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Synthesis and fastness properties of disperse dyes gotten from some aniline derivatives

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ABSTRACT

Selected monoazo disperse dyes were synthesized via the diazotization and coupling method and were characterized by their melting point, Infra- red (IR) and Ultra- violet (UV) spectroscopy. The dyes exhibited solvatochromic effects in solvents of different dielectric constants (methanol, ethanol and ethyl acetate). Results showed that dyes with more electron withdrawing groups had lower wavelengths when compared with their counterparts. The dyes were applied on nylon, cotton and polyester fabrics and tested for light, wash and heat fastness properties. All the synthesized dyes had a deep dyeing on nylon fabric than the other fabrics. However, the fastness properties on nylon were found to be the least and polyester fabric had the best.

Keyword: Disperse dyes; dyeing; light fastness; wash fastness; heat fastness; diazotization; coupling; solvatochromism.

1. INTRODUCTION

Disperse dyes are organic colours which exhibit extremely low water solubility and vary in the kind of chromophore present. These include azo, anthraquinone, nitro, methine, benzodifuranone and quinoline based structures. They are widely applied to hydrophobic fibres such as polyester and nylon (Alya & Morsy, 2020). This is because of their impressive colour fastness and the possibilities of obtaining a wide range of colour shades (Rezaul et al., 2021). They are often substituted azo, anthraquinone or diphenylamine compounds which are not ionic and contain no water solubilizing group in them but have been reported to have potential research value in the development of good light resistant dyes (Xiyu et al., 2020).

The discovery of disperse dyes is recorded to have been made in 1924 by James Badiley and Holland Ellis. However, due to their poor substantivity for natural fibres, their chemistry developed in the 1950s, when secondary cellulose acetate and polyester came into existence (Hadiza, 2019). Various studies on disperse dyes have shown that they are of lesser cost than most synthetic dyes and this offered a quick replacement of traditional natural dyes in the dyeing industry (Markandeya & Mohan, 2017). Also, in addition to be used primarily for dyeing, specialized disperse dyes which have displayed impressive biological

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efficiency against some Gram-positive and Gram-negative pathogenic bacteria as well as fungi have been synthesized in recent times (Gaffer et al., 2016; Hanieh et al., 2017; Alnassar et al., 2019).

One of the most popular classes of polycondensation polymers with an ester functional group on the main chain are polyesters. Polyester fibres have a highly compact and crystalline structure and are the most produced among all man-made fibres (Camlibel, 2018; Daria et al., 2019). They are made up of at least 85% by weight of an ester and a dihydric alcohol and a terephthalic acid, and are typically identified as polyethylene terephthalate (PET) (Jeyakodi et al., 2021). Polyester is utilized in a variety of applications, including textile, automotive, medical, electrical, and construction, as well as in the form of fibres, filament, fabric, composites etc. (Camlibel, 2018).

In view of production and uses, PET is the most important synthetic fiber used in the world (Gong et al., 2018). Generally, disperse dyes are noted as the most suitable for dyeing polyester, as they have shown good to excellent light and washing fastness properties (Chaitannya et al., 2020). Just like polyester, nylon is synthetic and can be dyed using disperse dyes (Rashedul & Fatema, 2021). However, its dyeing process is not an energy-intensive one like that of polyester, as dyeing is carried out at the boil and this can be attributed to its amorphous structure (Obi & Onuh, 2022; Ashitosh et al., 2018). On the other hand, cotton is readily dyed with reactive dyes, direct dyes or vat dyes. However, recent studies have shown that azo-anthraquinine dyes can also be used in dyeing cotton, and this is because of their attractive advantages which include tinctorial strength and excellent light stability. More so, disperse dyes have also been reported to be able to dye cotton fabrics (Obi et al., 2022).

Objectives of this Study

This work sets out to synthesize monoazo disperse dyes by using aniline derivatives as diazonium component and salicylic acid and phenol as coupling components; The effects of the aniline derivatives and coupling components on the spectra properties of the synthesized dyes are also studied; Generally, cotton fabrics are known to be dyed with direct, reactive or vat dyes. This study sets to examine and compare the fastness properties of the synthesized disperse dyes on cotton fabric as well as on nylon and polyester fabrics.

2. MATERIALS AND METHODS

Reagents

All the chemicals used for this work were obtained from commercial sources. They include 3-nitroaniline, 3-bromoaniline, sulphanilic acid, phenol, 1-naphthol, salicylic acid, NaOH, HCl, dimethylformamide (DMF), $NaNO_2$.

Preparation of the Dyes

The monoazo disperse dyes were prepared by the diazotization of all the different diazonium component and coupling them individually with the assigned coupling components (phenol, 1-naphthol and salicylic acid) as outlined in the general procedures (Obi et al., 2022). Melting point was measured using electrothermal 9100 instrument and are uncorrected. Infra-red (IR) spectra were recorded in the solid state using a Buck scientific infrared spec M530 instrument and the Ultraviolet-Visible Spectra (UV-VIS) was measured using a Jenway 6850 UV/VIS machine.

General Procedure for Diazotization

A weighed amount of 1.72 g of 2-bromoaniline was added into a beaker containing 10 cm^3 of concentrated HCl solution and this was immediately placed in an ice bath to maintain the temperature below 5^oC. 0.69 g of $NaNO_2$ was dissolved in 5 cm^3 of water and the solution was added slowly to the acid mixture by using a dropper while stirring continually for about 10 minutes to give the diazonium salt. The temperature of the solution was maintained at below 5^oC by keeping it in an ice bath. This procedure was repeated for 4-sulphanilic acid by using 1.73 g.

General Procedure for Coupling

A weighed amount of 0.94 g of phenol was dissolved in a beaker containing 5 cm^3 of 2.50 M NaOH. The solution was maintained below 5^oC in an ice bath. The diazonium salt solution was added slowly to the coupling component 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 is completed. The dyes were filtered, recrystallized from ethanol, washed thoroughly and dried in an oven at low temperature (42^oC). This procedure was repeated by using 1.38 g of salicylic acid.

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Dyeing of the Fabric

Dyeing of Cotton and Nylon Fabrics

A weighed mass of 2.0 g of the dye was dissolved in 100 cm^3 of water at room temperature to give a 2% dye stock solution. Material to liquor ratio of 1:50 was maintained all through the dyeing process. The fabric sample was wetted and excess water squeezed out with the aid of a filter paper. Thereafter, the sample was introduced into the dye bath at a temperature of 50°C. The temperature was later increased to 100°C for 20 minutes and dyeing continued at this temperature for 60 minutes. After dyeing, the fabric was removed, rinsed thoroughly with water, air dried and presented for fastness tests (Obi et al., 2022; Emmmanuel et al., 2022).

Dyeing of Polyester Fabric

Dyeing was carried out by using the high temperature high pressure (HTHP) method. 1% stock solution was prepared by dissolving 1g of the dye in 100 g of DMF solvent. Material to liquor ratio of 1:50 was maintained all through the dyeing process. The fabric was immersed into the dye bath at 50°C after being wetted out and dried. The temperature was increased to 130°C and dyeing continued at this temperature for 100 minutes while a pH of 4.5-5.5 was maintained by using glacial acetic acid. Thereafter, the dyed sample was subjected to cleaning treatment at 70°C for about 20 minutes by using 2 gpl NaOH and 2 gpl sodium hydrosulphite together. After which, the dyed sample was rinsed in cold water, air dried and presented for fastness tests (Ashitosh et al., 2018).

Wash Fastness Test

The dyed sample was immersed in a detergent solution containing 0.50 g of detergent in 30 cm^3 of distilled water. The sample was stirred gently for 30 minutes and then removed and rinsed thoroughly with water. Thereafter, the sample was air dried and the changes in shades were related to the standard gray scale rating (grade 1- 5) where 1 is poor and 5 is excellent (Obi et al., 2022)

Light Fastness Test

The dyed sample was firmly attached to a white cardboard paper and exposed to 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 (Emmmanuel et al., 2022).

Heat Fastness Test

The dyes sample was placed on a white background and ironed for about 30 seconds using a pressing iron set at about 60 °C. The ironed sample was compared with the control sample to see if any colour change had occurred. The changes in colour shades were related to the standard gray scale rating (grade 1-5) where 1 is poor and 5 is excellent (Obi et al., 2022; Emmmanuel et al., 2022).

3. RESULTS AND DISCUSSION

The general structure of the synthesized disperse dyes is shown below;

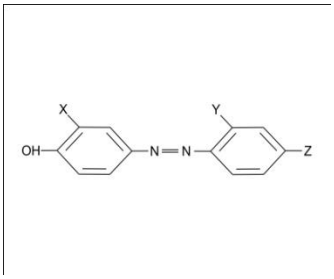

Figure 1 General Structure of the Synthesized Dyes

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Dye 1: X= Z= H; Y= Br

Dye 2: X= COOH; Y= Br; Z= H

Dye 3: X= Y= H; Z= SO_3H

Dye 4: X= COOH; Y= H; Z= SO_3H

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The impure disperse dyes were recrystallized from ethanol and their purity was monitored by using Thin Layer Chromatography (TLC). All the dyes were obtained in good yields except dye 1 which had a percentage yield of 42 %. The reason for its low yield as compared with the other synthesized dyes was undetermined. Table 1 shows the physical characteristics of the synthesized monoazo disperse dyes.

Table 1 Physical Characteristic of the Synthesized Dyes

Dye No.	Colour	Texture	Melting Point (°C)	Weight (g)	Percentage Yield (%)
1	Lamp black	Jelly	129	2.3	42
2	Rich gold	Powdered	168	4.5	70
3	Cadmiun yellow	Jelly	143	3.1	56
4	Lead tin yellow	Powdered	135	5.4	84

The structure of the monoazo disperse dyes synthesized were verified by infra- red (IR) spectroscopy and the spectra confirmed the presence of various functional groups as shown in table 2 (Obi et al., 2022; Emmmanuel et al., 2022; Joseph et al., 2020).

Table 2 IR Spectra of the Synthesized Dyes

Functional group	Wave number (cm^{-1})
V_{O-H}	3650-3640
V_{C-H}	3070-3060
V_{Ar-H}	1617-1580
$V_{C=C}$	1600-1560
$V_{N=N}$	1500-1400
V_{C-N}	1400-1370
V_{COOH}	1725- 1700
V_{C-Br}	660-600
V_{SO3H}	1365- 1340

The maximum absorption wavelengths (λ_{max}) of the dyes as carried out in methanol, ethanol and ethyl acetate solvents are shown on Table 3. These solvents had an effect on the $\lambda_{max.}$ It was observed that all the dyes in methanol solvent had the highest λ_{max} value while ethyl acetate solvent had the least value. This is as a result of the higher polarity of the methanol solvent as compared with the other solvents. This higher polarity of the methanol solvent in turn stabilizes the lowest unoccupied molecular orbital (LUMO) of the dyes thereby reducing the transition energy and promoting more $\pi \rightarrow \pi^*$ transitions. The same effect is recorded for the ethanol and ethyl acetate solvents, but, in a smaller extent. This infers that a positive solvatochromic effect occurred in all the solvents used. Solvatochromism is the tendency of a chemical compound to change colour due to a change in the polarity of the solvent. Also, the introduction of electron withdrawing groups as auxochromes (-SO_3H, -COOH, -OH and Br) to the benzene ring caused a marked change on the λ_{max}. More electron withdrawing group have a greater inductive effect on the benzene ring that a lesser electron withdrawing group. Thus, -SO_3H, being the most electron withdrawing group in the dyes, had the highest inductive effect on the benzene ring than others. This higher inductive effect leads to the deactivating of the benzene ring, which consequently results in a hypsochromic shift as compared with the other electron withdrawing groups. This is seen in the reduced wavelength of dye 4 as compared with the other synthesized dyes.

A comparison of dyes 1 and 2 showed that both contain an hydroxyl group (-OH). In addition to this, dye 2 also have a carboxylic acid group (-COOH). The λ_{max} value of dye 2 was found to be lower than that of dye 1. This could be because of the presence of the extra electron withdrawing group (-COOH) in addition to the hydroxyl group (-OH) in the structure of dye 2. This extra electron withdrawing group leads to a further deactivation of the benzene ring thereby causing a further reduction in the wavelength of the dye. This is the same for dyes 3 and 4. Comparing dyes 2 and 4, the λ_{max} values showed that dye 2 had a higher value than dye 4. Again, this could be because of the presence of the -SO_3H group in dye 4 which deactivates the ring more than the -Br group in dye 2. The -Br group is in an ortho position to the azo group (in dye 2), while the -SO_3H group is in a para position to the azo group (in dye 4). It is expected that an electron withdrawing group in an ortho position to an azo group will exert more

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inductive effect on the benzene ring than an election withdrawing group in a para position (Obi et al., 2022; Emmmanuel et al., 2022). However, the reverse is the case here. -SO_3H which is in a para position (in dye 4) have a more electron withdrawing inductive effect than the -Br group (in dye 2) which is in an ortho position. This could be attributed to the very high electron withdrawing effect of -SO_3H when compared with -Br. This explains the higher λ_{max} value of dye 2 when compared with dye 4.

Table 3 UV Spectra of the Synthesized Dyes in Solvents of Different Dielectric Constant.

Dye No.	λ_{max} in Methanol (nm)	λ_{max} in Ethanol (nm)	λ_{max} in Ethyl acetate (nm)
1	450	443	438
2	420	416	411
3	423	419	413
4	417	409	401

The synthesized dyes were used to dye cotton, nylon and polyester fabrics. The interaction between the dyes and the fabrics determined their colour shade and consequently their fastness properties. Cotton and nylon were dyed using the same solvent and under the same dyeing conditions. However, it was noticed that the colour strength of the cotton was not as solid as that of nylon. Nylon absorbed more dye molecules than cotton in simultaneous dyeing and this may be due to the crystalline nature of cotton which makes dye diffusion more difficult. Nylon is amorphous and therefore very easy for dye molecules to diffuse (Obi et al., 2022).

The polyester fabric was dyed using DMF solvent and the colour shade obtained was deeper than was obtained for cotton. Although polyester fabric is more crystalline than cotton, polyester absorbed more dye molecules than cotton because of the extended dyeing time and higher temperature. This led to an increase in the absorption of the dye molecules by the polyester fabric regardless of its higher crystallinity.

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

Dye no	Nylon			Cotton			Polyester		
	LFT	WFT	HFT	LFT	WFT	HFT	LFT	WFT	HFT
1	4	4	4	4	4	4	4	4-5	4
2	4	3	3-4	4-5	4	4	4-5	4-5	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: LFT= Light fastness test; WFT= Wash fastness test; HFT= Heat fastness test; 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.

Colour fastness is the ability of a coloured fabric to resist colour degradation due to various influences such as heat, light, etc. The variation in the shade of the fabric results from both the nature and position of the substituent's present on the diazotized compound (Ozougwu & Ayakoha, 2017). Generally, the fastness tests on all the fabric range from fair to very good. Polyester was found to have better fastness properties than cotton. Cotton on the other hand, had better fastness ratings that nylon for each of the dyes used. The wash, light and heat fastness properties of the dyes are tabulated on table 4. Light fastness is the extent to which a dye resists fading due to light exposure. Light fastness is governed by factors such as wavelength of the incident radiation; dye-fibre system; effective humidity; dye induced catalytic action; temperature etc. Most importantly, the compactness of dye and fibre structures is a key determinant of light fastness. A more compact fibre structure obstructs pores and does not allow the passage of oxygen or moisture inside the fabric, thereby suppressing fading (Aamer & Ghulam, 2018).

Cotton is more crystalline in structure than nylon. Thus, cotton is more compacted and will not readily allow the passage of light, moisture or oxygen into it. This could have reduced the rate of colour fading of the cotton fabric than it was observed on the nylon fabric. Hence, cotton had better light fastness properties than nylon for all the dyes synthesized. This same explanation applies to polyester. Polyester fabric is found to have the best light fastness property and this can be attributed to his highly compact structure than cotton or nylon fabric. Wash fastness is the resistance offered by coloured fabric to retain colour after being with soaps and/ or detergents. Various factors such as the size of the dye, solubility of the dye, detergent formulation, etc. influence the wash fastness of a dye. However, the crystallinity of the fabric plays a key role in wash fastness (Aamer & Ghulam, 2018). As

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stated earlier, nylon is more amorphous than cotton and polyester. Hence, dye molecules will migrate easily into it during dyeing and consequently, migrate easily out of it during washing. This results in a lesser wash fastness than is observed for cotton and polyester fabrics.

Heat fastness is the extent to which a dye resists fading due to exposure to high temperature. Factors which influence heat fastness include polarity of the dye, intermolecular binding force between the dye and fibre, etc. However, a significant factor is the nature of the fabric. As earlier stated, nylon fabric is the least crystalline. Thus, there is a faster diffusion of the dyes into the fabric than is observed in both cotton and polyester fabrics in simultaneous dyeing. This consequently affects the ease at which the dyes fade when exposed to high temperature. Amorphous fabrics have more pores than crystalline fabrics whose structures are well layered. This well-layered structure limits the transfer of heat into the fabric, hereby reducing the amount of heat that gets to the dye and thus reducing the dye fading rate as compared with the amorphous fabric. This explains why the cotton fabric had higher heat fastness values than is observed with nylon fabric.

4. CONCLUSIONS

These studies have shown that electron with drawing groups have a marked effect on the maximum wave length of monoazo disperse dyes. This study has also shown that cotton which is known to be generally dyed using direct, reactive, or vat dyes, can also be dyed with disperse dyes. Although the colour shade on cotton may not be as bright when compared with nylon fabric, the fastness ratings of these disperse dyes on cotton is quite impressive than on nylon. More so, all the dyes synthesized showed good fastness properties on the various fabrics used. This suggests that that these dyes can be used in textile application without any need of a mordant. In addition, because of their remarkable colours, these dyes can be harnessed in various dye technology processes.

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Author's Contributions

All the authors contributed equally to the conceptualization, analyses and writing of this manuscript.

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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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