



RESEARCH ARTICLE

SYNTHESIS OF SOME NOVEL AZO DYES AND THEIR DYEING, REDOX PROPERTIES

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ARTICLE INFO

Article History:

Received 11th May, 2025
Received in revised form
24th June, 2025
Accepted 19th July, 2025
Published online 30th August, 2025

Keywords:

Azo Dyes, Diazotization textile, Cyclic
Voltametry, Redox, Hazardous.

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ABSTRACT

Azo dyes are commonly used colorants in consumer goods like leather, textiles, cosmetics, and agriculture. They are also used as indicators in labs. The synthesis of several new azo dyes, beginning with 3-amino salicylic acid, is described in this paper. The produced dyes' redox potentials were examined, and their capacity to dye cotton textiles. By using electronic absorption, infrared, ¹H NMR, ¹³C NMR. the products' structure was verified.

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Citation: Nagesh D. Gattuwar. 2025. "Synthesis of some novel azo dyes and their dyeing, redox properties." *International Journal of Current Research*, 17, (08), 34466-34468.

INTRODUCTION

Large-scale synthetic dye production is widely used in a variety of industries, including textile dyeing, paper printing, color photography, pharmaceuticals, food, and cosmetics. The textile sector is one of the biggest consumers of these dyes¹. Azo dye-containing industrial effluents pose a risk to human health because, in anaerobic environments, they can transform into toxic and/or cancer-causing substances². Most significantly, these are subjected to reductive cleavage, which produces aromatic amines with known mutagenic and/or carcinogenic qualities. As a result, it is illegal to use some azo dyes anywhere in the world. Diazotization of the primary aromatic amine is the general process for creating azo dyes³⁻⁵. This is followed by coupling with one or more nucleophiles; hydroxy and amino groups are frequently utilized coupling components. The dyeing capabilities of novel mordant and disperse azo dyes made by coupling different diazo solutions of aromatic amines with 2,4-dihydroxybenzophenone have been investigated. Alkali-clearable azo disperse dyes with a fluoro sulfonyl group based on naphthylimide were created. These dyes demonstrated good leveling, wash and sublimation fastness, and moderate light fastness when applied to polyester fabric using the HT method.⁷ The study of dye stability and structure heavily relies on thermal analysis. One of the primary requirements for dyes used in high-temperature processes like

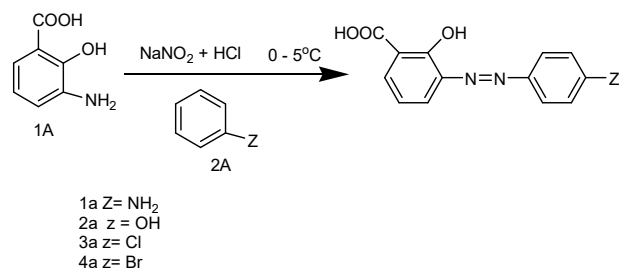
dyeing, printing, and photocopying, as well as in high-tech fields like lasers and electro-optical devices, is their ability to withstand heat at elevated temperatures. Thermogravimetry (TG) was used to examine the thermal behavior of azo dyes with sterichindered groups and identify the products of pyrolysis⁸.

MATERIALS AND METHODS

The suppliers of the chemicals and reagents used were Merck Chemicals in India. Electronic absorption studies were conducted using a UV-visible spectrometer (UV-160 Shimadzu) with a solution of the synthesized compounds in dimethyl sulphoxide. The absorbance values are provided in the bracket, and the absorption maxima are measured in nm. Shimadzu 8300 spectrometers were used to record KBr pellet infrared spectra. On a Bruker Supercon 400 MHz spectrophotometer, the ¹H and ¹³C NMR spectra were captured with TMS serving as an internal standard and CDCl₃ as the solvent. In δ ppm, the chemical shifts are expressed. In order to visualize the spots under UV light, thin layer chromatography (TLC) was carried out on pre-coated Silica Gel sheets (HF 254, sd-fine) using n-hexane: ethyl acetate (7:1) eluent.

General process for making azo dyes: 3-amino-2-hydroxybenzoic acid and 15 ml of dil. hydrochloric acid and

25 ml of water were added, and the mixture was cooled to 0°C in water bath.



A cold solution of NaNO₂ (0.01 mole) in water (8 ml) was gradually added to this cold solution while the flask was being continuously shaken. A cold solution of freshly distilled 2A (0.01 mole) in dil. hydrochloric acid was added to the pale yellow solution of diazonium chloride that resulted from the reaction's completion. to this yellow solution; The progress of the reaction was monitored by TLC. After the completion of the reaction, the solid separated was filtered and recrystallized from dimethyl sulphoxide. The reaction afforded 2-Hydroxy-3-[(4- amino-3-methylphenyl)diazenyl]benzoic acid (4a) in 62% yield, mp : 94°C. The same procedure was used in all cases, of 1a to 4a the compounds.

RESULTS AND DISCUSSION

1a): Obtained as yellow solid in 62% yield. UV-Vis: λ_{max} 418 (0.251) nm. IR (KBr): 3434, 3210, 3052, 2910, 1616, 1440 cm⁻¹. ¹H NMR (CDCl₃): δ 5.15 (s, 1H, OH), 6.21 (s, 2H, NH₂), 6.85 (dd, 2H, Ar-H), 7.56-7.90 (m, 5H, Ar-H), 10.86 (s, 1H, COOH). ¹³C NMR (CDCl₃): δ 113.1 (2C), 115.0 (1C), 121.3 (1C), 125.4 (3C), 129.1 (1C), 132.0 (1C), 145.6 (1C), 150.6 (1C), 156.5 (1C), 171.3 (1C). Anal. Calcd. for C₁₃H₁₁N₂O₃; MW 257 : C, 60.70, H, 4.31, N, 16.33%;

Table 1. Redox Properties of synthesized dyes (1a-1d)

Compound	Oxidation				Reduction				Scan rate mV/sec
	E ₁ (mV)	I ₁ (μA)	E ₂ (mV)	I ₂ (μA)	E ₁ (mV)	I ₁ (μA)	E ₂ (mV)	I ₂ (μA)	
1a	465	-9.48	921	-10.75	266	14.76	--	--	10
1b	425	-32.6	896	-11.32	258	12.66	11.22	--	20
1c	578	-11.95	867	-12.75	283	13.09	--	--	20
1d	481	-13.14	--	--	401	14.1	--	21.6	50

1b Obtained as golden solid in 54% yield. UV-Vis: λ_{max} 426 (0.306) nm. IR (KBr): 3438, 3213, 3051, 2909, 1624, 1455 cm⁻¹. ¹H NMR (CDCl₃): δ 2.22 (s, 3H, CH₃), 5.22 (s, 1H, OH), 6.12 (s, 2H, NH₂), 6.75 (d, 1H, Ar-H), 7.70-7.94 (m, 5H, Ar-H), 10.80 (s, 1H, COOH). ¹³C NMR (CDCl₃): δ 17.2 (1C), 112.1 (1C), 121.4 (1C), 122.0 (1C), 122.6 (1C), 125.0 (2C), 128.2 (1C), 129.4 (1C), 140.2 (1C), 155.2 (1C), 170.1 (1C). Anal. Calcd. for C₁₄H₁₃N₃O₃; MW 271 : C, 61.99, H, 4.83, N, 15.49%;

1c Obtained as yellow solid in 54% yield. UV-Vis: λ_{max} 449 (0.325), 541 (0.099) nm. IR (KBr): 3446, 3221, 3060, 2919, 1722, 1632, 1431, 1328 cm⁻¹. ¹H NMR (CDCl₃): δ 5.30 (s, 1H, OH), 6.22 (s, 2H, NH₂), 7.10 (d, 1H, Ar-H), 7.75-7.98 (m, 5H, Ar-H), 10.68 (s, 1H, COOH). ¹³C NMR (CDCl₃): δ 108.3 (1C), 115.3 (1C), 119.2 (1C), 121.4 (1C), 123.0 (1C), 125.2 (1C), 129.1 (1C), 133.5 (1C), 137.8 (1C), 141.4 (1C), 151.2 (2C), 156.1 (1C), 170.1 (1C). Anal. Calcd. for C₁₃H₁₀N₄O₅;

MW 302 : C, 51.66, H, 3.33, N, 18.54%; Found: C, 51.60, H, 3.31, N, 18.48%.

1d Obtained as faint solid in 54% yield. UV-Vis: λ_{max} 474 (0.493) nm. IR (KBr): 3448, 3232, 3031, 2924, 1635, 1475 cm⁻¹. ¹H NMR (CDCl₃): δ 5.30 (s, 2H, OH), 7.12 (dd, 2H, Ar-H), 7.75-8.14 (m, 5H, Ar-H), 10.84 (s, 1H, COOH). ¹³C NMR (CDCl₃): δ 115.1 (2C), 123.2 (2C), 158.7 (1C), 145.6 (1C), 121.8 (2C), 133.1 (1C), 115.3 (1C), 157.2 (1C), 125.2 (2C), 170.2 (1C).

Table-1: Oxidation-reduction potentials of compounds 4a-f measured by cyclic voltammetry.

Redox Potentials: The cyclic voltammetry studies on the synthesized compounds 1a- 4a has been carried out at the concentration of 25 mM in DMSO as solvent. Britton Robinson buffer (0.04 M of H₃PO₄, H₃BO₃, CH₃COOH) and 0.2 M NaOH solution was used as supporting electrolyte and pH was maintained at 2. A stream of nitrogen was passed into the reaction mixture and then was thermostat. The potential scan rate, current sensitivity, initial potential and final potential were recorded. The resulting current was measured as a function of applied potential. The results were summarized in table-1. According to the study's findings, the produced compounds experience oxidation and reduction under particular circumstances. The compounds' redox potentials imply that they could be employed as indicators in redox titrations. Applications in textiles:

Textile applications: The ability of the synthesized compounds 1a–1d to dye cotton fabrics has been investigated. On cotton textiles, compounds 4c and 4f demonstrated good dye absorption qualities, whereas the other compounds displayed worse absorption capabilities.

These dyes had a high absorption intensity and produced vivid, intense colors on cotton textiles, according to the results of electronic absorption spectra. According to measurements of the dyed fabrics' color parameters, the dyes have very good fastness qualities, intense absorption, and excellent absorption on cotton fabrics.

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