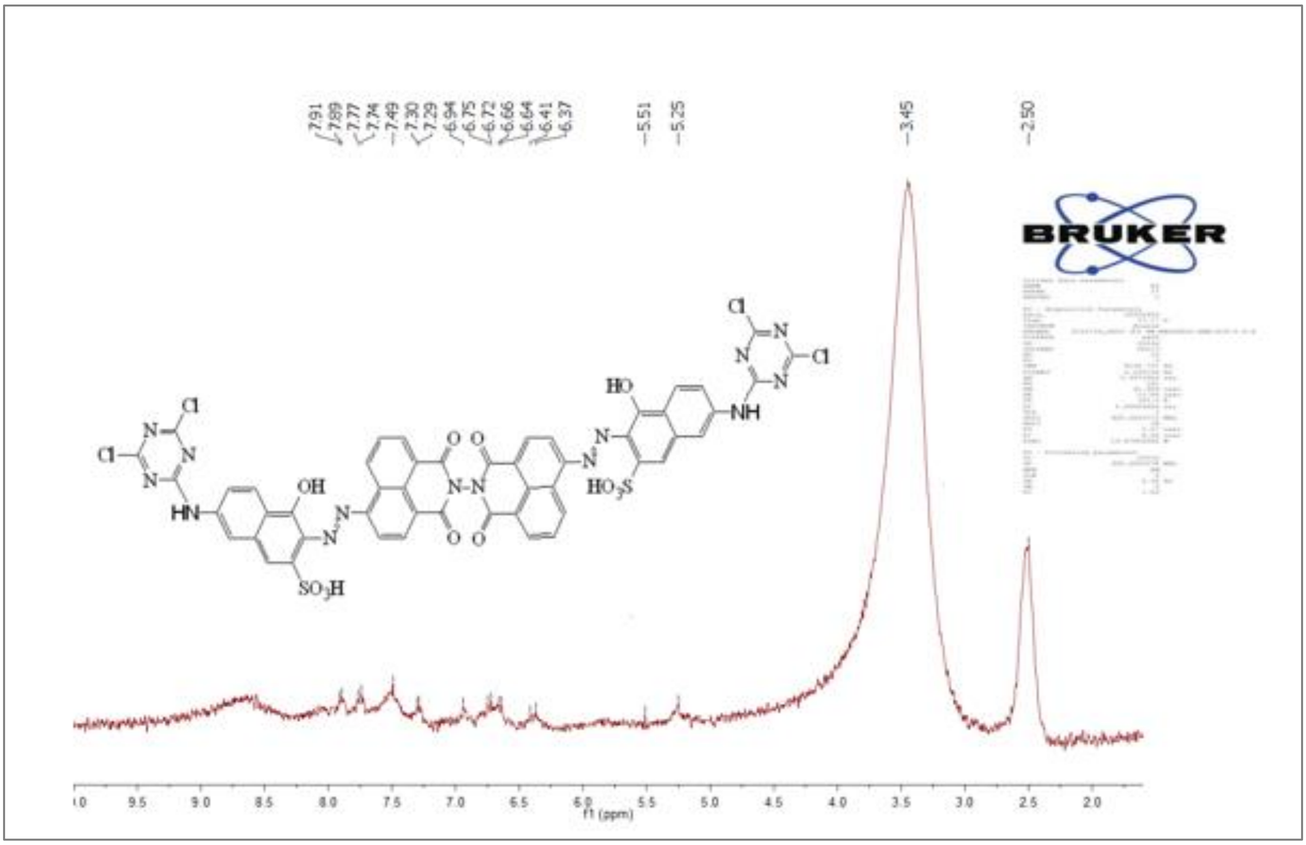


Figure B. ^{1}HNMR spectra of dye A_2

from 300-338 **(℃)**. The percentage yield of the synthesized dyes is also very high, although very few have low yields, ranging from 68-92 %. The high percentage yield recorded indicates that the experimental conditions were controlled (Alaa and Tarek, 2006). The synthesized dyes were also very soluble in water which is an indication that the structure of the synthesized dyes are highly branched and the position of the functional groups in the carbon chains also favours the solubility of the dyes (OH, SO_3 H) (Patel and Patel, 2014; Patel Pratixa et al.,2015);. The colour or hue of the synthesized bifunctional dyes ranges from blue, brown, orange, black, purple, red, and pink due to the presence of functional groups such as NH_2 and -OH on the dye structure.

Spectroscopic characteristics of the synthesised bifunctional reactive dyes A_1-A_5 based on naphthalimide derivatives

The visible absorption maxima of the synthesised bifunctional reactive dyes as recorded in Table 1.7 felt within the visible region (400 - 700 nm) of the electromagnetic spectrum. The values of the molar extinction coefficient (ε) that were determined by Beer-Lambert's law are in the range of 2.26×10^{-4}–8. 21×10^{-4} (L/mol^{-1}cm^{-1}), which is an indication of high absorption intensity of the synthesised bifunctional reactive dyes in DMF, respectively. However, the synthesised bifunctional reactive dyes have different chromophoric functionalities but the same bridging groups, and it is also apparent that the value of λmax depends on the coupling

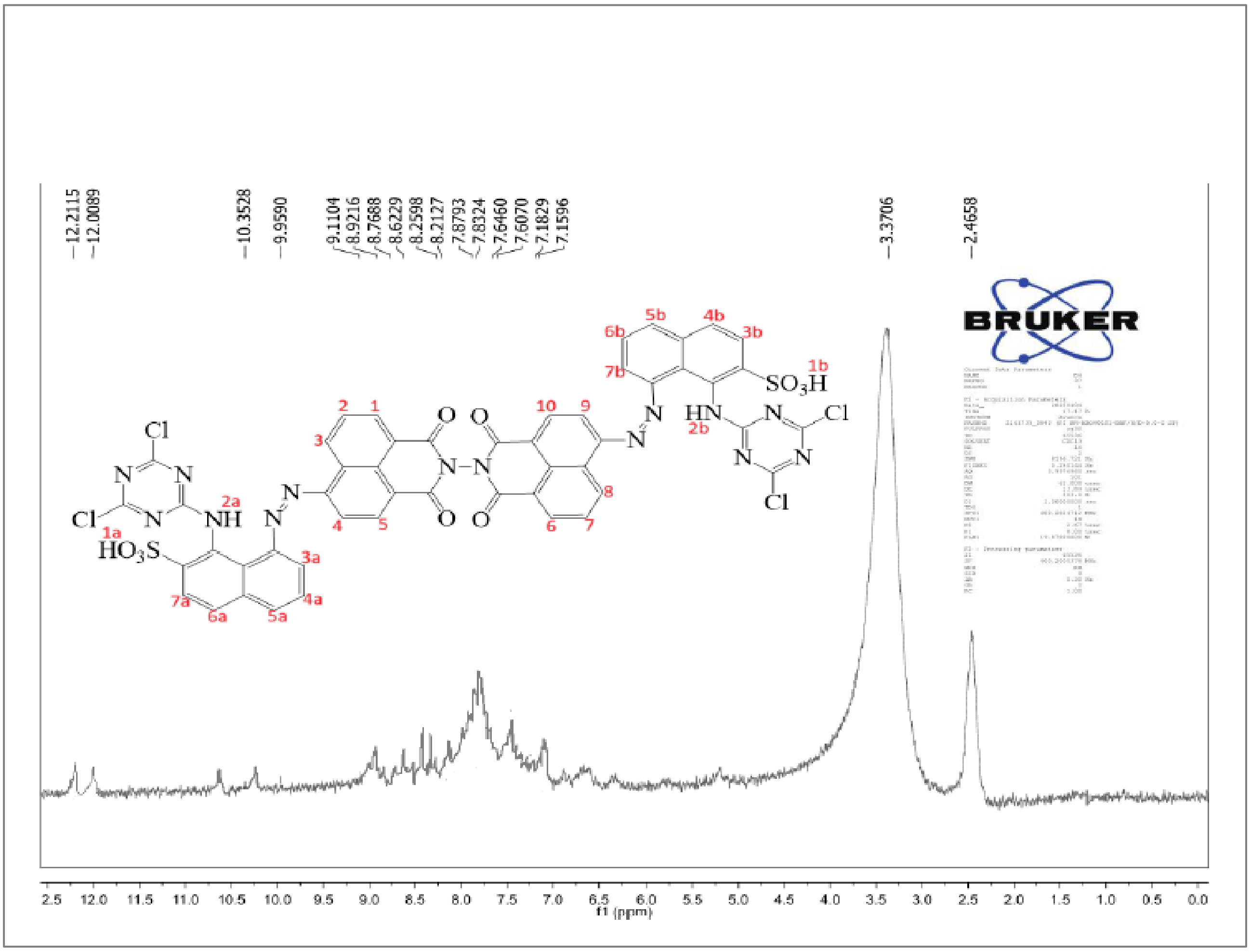


Figure B. ^{1}HNMR spectra of dye A_3

components used. The presence of electron-donating or electron-attracting (withdrawing) groups at suitable positions on the coupler ring affects the absorption characteristics of the synthesised azo-reactive dyes (Patel and Patel, 2014). The wavelength of maximum absorption (⅄max) appeared in the visible region of the spectrum, which is attributed to the p-p*. Reaction of 6, 6-diamino 1H, 1'H, 3h, 3' H (2, 2' – bibenzo [de] isoquinoline] 1, 1'3, 3 – teraone with cyanurated H-acid gave dye A_1, which absorbed at 525 nm in DMF, 511nm in distilled water,509 nm in ethanol and 514 nm in ethanol plus HCL which dye A_1 in DMF is more bathachromic which could be attributed to the presence of electron donating or electron attracting (withdrawing) groups at the suitable position of the coupler ring and polar excited states of the synthesised bifunctional reactive dyes are stabilized by polarization interactions forces as the polarisability of the solvent increases but when cyanurated H-acid was replaced with cyanurated R-ac id which gave dye A_2 and absorbed at 521 nm, which gave hypsochronic shift of 4 mm when compared to dye A_1. This agrees with the findings of Koh and Greaves (2013) and the presence of an electron acceptor group in the teteazo components and an electron-donating group in the coupler. Replacing cyanurated R with J-acid dye A_3 was synthesized, which absorbed at 511 nm in DMF and gave a hypsochromic shift of 14 nm positive solvatochromic when compared to dye A_1 and a hypsochromic shift of 10 nm negative solvatochromic when compared to dye A_2 due to the presence of the sulphonic group and tetra azo component on the coupling components. Meanwhile, replacing J-acid with gamma–acid, dye A4 was synthesized, which absorbed at 422 in DMF and gave a hypsochromic shift of 103 nm compared to dye A1, a hypsochromic shift of 99 nm compared to A2, and a hypochromic shift of 87 nm compared to dye A3, respectively. However, replacing cyanurated gamma-acid with Tobias acid, dye A_5 was synthesized absorbed at 511 nm in DMF and gave a hypsochromic shift of 14 nm when compared to A_1, hyposchromic shift of 10 nm when compared to dye A_2 when compared to dye A_3 at on the same wavelength, when compared to dye A_4 bathochromic shift 89 nm respectively.

Infrared Spectra of the Synthesized intermediate and bifunctional Reactive Dyes A_1-A_5 Based on Naphthalimide Derivatives

6, 6- diamino 1H, 1H, 3H, 3H – (2, 2- bibenzo [de] isoquinoline) -1, 1' 3, 3' – tetraone, being a naphthalimide derivative, is synthesized by the reaction of acenaphthene as a starting material, and several reaction steps like nitration, oxidation, amination, and reduction were involved. The crystal colour, yield, and melting point were dark brown, 58 %, and 278-280 ℃, respectively.

The IR spectrum of the synthesised intermediate A showed all the basic functional groups indicated by the characteristic bands (stretching and bending vibration). The infrared (KBr cm^{-1} Figure 1. A) spectrum of the synthesized intermediate A spectrum shows a signal at 3067 for NH/OH stretching vibration, at 2784 C-H stretching vibration, at 2583 C-H aliphatic stretching vibration, at 2106 C=C alkyne stretching vibration, at 1670-N-C=O stretching vibration, and at 1622C=O stretching vibration. The H' NMR spectrum (Figure B. 1) shows peaks 6. 27-4. 42 ppm, which corresponds to the proton on the amine group. The peaks at 7. 78-7. 94ppm (13H, 7Ht) correspond to the (13H, 7H) at the aromatic region of the spectrum. The peak at 7. 22-7. 41ppm corresponds to the (8H, 12H d) at the aromatic region of the spectrum. The mass spectrometry MS (Figure A6) analysis showed positive charge fragments like $C_2 H_5 No^+$, $C_6 H_5 N^+$, $C_{12} H_5 No^+$, $C_{12} H_7 N^{+}{}_{4}$, $C_{17} H_3 N_3 O_{+}{}^{4}$, M^+ with a corresponding mass to charge ratio (M/Z) of 43. 0, 75. 0, 105. 0, 177. 0, 207. 0, 422. 0. The molecular ion (M+) peak at M/Z is 422.0 which is in agreement with the formula mass 422.40g/mol.

The FT-IR spectral of dye A_1

Dye A_1 was synthesized by tetrazotizing 6, 6-diamino 1H, 1H, 3H, 3'H (2, 2-bibenzo[de] isoquinoline) 1, 1'3, 3-tetraone with cyanurated H-acid. The crystal colour, percentage yield, and melting point were Blue, 82%, and 304-306℃, respectively. The IR spectrum of the synthesized dye A_1 showed all the basic functional groups indicated by the characteristic bands (stretching and bending vibration) at the IR spectra IR vmax cm^{-1}), Figure A, 1,) of the dye A1 showed stretching vibration at 3204 for OH, 3070 for C-H, N-H stretching vibration at 2121. For C-H, 1599 for N=N 1648 for N-H bend vibration 1299 for C-N stretching vibration, at 1372 for N=O, C-C, l709-743 cm^{-1} stretching vibration.

The H'NMR spectrum (Figure B, 2) (400 MHz, DMSO) shows a peak at 2. 0-3. 43 $^{\delta}$ ppm corresponds to the proton on the sulphonic group ($S0_3H$), the peak at 4. 0-5. 25 $^{\delta}$ ppm corresponds to the proton on the amine group (NH), the peak at 5. 35-5. 51 $^{\delta}$ ppm corresponds to the proton on the hydroxyl group (OH). The peak at 7. 22-6. 94 $^{\delta}$ ppm corresponds to the aromatic region of the spectrum. The peak at 7. 78-7. 74 $^{\delta}$ ppm corresponds to the aromatic region of the spectrum.

The FT-IR spectral of dye A_2

Dye A_2 was synthesized by tetrazotizing 6, 6-diamino 1H, 1H, 3H, 3'H (2, 2-bibenzo (de) isoquinoline) 1, 1'3, 3-tetraone with cyanurated R-acid. The crystal colour, percentage yield, and melting point were red, 74%, and 300-302 ℃, respectively. The IR spectrum of the synthesized dye A_2 showed all the basic functional groups, as indicated by characteristic bands (stretching and bending vibrations). The IR spectrum (IR vmax cm) Figure A_2) of the dye A_2 showed stretching vibration at 3432 O-H, -2840-2985 C-H stretching vibration 1791 N=N stretching vibration, 1648 N-H bending vibration 1439-1417 C-N stretching vibration, 1372 N-O stretching vibration. 709-766 cm^{-1} C-Cl stretching vibration. The H'NMR spectrum (Figure B, 2) (400 MHz, DMSO) shows a peak at 2. 0-3. 43 $^{\delta}$ ppm corresponds to the proton on the amine groups (NH), the peak at 10.05-10. 25 $^{\delta}$ ppm corresponds to the proton on the hydroxyl group protons (OH), the peak at 11. 35-12. 25 $^{\delta}$ ppm corresponds to the proton on the sulphuric group (SO_3H). The peak at 7. 16- 8. 15 $^{\delta}$ ppm corresponds to the aromatic group protons of the spectrum.

The FT-IR spectral t of dye A_3

Dye A was synthesized by tetrazotizing 6, 6-diamino 1H, 1H, 3H, 3'H (2, 2-bibenzo (de) isoquinoline) 1, 1'3, 3-tetraone with cyanurated J-acid. The crystal colour, percentage yield, and melting point were Brown, 88%, and 300-302 ℃, respectively. The IR spectrum of the synthesized dye A_3 showed all the basic functional groups indicated by the characteristic of the band. (Stretching and bending vibration. The (IR Vmax cm-1) (Figure A. 3, Appendix A) of the dye A_3 showed stretching vibration at O-H 3592, N-H stretching vibration at 3439 C-H stretching vibration at 2840-2996, -S-C-N stretching vibration 2508-2162, C-O stretching vibration at 1748, N-H bending vibration at 1648, N-O stretching vibration 1369, C-N stretching vibration 1417-1439, N=N stretching vibration at 1588, C-Cl stretching vibration at 728-762 cm^{-1}. The H'NMR spectrum (Figure B, 2,) (400 MHz, DMSO) shows a peak at 12.00-12. 30 $^{\delta}$ ppm corresponds to the proton on the sulphonic group ($S0_3H$), the peak at 9.95-10.35. $^{\delta}$ ppm corresponds to the proton on the amine group (NH), the peak at 5. 35-5. 51 $^{\delta}$ ppm corresponds to the proton on the hydroxyl group (OH) . The peak at 7. 78-9. 95 $^{\delta}$ ppm corresponds to the aromatic protons region of the spectrum.

The FT-IR spectral of dye A_4

Dye A_4 was synthesized by tetrazotizing 6, 6-diamino 1H, 1H, 3H, 3'H (2, 2-bibenzo [de] isoquinoline) 1, 1'3, 3-tetraone with cyanurated γ-acid. The crystal colour, percentage yield, and melting point were Black, 80%, and 308-3010 ℃, respectively. The IR spectrum of synthesized dye A4 showed all the basic functional groups, as indicated by characteristic bands (stretching and bending vibrations). The IR spectrum (IR Vmax cm^{-1}) Figure A-4) of the dye A_4 showed stretching vibration at O-H 3435, C-H stretching vibration at 2840-2985, C-O stretching vibration at 1819 N-C stretching vibration at 1704-1745, N=N stretching vibration at 1588-1652, C-Cl stretching vibration at 724-762 SO_3H stretching vibration 829-940 cm^{-1}.

The FT-IR spectral of dye A_5

Dye A_5 was synthesized by tetrazotizing 6, 6-diamino 1H, 1H, 3H, 3'H (2, 2-bibenzo [de] isoquinoline) 1, 1'3, 3-tetraone with cyanurated Tobias-acid. The crystal colour, percentage yield, and melting point were Orange, 76%, and 300-302 ℃, respectively. The IR spectrum of synthesized dye A_5 showed all the basic functional groups, as indicated by characteristic bands (stretching and bending vibrations). The IR spectrum (IR Vmax cm-1)

Figure A-5) of the dye A_5 showed stretching vibration at OH 3432, N-H stretching vibration 3238, C-H stretching vibration at 2840-2985, C-O stretching vibration at 1704-1748, C-Cl stretching vibration at 724-762, SO3H stretching vibration at 829-992 N=N stretching vibration at 1588-1648 cm^{-1}.

Light fastness

The light-fastness rating of the dyed fabric is shown in Table 1.8. The test is carried out by exposing each dyed sample to daylight alongside eight blue wool standards. It was observe that light fastness of dye A_1, A_2, and A_3 which is obtained by coupling tetrazonium 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 bifunctional reactive dyes gave a light fastness rating (3-5) moderate, good and very good on chrome tanned leather (Clark, 2011). The type of bridging group used in the dye synthesis plays an important part. High ratings of strength properties are given by the covalent bonding connections between the dye and fibre (J.E. Ishegbe et al., 2014) Other dyes gave very good to excellent fastness this, may attributed to the molecular structure of the dyes, planarity and dye substituents that provided shield from radiant energy on azo chromophores, therefore, the this effect conferred good light fastness properties on the dyes (Sekar and Deulgaonkar, 2007).

Washing Fastness

It is a very important requirement that coloured fibres withstand the conditions they may encounter during processing, the following colouration, and their subsequent daily use. Wash fastness testing is important when bifunctional reactive dyes are used, as the dyes are soluble in water. In water if not covalently bonded to the fabric, the dyes may be lost during washing. This makes wash fastness testing an important parameter for assessing this requirement, and it involves determining the resistance of dyes to alkaline washing (Table 1.11). It shows the wash fastness of the dyes coupled with terazonium salt, cyanurated 2-naphthol, 3,6-disulfonic acid, and 8–amino–1–naphthol; 3,6. Disulphonic acid, 2-amino-8-naphthol sulphonic acid. These dyes gave purple, brown, and pink 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 – A_1-A_3) gave a wash fastness rating on chrome-tanned leather (3-5) good, very good, and excellent, while on cotton fabric gave a wash fastness rating (5) excellent. It was found that the substituents with higher electron-donating property afforded lower hydrolysis rate constants. It could be seen that cotton fabric showed better wash fastness than chrome tanned leather, which could be due to the fabric's texture: cotton involves high diffusion and penetration, whereas chrome tanned leather, being an interweave, involves slow diffusion and penetration (Ezeribe, et al., 2013). Bifunctional reactive dyes exhibited good to excellent fastness to washing on cotton fabric while the dyes showed good to excellent fastness to washing on chrome tanned leather respectively and this may be due to the presence of solubilising group and covalent linkage which affect solubility and washing ability of the dye-out of the dyed chrome tanned leather While the wash fastness of the dyes coupled with terazonium salt, each portion of dye A_1 –A_5 solution gave a wash fastness rating on chrome tanned leather (3-5) good, very good and excellent, while on cotton gave a wash fastness rating (4-5) very good and excellent The high ratings of fastness properties could be referred to the covalent binding linkages between the dye and fiber (Ukanah et al., 2020) The wash fastness property of the chrome tanned leather and cotton dye fabrics were generally good very good to excellent.

CONCLUSIONS

In conclusion, we reported the synthesis of bifunctional reactive dyes with naphthalimide derivatives were synthesized starting with the following reaction: nitration, oxidation, amination, and reduction to synthesize the intermediate A [6, 6'-diamino-1H, 1'H, 3H, 3'H-[bibenzo[de]isoquinoline]-1, 1'3, 3'-tetraone,] which was tetrazotised with cyanurated coupling components such as H-acid, R-acid, J-acid, γ -acid, and Tobias acid. The synthesized dyes were purified by the recrystallization method. The structure of the synthesized dyes were comfirm using UV –visible,, FT-IR, MS and ^{1}HNMR spectroscopic technique., Spectrophotometric investigations of the dyes in solvents of different polarities were measured to obtained absorption maxima, molar extinction coefficient and salvatochromic effects These dyes gives percentage yield of 76-88%, molecular weight of 1262.85-1454.98 g/mol, melting point 300-306 ℃ with various hues such as blue, red, brown, black and orange crystals respectively. The planarity of molecules and solvent polarity play important roles in the emission spectra of dyes. The chemical link between cellulose and a covalent bond, which is particularly resistant to acid hydrolysis, gives synthetic colours and their exceptional durability. The MCT groups' presence enhances the dye's substantivity and demonstrates strong wash-off and high fastness. The dyes produced brown, purple, orange, pink, and yellow colours on cotton fabrics and chrome-tanned leather. The difference in colour of the dyes could be attributed to both the nature and position of the substituent and the various coupling components. The dyes gave satisfactory performance, with percentage exhaustion ranging from 70-89% and fixation ranging from 64-93% on cotton fabrics. The fastness properties to washing rating very good to excellent (4-5), to light fastness rating good to very good (5-6), and to perspiration to both the alkaline and acidic rating, very good to excellent (4-5) media of the dyes on chrome tanned leather and cotton fabrics showed good to excellent performance. The presence of the triazine group in the dye molecule improves exhaustion, fixation, and fastness properties. Furthermore, the use of large quantities of salt and alkali is widely considered ecologically unfavorable; therefore, the development of novel modifications to the dye structure and the use of minimum salt for application are of great interest. Furthermore, the use of large quantities of salt and alkali is widely considered ecologically unfavorable; therefore, the development of novel modifications to the dye structure and the use of minimum salt for application are desirable.

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