

# Synthesis and characterization of monoazo disperse dyes from salicylic acid and phenol as coupling components

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## ABSTRACT

Synthesis of four varieties of monoazo disperse dyes derived from arylamine was studied. The dyes were synthesized by diazotization of aryl amines and then coupled to salicylic acid and phenol as coupling components. The impure dyes obtained were purified using Thin Layer Chromatography (TLC) to give dyes (Figure 1) with good colouristic properties. The purity and identities of all the dyes were confirmed by melting point, IR and UV spectroscopy. The UV spectra of all the dyes were carried out in solvents of different dielectric constants (methanol, ethanol and ethyl acetate). The results showed solvatochromic effect. Dyeing and fastness properties of all the dyes synthesized were carried out on nylon, cotton and polyester fabrics. Deep dyeing was observed on nylon fabric because of the amorphous structure of the fabric as compared with cotton and polyester fabrics which have crystalline structures.

**Keyword:** synthesis, diazotization, coupling, spectral analysis, Solvatochromism and fastness properties.

## 1. INTRODUCTION

Organic colors known as disperse dyes are used frequently on hydrophobic fibres like polyester and nylon because they are less water soluble (Al-Etaibi & El- Apasery, 2020). This is due to their remarkable colour fastness and the ease with which a variety of colour tones can be obtained (Obi et al., 2022; Karim et al., 2021). They are frequently substituted azo, anthraquinone, or diphenylamine compounds that are not ionic and do not have a group that makes them water soluble, but which have been claimed to have potential research use in the creation of good light- resistant dyes (Xiyu et al., 2020).

The discovery of disperse dyes is credited to James Badiley and Holland Ellis in 1924. But because of their low substantivity for natural fibres, secondary cellulose acetate and polyester were produced in the 1950s, which changed their chemistry (Abdullahi, 2019). Disperse dyes have been the swift replacement for traditional natural dyes in the dyeing business due to research showing that they are less expensive than most synthetic dyes (Shukla & Mohan, 2017). Additionally, specialized disperse dyes that have impressive biological efficacy against some Gram- positive and Gram- negative pathogenic bacteria as well as

fungi have been created recently in addition to being used primarily for dyeing (Hatem et al., 2016; Shaki et al., 2017; Alnassar et al., 2019).

Polyester fibre is the most widely produced of all artificial fibres and has a very compact and crystalline structure (Mashaly et al., 2014; Kaminska et al., 2019; Farah et al., 2015). Disperse dyes are typically regarded as the best options for colouring polyesters because of their good to exceptional light and washing fastness capabilities (Chaitannya et al., 2020). Nylon is synthetic like polyester and can be coloured using disperse dyes. Due to its amorphous structure, however, its dyeing procedure is less energy-intensive than that of polyester because dyeing is done at the boil (Obi & Onuh, 2022; Pawar et al., 2018). Reactive dyes are easily used to colour cotton. Azo-anthraquinone dyes can, nevertheless, be employed, according to recent investigations. This is due to their alluring advantages, which include exceptional light stability and tinctorial strength.

Diazotization and coupling reactions are both important for the synthesis of azo dyes. Diazotization is simply the process of converting a primary amine to a diazonium salt. Diazotization is carried out by treating the aryl amine with nitrous acid, which is normally generated *in situ* with HCl and NaNO<sub>2</sub>. The nitrous acid produces the nitrosonium ions which then reacts with the aryl amine. Overall, this mechanism involves the removal of the two hydrogen atoms from the aniline derivatives and the formation of a triple bond between the two nitrogen atoms. The hydrogen atoms earlier removed are bonded to oxygen to form water and booted off as a leaving group.

Diazotization is only possible for primary alkyl or aryl amines. For secondary aryl or alkyl amines, an N- hydroxyazo compound cannot be formed because there is no other hydrogen to be removed.

Coupling reaction may be completed immediately after the components are mixed or after a long time (Sharma, 2011).

The goal of this research is to synthesize monoazo disperse dyes derived from various alkyl amines (as diazonium components) and, salicylic acid and phenol (as coupling components). More so, to analyze the spectra and fastness characteristics of these dyes on cotton, nylon, and polyester materials.

## 2. MATERIALS AND METHODS

### Reagents

They include N, N-dimethylaniline, N, N-diethylaniline, phenol, salicylic acid, NaOH, HCl, Dimethylformamide (DMF).

### Preparation of the Dyes

The general techniques (Methods 1 and 2) were followed to prepare the monoazo disperse dyes by diazotizing each of the several diazonium components and coupling each one with the designated coupling components (phenol and salicylic acid). The uncorrected melting point was determined using an Electrothermal 9100 equipment. Ultraviolet- Visible Spectra (UV-VIS) were measured using a Jenway 6850 UV/VIS device, and infra- red (IR) spectra were acquired in the solid state using a Buck scientific infra- red spec M530 apparatus.

### Method 1 General Procedure for Diazotization

In a beaker containing 10 cm<sup>3</sup> of concentrated HCl solution, 1.27 ml of N, N-dimethylaniline was weighed and added to it. The beaker was then immediately placed in an ice bath to keep the temperature below 5 °C. 0.69 g of NaNO<sub>2</sub> was dissolved in 5 cm<sup>3</sup> of water and added to the acid solution gently while stirring for 10 minutes. The resulting solution was left in an ice bath to retain its temperature at or below 5 °C. This produced the diazonium salt solution. For N, N-diethylaniline, this process was repeated using 1.6 ml (Salah & El- Badry, 2012; Sharma, 2011).

### Method 2 General Procedure for Coupling

A measured 0.94 g of phenol was dissolved in a beaker containing 5 cm<sup>3</sup> of 2.50 M NaOH. The temperature of the solution was kept at or below 5 °C by using an ice- bath. This produced the coupling component solution. By using a dropper and stirring for about 30 minutes, the diazonium salt solution was gradually added to the coupling component solution. To make sure the reaction was finished, this was left in an ice bath for an extra 20 minutes. The synthesized dyes were filtered, completely washed, and recrystallized from ethanol. They were dried in an oven (42 °C). To produce the coupling compound of the salicylic acid, the same process was repeated by using 1.38 g of salicylic acid (Salah & El- Badry, 2012; Sharma, 2011).

### **Dyeing of Cotton and Nylon Fabrics**

A 2% dye stock solution was created by dissolving a weighted quantity of 2.0 g of the dye in 100 cm<sup>3</sup> of water at room temperature. Through out the dyeing process, a material to liquid ratio of 1:50 was maintained. The cloth sample was moistened, and extra water was removed with the use of filter paper. The sample was thereafter added to the dye bath at a temperature of 50 °C. Later, the temperature was raised to 100 °C for 20 minutes, after which the dyeing process continued for 60 minutes at that temperature. Following dyeing, the fabric was removed, properly washed with water, allowed to air dry, and then presented for fastness tests (Obi et al., 2022; Emmmanuel et al., 2022).

### **Dyeing of Polyester Fabric**

High Temperature High Pressure (HTHP) method of dyeing was used. 1 g of the dye was dissolved in 100 g of DMF solvent to create a 1% stock solution. A 1:50 material to liquor ratio was kept through out the dyeing process. After being dried and wetted out, the fabric was placed in to the dye bath at 50 °C. A pH of 4.5– 5.5 was maintained by applying glacial acetic acid as the temperature was raised to 130 °C and dyeing continued for another 45 minutes. The dyed sample was then cleaned using a solution of 2 gpl NaOH and 2 gpl sodium hydrosulphite for 20 minutes at 70 °C. The coloured sample was then rinsed in cold water, dried by air, and presented for fastness tests (Obi & Onuh, 2022).

### **Wash Fastness Test**

A detergent solution made up of 0.50 g of detergent in 30 cm<sup>3</sup> of distilled water was used to submerge the coloured sample. After 30 minutes of gentle stirring, the sample was withdrawn and carefully water- rinsed. Thereafter, the sample was air dried, and the variations in hues were compared with the control sample using a gray scale (1–5), where 1 represents poor and 5 represents excellent (Odilora & Omatseye, 2000).

### **Light Fastness Test**

The sample that had been dyed was firmly fastened to a sheet of white cardboard and left in the sun for three hours. The variations in hues were compared with the control sample using a gray scale (1–5), where 1 represents poor and 5 represents excellent (Odilora & Omatseye, 2000; Ukponmwan et al., 1999; Heid et al., 2003).

### **Heat Fastness Test**

A pressing iron set at 60 °C was used to iron the dye sample for 30 seconds on a white background. To check for colour changes, the control sample and the ironed sample were compared. The variations in colour tones were checked using a gray scale (1–5), where 5 is excellent and 1 is bad (Odilora & Omatseye, 2000; Ukponmwan et al., 1999).

### **Percentage Yield of the Dyes**

This was calculated by using the mole ratio formula. The formula used is as follows;

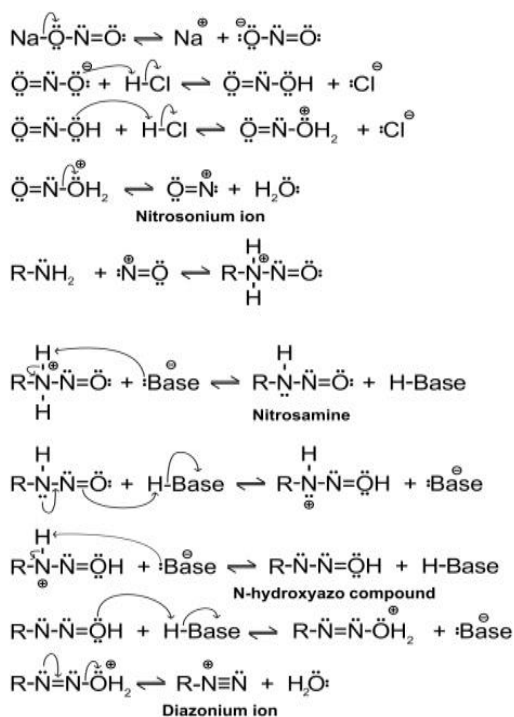
$$\text{Percentage Yield (\%)} = \text{MP} / \text{MR} \times 100\%$$

Where MP= mole of product(s)

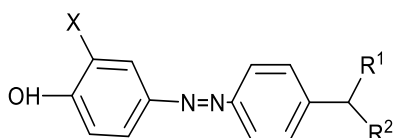
MR= mole of reactant(s) (Mohammed et al., 2013).

## 3. RESULTS AND DISCUSSION

The dyes were synthesized through the diazo- coupling method. A possible mechanism for the diazotization of the aryl amines used for this synthesis is shown in scheme 1, while the general structure of the synthesized disperse dyes is shown in figure 1.



**Scheme 1** Mechanism of Diazotization of the Aryl Amines



**Figure 1** General Structure of the Synthesized Dyes

Where

Dye 1: X= H; R<sup>1</sup> = R<sup>2</sup>= CH<sub>3</sub>

Dye 2: X= COOH; R<sup>1</sup> = R<sup>2</sup>= CH<sub>3</sub>

Dye 3: X= H; R<sup>1</sup> = R<sup>2</sup>= C<sub>2</sub>H<sub>5</sub>

Dye 4: X= H; R<sup>1</sup> = R<sup>2</sup>= C<sub>2</sub>H<sub>5</sub>

The dyes were recrystallized in ethanol solvent. The percentage yield of the dyes were calculated using the mole ratio formula as stated in Mohammed et al., (2013). Phenol coupled at the para position. Table 1 shows some physical characteristics of the dyes. Dyes 1 and 2 gave good yields, while dyes 3 and 4 gave low yields. However, no cause was ascertained for the low yields of dyes 3 and 4.

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

Dye No.	Colour	Texture	Melting Point (°C)	Weight (g)	Percentage Yield (%)
1	Grey	Jelly	211	2.6	54.15

2	Dark grey	Jelly	256	3.7	65.15
3	Coffee	Jelly	321	2.0	37.30
4	Limber	Powdered	369	1.7	27.20

The functional groups present in the dyes were seen via the infra-red characterization of the dyes as shown on table 2. The IR scan showed the presence of vibrational O-H, C-H, Ar-H, >C=C (aromatic), -N=N- bonds (Joseph et al., 2020).

**Table 2** Functional Groups Present in the Synthesized Dyes

IR PEAKS (cm <sup>-1</sup> )					
V <sub>O-H</sub>	V <sub>C-H</sub>	V <sub>Ar-H</sub>	V <sub>C=C</sub>	V <sub>N=N</sub>	V <sub>COOH</sub>
3649-3648	3067-3065	1617-1579	1596-1556	1496-1491	1725-1699

The maximum absorption wavelength ( $\lambda_{\max}$ ) of all the dyes synthesized when conducted in solvents of different dielectric constants (methanol, ethanol, and ethyl acetate) are shown on Table 3. The  $\lambda_{\max}$  was affected by these solvents. The wavelength values of the dyes in methanol were the longest; while those in ethyl acetate were the shortest. This could be due to methanol's stronger polarity when compared with the other solvents (ethanol and ethyl acetate). In turn, this high polarity of methanol leads to the stabilization of the dye's Lowest Unoccupied Molecular Orbital (LUMO), which lowers the transition energy and encourages more transitions. This implies the occurrence of a solvatochromic effect. Infact, a positive solvatochromism is observed to have occurred as shown on Table 3.

Furthermore, the addition of electron donating groups; methyl (CH<sub>3</sub>) and ethyl (C<sub>2</sub>H<sub>5</sub>) as auxochromes to the benzene ring had a noticeable impact on the wavelengths of the dyes. Comparing dyes 1 and 3, it was observed that dye 3 had a higher wavelength than dye 1. This could be because in dye 3, the electron donating group is an ethyl (C<sub>2</sub>H<sub>5</sub>) group as opposed to a methyl group in dye 1. Generally, alkyl groups are known to be electron donating groups. An ethyl group will have a more electron donating inductive effect than a methyl group. This electron donating effect will activate the benzene ring thereby increasing the wavelength of the dye. Thus, dye 3 experienced a bathochromic shift or effect with regards to dye 1. This is the same for dyes 2 and 4.

When dyes 1 and 2 are compared, the wavelength ( $\lambda_{\max}$ ) value of dye 2 was observed to be lower than dye 1 as shown on Table 3. This may be due to the additional electron withdrawing group (-COOH) in dye 2 structure. This -COOH group deactivates the benzene ring and causes a decrease in the dye wavelength. Thus, dye 2 records a hypsochromic shift with regards to dye 1. The same holds true for dye 3 and 4.

**Table 3** Maximum Absorption Wavelengths of the Synthesized Dyes

Dye No.	$\lambda_{\max}$ in Methanol (nm)	$\lambda_{\max}$ in Ethanol (nm)	$\lambda_{\max}$ in Ethylacetate (nm)	Molar Extinction Coefficient (dm <sup>3</sup> mol <sup>-1</sup> cm <sup>-1</sup> )
1	447	443	439	2.44 × 10 <sup>4</sup>
2	444	437	432	3.25 × 10 <sup>4</sup>
3	465	461	453	3.64 × 10 <sup>4</sup>
4	432	426	421	3.87 × 10 <sup>4</sup>

The molar extinction coefficient of the dyes is shown on Table 3. Molar extinction coefficient of a dye tells how intense the absorption of the dye would be. It can be seen from Table 3 that the molar extinction coefficient values of all the synthesized dyes range from 2.44 × 10<sup>4</sup> to 3.87 × 10<sup>4</sup>. This range is acceptable for any commercial dye. This implies that all the dyes synthesized can be harnessed for large scale industrial dyeings. More so, dyes with high molar extinction coefficient value will require little quantity for its application during the dyeing of any fabric (Feng et al., 2016; Yu-Wen et al., 2022). This implies that dyes 2, 3 and 4 will require a little quantity of it to be used for dyeing than dye 1, under the same dyeing conditions.

Polyester, nylon and cotton materials were all dyed using the synthesized dyes, and the colour ratings of the fastness properties of the dyes on these fabrics is tabulated on Table 4. The deepest colour hue was discovered to be that of the nylon fabric. This might be due to nylon's greater amorphousness when compared with the other materials (polyester and cotton). This amorphous nature of nylon permitted more dye molecules to be absorbed into the structure of nylon during the dyeing process than was observed in

the case of polyester and cotton. Hence, nylon fabric had the deepest colour depth than cotton and polyester fabrics (Obi et al., 2022; Obi & Onuh, 2022).

**Table 4** Fastness Ratings Comparison of the Synthesized Dyes on Cotton, Nylon and Polyester Fabrics.

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

Key: 1= extremely poor; 1-2= very poor; 2= poor; 2-3= fair; 3= moderate; 3-4= moderately good; 4= good; 4-5= very good; 5= excellent.

According to table 4, the results of the fastness tests for all of the fabrics range from fair to very good. Light fastness is the resistance to the fading of the colour of a dyed fabric by the action of light. Likewise, wash fastness and heat fastness are the resistance to the fading of the colour of a dyed fabric by the action of washing and heat respectively. Many factors are responsible for light, heat and wash fastness. These include the size of the dye, the nature of the detergent used (in the case of wash fastness), the amount of moisture trapped in the structure of the fabric, photo-oxidation and photo-reduction, temperature, etc. However, one important determining factor that is prevalent in wash, light and heat fastness is the nature of the fabric being dyed. The crystallinity or amorphous nature of any fabric affects the level of dye up-take (exhaustion) and fastness ratings. As seen from Table 4, the fastness ratings of polyester fabric was found to be greater than that recorded for cotton, which was greater than that recorded for nylon. This could be because polyester fabric is highly crystalline and so its structures are well layered. This well layered structure greatly prevents the inward migration of the dyes during the dyeing period. However, once these dyes successfully migrate into the inner structure of the polyester fabric, they find it very difficult in migrating out. This could have caused the polyester fabric to have a better fastness rating than cotton and nylon.

Nylon is the most amorphous of all the fabrics dyed, hence, the dyes can easily migrate in through the pores during the dyeing process and migrate out during the fastness tests.

In addition, it can be seen from Table 4 that the wash fastness ratings of any of the fabric is low when compared with the heat fastness and light fastness for that same fabric. This could be because the detergent used in the case of wash fastness might have played an active role in weakening the bond or force of attraction between the dyes and the fabric.

## 4. CONCLUSION

This research has demonstrated that the maximum wavelength of monoazo dispersion dyes is significantly influenced by electron withdrawing and electron donating groups. On the various textile materials used, all of the synthetic dyes had good fastness characteristics. This implies that a mordant is not necessary when using these dyes in textile applications. This is a positive development considering the cost which the use of mordant will add to the dyeing process in large scale dyeings.

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### Author's Contributions

Juliana Chineze Obi: Conceptualization, designing and editing of the manuscript.

Ikechi Ikeagwuchi Dandy: Experimentation of this work and writing of the manuscript.

### Ethical approval

Not applicable.

**Informed consent**

Not applicable.

**Conflicts of interests**

The authors declare that there are no conflicts of interests.

**Data and materials availability**

All data associated with this study are present in the paper.

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