





Synthesis and Characterization of Diazo Dyes Derived from Aniline Derivatives and Salicylic Acid

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Abstract	Article History
<p>The heterogeneity of diazo dyes obtained from aniline derivatives were artificially manufactured by diazotizing agents such as bromo aniline and nitroaniline with coupled naphthol and salicylic acid. The dyes that contained impurities were recrystallized using ethanol as a solvent. Satisfactorily. The entire manufactured dyes showed good quality and reliability properties of diazo dyes. The diazo dyes were subjected to UV-VIS and IR which indicated the wavelength and functional group of the dyes respectively. The UV spectral of the dyes reveals spectral band position to a longer wavelength in the solvent of different polarities. When dyeing was performed on different materials, polyester material preeminent impact properties. Dyeing was carried out on nylon, cotton and polyester fabrics. Remarkably, nylon gave the earnest dye depth, while polyester has the preeminent fastness properties.</p>	<p>Received: 27 Apr 2025 Accepted: 04 May 2025 Published: 06 May 2025</p>
<p>Keywords: Diazo disperse dyes, Azodoic group, and Fastness properties.</p>	<p>Scan QR code to view*</p> 
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1. Introduction

Dyes are substances with the ability of coloring fabrics in such a way that they cannot be detached by either rubbing or washing (Sharma, 2011). It can also be understood to be a synthetic substance made from natural source, which is configured to chemically enter a material or attach itself on the surface of a material, made to chemically respond with, or settle within a substrate so as to transmit hue to that substrate with some extent of durability. Dyes are known to have hue due to the fact that they absorb light in the visible spectrum (400-700 nm); have not less than one light absorbing pigment; have a conjugated system; and show the bonding or combination of electrons (Burkinshaw, 2016).

2. Methodology

2.1 Experimental Procedures

The diazo disperse dyes were synthesized by diazotization of the different aminobenzene derivatives and coupled with naphthol and salicylic acid as outlined in the general procedure (Method A,B and C). All the reagents used were obtained from profitable sources. The melting point was measured using Melting point apparatus (Electrothermal 9100) and are uncorrected. IR spectra were recorded in the solid state

using Infra-red spectrometer (Buck Scientific Infrared Spec M530).

Method A: General Procedure for Diazotization:

In separate beakers containing 0.005 moles of aminobenzene derivatives (0.69 g of 2-nitroaniline; 0.69 g of 3-nitroaniline; 0.86 g of 3-bromoaniline), 100 cm³ of concentrated hydrochloric acid was poured in and this was without delay placed in an ice bath to maintain the temperature below 5 °C. 6.9 g of NaNO₂ was dissolved in 50 cm³ of water and the solution was added slowly to the HCl solution using a dropper while stirring continually for about 10 minutes to give solution of a diazonium salt. The solution was then kept below 5 °C in an ice bath (Sharma, 2011).

Method B: General Procedure for First Coupling

Into separate beakers containing 0.005 moles of the coupling components of choice, (0.72g of 1-naphthol; 0.72g of 2-naphthol; 0.69g of salicylic acid), a solution of NaOH made by dissolving 15.00 g of NaOH pellets in 150.00 cm³ of distilled water, was poured into the beaker. The solution was maintained beneath 5 °C. The prepared diazonium salt solution (from Method A) was added slowly to the coupling component

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solution using a dropper while stirring for about 30 minutes. This was left in an ice bath for an additional 20 minutes to ensure the reaction was completed. The synthesized monoazo dyes were filtered, washed thoroughly with water and air dried. The dried monoazo dyes were labelled, and were preserved and presented for the second diazotization reaction.

2.2 Preparation of Diazo Dyes

2.2.1 Method C: General Procedure for Second Diazotization

In separate beakers containing 0.005 moles of dried monoazo dye of interest, 100 cm³ of concentrated hydrochloric acid was poured in and this was immediately placed in an ice bath to sustain the temperature beneath 5 °C. 6.9 g of NaNO₂ was dissolved in 50 cm³ of water and the solution was added slowly to the HCl solution using a dropper while stirring continually for about 10 minutes to produce a solution of a diazonium salt. The solution was then kept below 5 °C in an ice bath (Sharma, 2011).

2.3 Dyeing of Fabric

Dye Application on Nylon and Cotton Fabrics

Separately, the synthesized dyes were used to dye cotton and nylon fabrics. The prepared fabric was introduced into the dye bath at a temperature of 50 °C. The temperature was later increased to 100 °C and dyeing continued at this temperature for an additional 10 minutes. After dyeing, the fabric was removed, rinsed thoroughly under running water and air dried (Mohammed, Farouk, Youssef and Mousa, 2013; Salah and El-Badry, 2012). Thereafter, they were presented for various fastness tests.

2.4. Dye Application on Polyester Fabric

Polyester fabric was dyed by using dimethylformamide in place of water. A dye liquor ration of 1:50 was used. 2 g of the dye of interest was dissolved in 100 g of dimethylformamide. Polyester fabric material was put to the dye bath at a temperature of 30 °C and dyeing continued as the temperature increased to 100 °C. After dyeing, the fabric was removed, rinsed thoroughly under running water and air dried (Mohammed, Farouk, Youssef and Mousa, 2013; Salah and El-Badry, 2012). Thereafter, they were presented for various fastness tests.

2.5 Wash Fastness Test

The dyed fabric was placed on a white background and ironed for 40 seconds using a pressing iron set at about 45 °C (mid-heat). The ironed sample was compared with the control sample to see if any color change had occurred. The changes in color shades were examined with respect to a gray scale rating (grade 1-5), where 1 is poor and 5 is excellent (Ukponmwan, Odilora, Ofor and Freeman, 1999; Odilora and Omatseye, 2000).

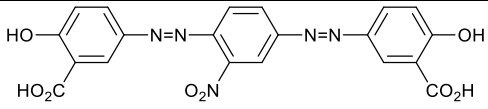
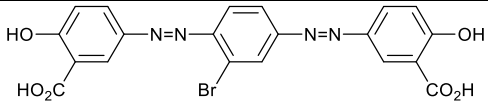
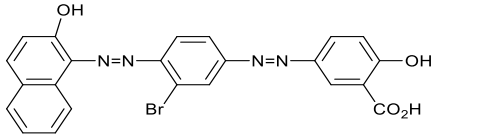
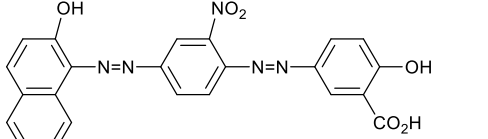
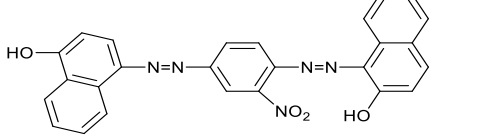
2.6 Light Fastness Test

The dyed fabric was strongly attached to a white background and exposed to sunlight for 3 hours. The side of the fabric which was exposed to sunlight was matched with the control sample to see if any colour change had occurred. The changes in colour shades were examined with respect to a gray scale rating (grade 1-5), where 1 is poor and 5 is excellent (Otutu, Efurhievwe.

3. Results

The synthesized diazo disperse dyes were systematically characterized through structural, physical, spectroscopic, and application-based evaluations, as presented in Tables 1 to 5.

Table 1: Names and proposed structural formula of the manufactured diazo

1		5,5'-((2-nitro-1,4-phenylene)bis(diazeno-2,1-diyl))bis(2-hydroxybenzoic acid)
2		5,5'-((2-bromo-1,4-phenylene)bis(diazeno-2,1-diyl))bis(2-hydroxybenzoic acid)
3		5-((3-bromo-4-((2-hydroxynaphthalen-1-yl)diazenyl)phenyl)diazenyl)-2-hydroxybenzoic acid
4		2-hydroxy-5-((4-((2-hydroxynaphthalen-1-yl)diazenyl)-2-nitrophenyl)diazenyl)benzoic acid
5		4-((4-((2-hydroxynaphthalen-1-yl)diazenyl)-3-nitrophenyl)diazenyl)naphthalen-1-ol

6		2-hydroxy-5-((4-((4-hydroxynaphthalen-1-yl)diazenyl)-2-nitrophenyl)diazenyl)benzoic acid
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Table 2: Physical Properties of Synthesized Diazo Dyes

Dye no	Hue	Texture	Melting Point (°C)	% yield	Weight (g)
1	Dark lemon	Crystalline	317	78	5.61
2	Grey green	Crystalline	295	62	6.40
3	Dark grey green	Crystalline	304	40	5.35
4	Black	Powdered	375	52	3.77
5	Dark brown	Powdered	411	55	3.89
6	Chocolate	Powdered	375	89.4	3.03

The chemical shape of the Diazo disperse dyes synthesized were verified by IR spectrometry and the spectra reveals the presence of aromatic ring, OH, C=C, COOH, C-NO₂ and the halogen group (Br) as shown in table 3.

Table 3: Infra-red spectra of the Disperse Dyes

IR Peaks (CM ⁻¹)							
V _{0-H}	V _{C-H}	V _{AR-H}	V _{C-C}	V _{N-N}	V _{COOH}	V _{C-BR}	V _{C-O}
3450-3805	2602-3610	1617-1580	1600-1560	1600-1450	805-730	665-600	1600-1400

Table 4: Wavelength of maximum absorption (λ_{Max}) of the synthesized Diazo dispersed dyes.

Dye	Solvents	λ_{Max}
1	Methanol	621
	Ethanol	614
	Ethyl acetate	596
2	Methanol	624
	Ethanol	616
	Ethyl acetate	594
3	Methanol	632
	Ethanol	619
	Ethyl acetate	601
4	Methanol	631
	Ethanol	629
	Ethyl acetate	611
5	Methanol	637
	Ethanol	631
	Ethyl acetate	609
6	Methanol	628
	Ethanol	622
	Ethyl acetate	603

Table 5: Fastness Ratings of the Synthesized Diazo Dyes on Cotton, Nylon and Polyester Fabrics

Dye no	Wash fastness			Heat fastness			Light fastness		
	Polyester	Cotton	Nylon	Polyester	Cotton	Nylon	Polyester	Cotton	Nylon
1	4	3-4	3	4	3-4	3	4	4	3-4
2	4	3-4	3	4-5	4	3	4-5	4	2-3
3	4	4	3-4	4	3-4	3	4-5	4	3
4	4	3-4	3	4	3-4	3	4	4-5	3
5	3-4	3	3	4	3-4	3	4-5	4	3
6	4	3	2-3	4	3-4	3	4	4	2-3

4. Discussion

The general structures of the manufactured dyes revealed the presence of chromophore group which gives the dye its colour such as $-\text{NO}_2$, $\text{C}=\text{C}$, $\text{C}=\text{O}$, $-\text{N}=\text{N}-$ and auxochrome group which enhances the colour of the dye such as OH , NH_2 and

affinity group helps bind the dyes to the substrate for example $-\text{COOH}$. The UV spectral of the dyes reveals spectral bands position to a longer wavelength in the solvents that have different polarities. The results also discovered that the dyes absorbed at the visible region of the electromagnetic spectrum. Again, this explains why the dyes are coloured.

5. Conclusion

This analysis have revealed that introducing electron eliminating groups on the benzene constituent of the Diazo disperse dyes played a remarkable impact on the wavelength and colour shade of the dyes. The entire dyes manufactured have moderate to very good fastness features. However, the wonderful fastness characteristics and good colour shade of these group of dyes could play a essential function in dye manufacturing industry.

Declarations

Availability of Data and Materials: The datasets used and/or analysed during the current study are available from the corresponding author upon reasonable request.

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