

# SYNTHESIS, SPECTROSCOPIC STUDIES AND FASTNESS PROPERTIES OF MONOAZO DYES DERIVED FROM SUBSTITUTED ARYLAMINES

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## ABSTRACT

Six monoazo dyes derived from arylamines substituted with bromine, chlorine and nitro groups were synthesized via diazo-coupling method and studied. Their functional groups were confirmed by an infra-red spectroscopy. Using solvents of different dielectric constants, the absorption spectra of these dyes showed positive solvatochromism with increasing solvent polarity. They also showed positive halochromism. The molar extinction coefficient of these dyes showed that they were all suitable for textile applications. More so, fastness tests revealed that all the dyes synthesized had better fastness properties on cotton fabric than on nylon 6,6.

**Keywords:** Azo Dyes, Solvatochromism, Halochromism, Fastness Properties.

## INTRODUCTION

Azo dyes are diazene-containing compounds. They are also called diamine or diimide and have the azo moiety (-N=N-) (Kilincarslan *et al.*, 2007; Valery *et al.*, 2017; Said *et al.*, 2020). Being the single largest synthetic chemical class of industrial colourants (Robinson *et al.*, 2001), they have been widely studied and used in recent years in various industrial productions because of their ease of availability, reasonable price (Gregory, 1990; Kate & Thakare, 2016) and applied utilities (Taura *et al.*, 2014); as they generally do not require numerous intermediate stages during manufacture (Choudhury, 2011).

They are highly coloured and have been used in colouring textile fabrics, photoelectronic applications, cosmetics, food, etc. (Chopde *et al.*, 2010; Yousefi *et al.*, 2012; Mijin *et al.*, 2018)

Generally, compounds with two phenol rings separated by an azo bond are known to be versatile and have received much attention in research areas (Patel, 2013).

Azo dyes are synthesized by the diazotization of a primary aromatic amine followed by the coupling of the resultant diazonium salt with an electron rich nucleophile (Sharma, 2011).

The addition of various substituents to the diazonium components which goes to act as auxochromes in the dye structures have been reported to not just influence only the colours, but also the absorption spectra of azo dyes (Soleimani & Taylor, 2008; Xu *et al.*, 2016). This work is centered on synthesizing azo dyes using various substituted aryl amines and studying their fastness

properties and absorption spectra in solvents of different dielectric constants.

## MATERIALS AND METHODS

The chemicals used for this work are NaOH, NaNO<sub>2</sub>, HCl, 1-naphthol, 2-naphthol, 2-bromoaniline, 3-chloroaniline, 3-nitroaniline, acetone, ethanol, and toluene. These were purchased from Cadifa Associates Nigeria Limited, Headbridge, Onitsha, Anambra State, Nigeria.

### Preparation of the Dyes

The procedures used for the synthesis of these azo dyes are as stated in Sharma (2014). However, 0.10 moles of the coupling and diazonium components were used instead of 0.01 moles. This was done in order to get larger quantities of the dyes.

### General Procedure for Coupling

The coupling component was prepared by adding 14.40g of 1-naphthol into a beaker containing 15.00g of NaOH dissolved in 150.00cm<sup>3</sup> of distilled water. This was placed in an ice bath and the temperature cooled to 0°C. The prepared diazonium salt was added very slowly to the coupling component using a dropper while stirring for about 15 minutes. This was left in the ice-bath and stirred for an additional 10 minutes to ensure a completed reaction. The synthesized azo dye was filtered, washed thoroughly, dried, and purified. The same procedure was repeated for 2-naphthol.

### Preparation of Dyes 1 and 2

The diazonium component was prepared by dissolving 6.90g of NaNO<sub>2</sub> in 50.00cm<sup>3</sup> of water. In another beaker, 17.20g of 2-bromoaniline was added into a beaker containing 100.00cm<sup>3</sup> of HCl and this was immediately placed in an ice bath. The temperature was cooled to 0°C. Thereafter, the NaNO<sub>2</sub> solution was added slowly to the beaker using a dropper while stirring continually for 10 minutes and maintaining the temperature at 0°C in order to generate the diazonium salt. The diazonium salt was then added to the coupling component of choice.

### Preparation of Dyes 3 and 4

The diazonium component was prepared by dissolving 12.80g of 3-chloroaniline in a beaker containing 100.00cm<sup>3</sup> of HCl solution, and this was immediately placed in an ice bath. The temperature was cooled to 0°C. In another beaker, 6.90g of NaNO<sub>2</sub> was dissolved in 50.00cm<sup>3</sup> of water and this was added slowly to the 3-

chloroaniline solution while stirring and maintaining the temperature at 0°C in order to generate the diazonium salt. The diazonium salt was then added to the coupling component of choice.

#### Preparation of Dyes 5 and 6

The diazonium component was prepared by dissolving 13.80g of 3-nitroaniline in a beaker containing 100.00cm<sup>3</sup> of HCl solution and this was placed in an ice bath. The temperature was cooled to 0°C. 50.00 cm<sup>3</sup> of water containing 6.90g of NaNO<sub>2</sub> was added slowly while stirring continually for 10 minutes and maintaining the temperature at 0°C in order to generate the diazonium salt. The prepared diazonium salt was added very slowly to the coupling component of choice.

#### Percentage Yield of the Dyes

The percentage yield of the dyes was determined using the formula:

$$\text{Percentage yield (\%)} = (\text{MP} / \text{MMP}) \div (\text{MR} / \text{MMR}) \times 100\%$$

Where MP = mass of the product

MMP = molar mass of the product

MR = mass of the reactant

MMR = molar mass of the reactant (Mohammed *et al.*, 2013).

#### Purification of the Dyes

The dyes were recrystallized by boiling in ethanol. The crystals were collected, washed severally with water, dried in the oven at 50°C and preserved for further analyses (Maradiya, 2001).

#### Ultraviolet-Visible Absorption Spectroscopy

The absorption spectra of the dyes in three solvents of different dielectric constants, namely ethanol, acetone and toluene were recorded using the ultraviolet-visible spectrometer (Jenway 6850 UV/VIS). The scan was repeated after a few drops of hydrochloric acid were added to the ethanol solvent.

#### Infra-Red Spectroscopy

Each of the dyes was presented for an infra-red scan using an infra-red spectrometer (Buck Scientific Infrared Spec M530) to obtain the functional groups present in the dyes.

#### Determination of Molar Extinction Coefficient (ε)

The molar extinction coefficient was calculated using the relation:

$$A = \epsilon CL$$

Where ε = molar extinction coefficient

A = absorbance

C = concentration of the dye in mole/litre

L = path length in cm (Joseph *et al.*, 2020).

#### Dyeing of the Fabric

Dyeing was carried out on cotton and nylon 6,6 fabrics using all the synthesized dyes separately. 2.00g of the dye was dissolved in 100.00cm<sup>3</sup> of water to produce a liquor ratio of 1:50 at room temperature. After the dye bath was set, 2.00g of the fabric was wetted and excess water squeezed out with the aid of a filter paper. The fabric was then introduced into the dye bath at a temperature of 50°C. The temperature was increased to 100°C over 15 minutes and dyeing continued at this temperature for 60 minutes. After dyeing, the fabric was removed, rinsed thoroughly with water and air dried (Salah & El-Badry, 2012; Mohammed *et al.*, 2013).

#### Wash Fastness Test

The dyed fabric was immersed in a detergent solution which was prepared by dissolving 0.50g of detergent in 30.00cm<sup>3</sup> of distilled water in a 250.00cm<sup>3</sup> beaker. The coloured fabric was stirred gently for 30 minutes. This was removed from the beaker, thoroughly rinsed with water and air dried. The changes in shades were related to the standard gray scale rating (grade 1-5) where 1 is poor and 5 is excellent (Otutu *et al.*, 2014; Efurhievwe *et al.*, 2014).

#### Light Fastness Test

The dyed fabric was firmly attached to a white cardboard paper and exposed to diffuse sunlight for 3 hours. The changes in shades were related to the standard gray scale rating (grade 1-5) where 1 is poor and 5 is excellent (William *et al.*, 2014; Joseph *et al.*, 2020).

#### RESULTS AND DISCUSSION

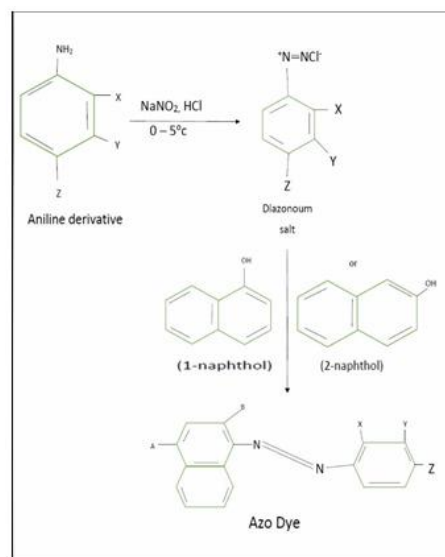
The physical properties of the dyes are tabulated in table 1, while their IUPAC names are shown on table 2. It can be seen from table 1 that the dyes made from 1-naphthol had lower melting point than those synthesized from 2-naphthol. This could be attributed to the higher melting point of 2-naphthol (O'Neil, 2001).

**Table 1:** Physical Properties of the Dyes

Dye	Texture	Melting Point (°C)	Colour	Weight (g)	Percentage Yield (%)
1	Crystal	136	Mulberry purple	12.47	72.51
2	Powdered	172	Dark brick red	13.22	76.84
3	Crystal	139	Penny brown	7.99	62.41
4	Crystal	177	Wine	8.39	65.52
5	Crystal	221	Organo brown	12.23	88.64
6	Powdered	255	Carrot orange	11.48	83.21

#### Reaction Scheme of the Dyes

The reaction of the various substituted arylamines with the coupling components is shown on scheme 1



Where Dye 1: X=Br; Y=Z=H; A=OH; B=H  
 Dye 2: X=Br; Y=Z=H; A=H; B=OH  
 Dye 3: X=Z=H; Y=Cl; A=OH; B=H  
 Dye 4: X=Z=H; Y=Cl; A=H; B=OH  
 Dye 5: X=Z=H; Y=NO<sub>2</sub>; A=OH; B=H  
 Dye 6: X=Z=H; Y=NO<sub>2</sub>; A=H; B=OH (William *et al.*, 2014)

**Scheme 1:** Reaction of Substituted Arylamines with 1-naphthol and 2-naphthol

### Absorption Spectra of the Dyes

Table 3 shows the  $\lambda_{max}$  values of the dyes in different solvents. The positions of the auxochromes; Cl, Br and NO<sub>2</sub> and hydroxyl group (OH) on the diazonium and coupling components respectively played a significant role in the  $\lambda_{max}$  of the dyes.

Comparing dyes 1 and 2, dye 2 had a higher  $\lambda_{max}$ , hence, showed a bathochromic shift when compared with dye 1. This is because the hydroxyl group is ortho and para to the azo group in dye 2 and dye 1 respectively. Thus, hydrogen bonding occurred easily between the azo group and the hydroxyl group in the ortho position. This consequently made dye 2 more polar than dye 1, thereby increasing the  $\pi \rightarrow \pi^*$  transitions in dye 2 and this caused a bathochromic shift. This comparison is also true for dye 3 and 4; and dye 5 and 6.

Comparing dye 1, 3 and 5, which have different substituents on the diazonium components, it can be seen from table 3 that dye 1 had the highest  $\lambda_{max}$  while dye 5 the least. This is due to the fact that NO<sub>2</sub> which is a stronger electron withdrawing group than Br tend to destabilize the benzene ring more, thereby increasing the energy of transition between the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) of the dye, which consequently leads to a hypsochromic shift. This comparison is also true for dyes 2, 4 and 6 (Soleimani & Taylor, 2008; Xu *et al.*, 2016).

### Effect of HCl on the $\lambda_{max}$ of the Dyes

The addition of a few drops of hydrochloric acid caused an increase in  $\lambda_{max}$  for all the dyes as seen in table 3. This can be attributed to the protonation of the  $\beta$ - nitrogen atom of the azo group which in turn caused an increase in the delocalization of the  $\pi$  - conjugated system, thereby facilitating more  $\pi \rightarrow \pi^*$  transitions. This caused a positive halochromism, hence, a bathochromic or red shift was observed (Yusuf *et al.*, 2004; Xu *et al.*, 2016).

### Effect of Solvents on the $\lambda_{max}$ of the Dyes

The absorption spectra carried out in three solvents of different dielectric constants showed that the dyes interacted with the solvents via a process called solvatochromism. Solvatochromism is the ability of a chemical substance to change colour due to a change in solvent polarity (Yusuf *et al.*, 2014). Results from table 3 showed that for each of the dyes, the  $\lambda_{max}$  value was in the order ethanol > acetone > toluene. This is because ethanol being the most polar of these solvents, had a greater polarity towards all the dyes than acetone and toluene. This higher polarity of ethanol helped to stabilize the LUMO of these dyes via a dye-solvent interaction by the dipolar effect and intermolecular hydrogen bonding forces (Mohammadi *et al.*, 2012; Hamidian *et al.*, 2013). This in turn caused a reduction in the energy of transition, resulting in more  $\pi \rightarrow \pi^*$  transitions and an increase in wavelength

(bathochromic or shift). The dielectric constants of these solvents as obtained from literature also showed that ethanol and toluene had the highest and lowest values respectively (Mehmet *et al.*, 2015; Dirgha & Nisha, 2019).

**Table 2:** IUPAC Names of the Dyes

Dye No	IUPAC Name
1	4-[(2-bromophenyl)diazenyl]naphthalen-1-ol
2	1-[(2-bromophenyl)diazenyl]naphthalen-2-ol
3	4-[(3-chlorophenyl)diazenyl]naphthalen-1-ol
4	1-[(3-chlorophenyl)diazenyl]naphthalen-2-ol
5	4-[(3-nitrophenyl)diazenyl]naphthalen-1-ol
6	1-[(3-nitrophenyl)diazenyl]naphthalen-2-ol

**Table 3:** Absorption Spectra of the Dyes in Different Solvents

Dye	$\lambda_{max}$ in Ethanol (nm)	$\lambda_{max}$ in Acetone (nm)	$\lambda_{max}$ in Toluene (nm)	$\lambda_{max}$ in Ethanol + HCl (nm)	Molar Extinction Coefficient, $\epsilon$ (Lmol <sup>-1</sup> cm <sup>-1</sup> )
1	472.00	461.00	458.00	487.00	3.64 × 10 <sup>4</sup>
2	474.00	470.00	459.00	483.00	3.25 × 10 <sup>4</sup>
3	465.00	455.00	450.00	476.00	3.87 × 10 <sup>4</sup>
4	468.00	459.00	453.00	473.00	2.44 × 10 <sup>4</sup>
5	454.00	447.00	437.00	457.00	3.27 × 10 <sup>4</sup>
6	457.00	450.00	442.00	460.00	1.24 × 10 <sup>5</sup>

### Molar Extinction Coefficient ( $\epsilon$ ) of the Dyes

The molar extinction coefficient of the dyes was calculated and tabulated in table 3. The solvent used for this determination was ethanol. The molar extinction coefficients were within the range of 2.44 × 10<sup>4</sup> - 1.24 × 10<sup>5</sup>. Technically, important dyes display molar extinction coefficient values in excess of 10<sup>4</sup> - 10<sup>5</sup> Lmol<sup>-1</sup>cm<sup>-1</sup>. Hence, the range of values obtained in table 3 is adequate for any commercial dye (Efurhievwe *et al.*, 2014; Otutu *et al.*, 2014; Agho *et al.*, 2017).

### IR Spectra of the Dyes

All the dyes synthesized showed absorptions between 3649cm<sup>-1</sup> and 3648cm<sup>-1</sup> which indicated the presence of a free O-H bond; absorptions between 3067cm<sup>-1</sup> and 3065cm<sup>-1</sup> which indicated the presence of a C-H (aromatic) bond; absorptions between 1617cm<sup>-1</sup> and 1579cm<sup>-1</sup> which indicated the presence of an aromatic ring (Otutu *et al.*, 2014; Agho *et al.*, 2017); and absorptions between 1596cm<sup>-1</sup> and 1556cm<sup>-1</sup> which showed an aromatic C=C bond present (Joseph *et al.*, 2020). More so, absorptions between 1496cm<sup>-1</sup> and 1491cm<sup>-1</sup> indicate the presence of an azo group (-N=N-) (Mohammed 2011; Lata *et al.*, 2017).

Dyes 1 and 2 showed absorptions between 661cm<sup>-1</sup> and 601cm<sup>-1</sup> indicating the presence of C-Br bond and dyes 3 and 4 showed absorptions between 793cm<sup>-1</sup> and 730cm<sup>-1</sup> indicating the presence of C-Cl bond. Dye 5 and 6 showed absorptions between 1597cm<sup>-1</sup> and 1326cm<sup>-1</sup> indicating the presence of a nitro group (NO<sub>2</sub>) (Pavia *et al.*, 2001).

### Wash Fastness Properties

It can be seen from table 4 that the dyes had a moderate to good wash fastness in cotton fabric, and a poor to good wash fastness in nylon 6,6. The lesser fastness properties of nylon 6,6 fabric can be attributed to the more amorphous nature of the fabric. The dyes easily diffuse into nylon 6,6 during the dyeing process thereby giving a better colour shade than in cotton. However, this easy diffusion of the dye molecules into the fabric becomes a weakness as during the washing test, these dyes easily diffuse out of the fabric. In cotton, the structure is highly crystalline hence, dye molecules find it hard to diffuse into the fabric and once in, these dye molecules are not easily removed because of the stronger dye-fibre bond being formed (Gundogen & Eren, 2014; Otutu *et al.*, 2015; Aamer & Ghulam, 2018).

### Light Fastness Properties

Results from table 4 showed that of all the dyes, dyes 2 and 6 had the best light fastness in both fabrics. These dyes are said to be more photo stable than the other dyes. This could be attributed to the bromo and nitro substituents on the benzene ring in dyes 2 and 6 respectively (Otutu *et al.*, 2014; Efurhievwe *et al.*, 2014; Aamer & Ghulam, 2018).

**Table 4:** Fastness Properties of Synthesized Dyes

Dye no	Cotton		Nylon 6,6	
	Wash Fastness	Light Fastness	Wash Fastness	Wash Fastness
1	3-4	3-4	3	4
2	2-3	4-5	2	4
3	3	2	2-3	2
4	3	3	1-2	1-2
5	3	2-3	3	3
6	4-5	4	4	4

### Conclusion

This work has shown that the nature and position of electron withdrawing substituents (Cl, Br, NO<sub>2</sub>) have a remarkable influence on the colour of the dyes as well as their  $\lambda_{max}$ . All the dyes synthesized showed positive halochromism which is a characteristic of azo dyes. They also exhibited positive solvatochromism with increasing solvent polarity. This work further revealed that the dielectric constants of ethanol, acetone and toluene are in the order ethanol > acetone > toluene as dielectric constant varies directly with solvent polarity. Fastness evaluation of these dyes revealed that cotton fabric had better fastness properties than nylon 6,6 fabric for the parameters tested. The value of the molar extinction coefficients for all the dyes synthesized are within the range for textile application. However, dye 6 had a very high molar extinction coefficient than the other dyes. Hence further studies are encouraged to understand the reason for this sharp increase and its implication on textile and non-textile applications.

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