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SYNTHESIS OF VARIETIES OF MONOAZO DISPERSE DYES DERIVED FROM AMINOBENZENE DERIVATIVES

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Abstract: Varieties of disperse dyes derived from amino benzene derivatives (figure 1) were synthesized by diazotization and coupling to phenol and salicylic acid. The impure dyes obtained were recrystalized using ethanol solvent. Attractively, all the dyes synthesized showed moderate to good colouristic characteristics. The purity and structural identities of the dyes were confirmed by melting point, UV and IR spectrometry. The UV spectra of the dyes revealed solvatochromism in solvent of different polarities. Dyeing was carried out on nylon, cotton and polyester fabrics. Amazingly, nylon gave the deepest dye depth while polyester had the best fastness properties.
Keywords— azo dyes; solvatochromism; fastness properties; wavelength

1. INTRODUCTION

Dyes are organic materials which can either be absorbed, adsorbed or deposited into a substance in order to give colour to that substance, and would have some level of permanence [1]. Dyes possess auxochromes which are known to affect the colour shade of the chromophore. Auxochromes do this by changing the wavelength and the intensity of absorption [2].

Great quantities of dyes are manufactured yearly, with over hundreds of thousands of synthetic dyes available in the market [3]. However, the three most common chemical groups present in these dyes are azo, anthraquinone and phthalocyanine groups [4, 5]. These classes of dyes are suitable for the dyeing of polyester, nylon and cotton fabrics [6].

Polyester fibres are known to be of a highly compact and crystalline structure. Of all the man-made fibres, they are the most produced. More so, cotton has a compact crystalline structure but not as seen in polyester, where as, nylon is known to be amorphous in nature [7].

2. MATERIALS AND METHODS

The disperse dyes were synthesized by the diazotization of the different aminobenzene derivatives and coupled with phenol and salicyclic acid as outlined in the general procedure (Method A and B). All the reagents used were gotten from commercial sources. The melting points of the dyes were measured using a melting point apparatus and are uncorrected. IR spectra were recorded in the solid state using a Bruker Alpha PFT-IR Instrument.

2.1 Method A: General Procedure for Diazotization

3-Bromoaniline (1.72 g) was added into a beaker containing 10.0 cm3 of concentrated HCl and this was immediately placed in an ice bath in order to keep the temperature below 5 oC. NaNO2 (0.69 g) was dissolved in 5.0 cm3 of water and this solution was added slowly to the acid mixture by using a dropper while stirring for 10 minutes. This produced the diazonium salt. The temperature of the diazonium salt was maintained below 5 0C by keeping it in an ice bath. This procedure was repeated by using 1.38 g of 3- nitroaniline and 1.73 g of 4-sulphanilic acid.

2.2 Method B: General Procedure for Coupling

Phenol (0.94 g) was dissolved in a beaker containing 5.0 cm3 of 2.5 M NaOH. This solution was maintained below 5 0C in an ice bath. The diazonium salt solution was added slowly to the coupling component solution using a dropper while stirring for 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 which was operated at a temperature of 42 0C. This procedure was repeated by using 1.38 g of salicyclic acid.

2.3 DYEING OF FABRIC

2.3.1 Dyeing of cotton and nylon fabrics

A weighed amount of 2.0 g of the dye was dissolved in 100.0 cm3 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 0C. The temperature was later increased to 100 0C 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 [8].

2.3.2 Dyeing of polyester fabric

Dyeing was carried out by using the high temperature high pressure (HTHP) Method. 1% stock solution was prepared by dissolving 1.0 g of the dye in 100.0 g of dimethylformamide (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 0C after being wetted out and dried. The temperature was increased to 130 0C and dyeing continued at this temperature for 45 minutes while a pH of 4.5 – 5.5 was maintained by using glacial acetic acid. The dyed sample was rinsed in cold water, air dried and presented for fastness tests [8].

2.3.3 Wash Fastness Test

The dyed fabric was immersed in a detergent solution containing 0.5 g of detergent in 30 cm3 of distilled water. The fabric was stirred gently for 30 minutes, removed from the detergent solution and washed thoroughly with water. Thereafter, the fabric was air dried and the changes in shades were related to the standard gray scale rating (grade1-5) where 1 is poor and 5 is excellent [9].

2.4 Light Fastness Test

The dyed fabric 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 [10].

2.5 Heat Fastness Test

The dyed fabric was subjected to heat from a pressing iron set at 60 oC for 30 seconds. Thereafter, it was compared with the control sample. The difference in the colour shade was related to the gray scale rating (grade 1-5) where 1 is poor and 5 is excellent [10, 11].

3. RESULTS

The monoazo disperse dyes of the general structure (fig. 1) were synthesized. Tables 1 and 2 show the physical properties and the functional groups of the dyes respectively. The maximum absorption wavelength (λmax) values of the dyes in solvents of different dielectric constants are shown on table 3, while the fastness ratings of the dyes on nylon, cotton and polyester fabrics is displayed on table 4.

4. DISCUSSION

The impure monoazo dyes were recrystallized from ethanol and their purity was monitored by thin layer chromatography (TLC).

The structure of the monoazo disperse dyes synthesized were verified by IR spectrometry and their spectra confirmed the presence of aromatic ring, OH, C=C, C-S, COOH, C-NO2 and the halogen group (Br) as shown in table 2.

The λmax of all the synthesized monoazo disperse dyes were carried out in ethanol, methanol and ethylacetate solvents as illustrated in table 3. The data revealed that solvents had marked effects on the λmax values of the monoazo disperse dyes. The dyes in methanol showed a significant increase in the λmax values when compared with that of the other solvents. This increase in λmax value (bathochronic shift) with change in solvent polarity may be due to $\pi \rightarrow \pi^*$ transition which occured when the dielectric constant (or polarity) of the solvent increased.

Also, the introduction of electron withdrawing groups (NO2, SO3H, COOH and Br) to the benzene ring, caused a marked effect on the λmax. Dyes containing groups with more electron withdrawing inductive ability had lower wavelength values compared to those with lesser electron withdrawing inductive ability as depicted in table 3. Dyes 1 and 4 had the least wavelength values as compared with the other dyes of the same series.

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Dyeing was carried out on polyester, cotton and nylon fabrics. It was observed that the colour strength of the dyed cotton and polyester fabrics were not as solid as that of the dyed nylon fabric. The nylon fabric absorbed more dye molecules than the cotton fabric in simultaneous dyeing, and this may be due to the amorphous nature of nylon which made dye diffusion easier.

Generally, the fastness tests of the dyes on all the fabrics ranged from fair to very good as shown on table 4. However, the fastness properties on polyester fabric were found to be better than was recorded for cotton and nylon fabrics. This could be attributed to the highly crystalline structure of polyester which prevented the easy outward migration of the dye molecules after dyeing. Therefore, nylon being more amorphous in nature, exhibited the "easy in, easy out" phenomenon [11, 12].

5. CONCLUSION

This studies have shown that the introduction of electron withdrawing groups on the benzene component of the monoazo disperse dyes played a significant role in the wavelength and colour shade of the dyes. All the dyes synthesized had moderate to very good fastness properties. More so, the amazing fastness properties and good colour shade of these groups of dyes could play a vital role in synthetic dye industry.

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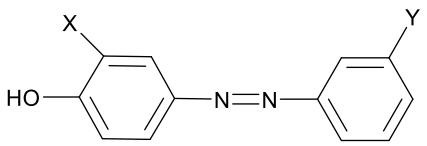

Fig 1

Dye 1: X =H, Y= NO_2

Dye 2: X = H, Y = Br

Dye 3: X = H, Y = SO_3H

Dye 4: X = COOH, Y = NO_2

Dye 5: X = COOH, Y = Br

Dye 6: X = COOH, Y = SO_3H

Fig. 1. *General structure of the synthesized Dyes*

Table 1: Physical characteristics of the synthesized monoazo disperse dyes

Dye no	Colour	Texture	Melting point (^OC)	Weight (g)	Percentage yield of dye (%)
1	Olive Green	Jelly	121	2.30	47.33
2	Rich Gold	Jelly	168	2.90	52.35
3	Cadmium yellow	Jelly	143	2.80	50.36
4	Red Earth	Powdered	144	5.80	98.98
5	Magenta	Powdered	132	5.50	84.10
6	Italia brown	Powdered	118	3.10	47.26

Table 2: Infra-red spectra of the monoazo disperse dyes

IR peaks (cm^{-1})							
V_{O-H}	V_{C-H}	V_{Ar-H}	V_{C-C}	V_{N-N}	V_{COOH}	V_{C-Br}	V_{C-NO2}
3650-3640	3070-3060	1617-1580	1600-1560	1500-1400	800-730	660-600	150-140

Table 3: UV spectra of dyes in solvents of different dielectric constant and their molar extinction coefficient (ε)

Dyes no	λ_{max} in methanol (nm)	λ_{max} in ethanol (nm)	λ_{max} in ethyl acetate (nm)	Molar extinction coefficient (ε) ($Lmol^{-1} cm^{-1}$)
1	456.00	451.00	443.00	2.44×10^4
2	474.00	470.00	463.00	5.52×10^4
3	468.00	462.00	458.00	5.72×10^4
4	358.00	352.00	344.00	3.64×10^4
5	373.00	360.00	357.00	3.87×10^4
6	365.00	357.00	351.00	3.25×10^4

Table 4: Wash fastness and light fastness on dyed nylon and cotton 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	3-4	4	4	3	3-4	4-5	4-5	4
2	4	4	4	4-5	4	4	4-5	4-5	4
3	4	3-4	3-4	4	3-4	4	4-5	4	4
4	4	3-4	4	4	3	3-4	4-5	4-5	4
5	4-5	3-4	4	4-5	4	4	4-5	4-5	4