

SYNTHESES OF NOVEL DISPERSE DYES BASED ON ARYLAZOPHENOLS: SYNTHESIS, CHARACTERIZATIONS AND APPLICATIONS

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ABSTRACT. Three compounds, phenyl propenone, *p*-tolyl propenone, and bromo phenylpropenone derivatives **3a-c** were produced in good yields via condensation derivatives of acetophenone with dimethyl formamide dimethyl acetal (DMFDMA) in presence of xylene as a solvent. Compounds **5a-f** were produced by coupling the enaminones **3a-c** with diazonium chloride **4a** or **4b**. Compounds **5a-f** were reacted with acetone to create novel disperse dyes **9a-f**. Mass spectra, (FT-IR), elemental analysis, and ¹H-NMR spectra were used to confirm the chemical structure of the new dyes. The new disperse dyes were used in dyeing polyester fabrics at low temperature, and fabrics with colors ranging from yellowish brown to dark yellowish brown were obtained. Thus, the different fastness properties of these dyed fabrics were studied, which gave excellent results. Finally, the biological activity of these new dyes was studied, which showed that they have biological activity against bacteria and fungi.

KEY WORDS: Azo dye, Carrier, Low temperature dyeing, Arylazophenols, Color fastness

INTRODUCTION

Enaminones chemistry has received a great attention because of their ease of use. Accessibility by synthesizing disperse dyes [1]. The importance of dyes based on enaminones were represented by a large number of publications [2, 3] regarding this type of dye, known for its extremely attractive dyes properties, especially high contrast gloss and azo dyes were derived from aniline [4] and splendid colors they produce [5]. Their derivatives have been used as intermediates in dye chemistry for a long time. The compounds based on enaminones were utilized as a precursor for the incorporation of fused hetero-aromatics ring cycles that easily presented pharmaceutical and biological activities [6-11]. The purpose of this study was to synthesize a novel disperse dyes based on arylazophenols by utilizing facile route. Also, in this study, the dyeing performance of disperse dyes **9a** to **9f** was analyzed when a 1.5% dispersant and 1% carrier concentration were used in a dye solution with a shade of 3% kept suboptimal to obtain optimal dyeing conditions. Color performance was presented using K/S estimation as the color strength of dyed fabrics. We were also able to introduce the potential biological activities of the new disperse dyes under investigation.

RESULTS AND DISCUSSION

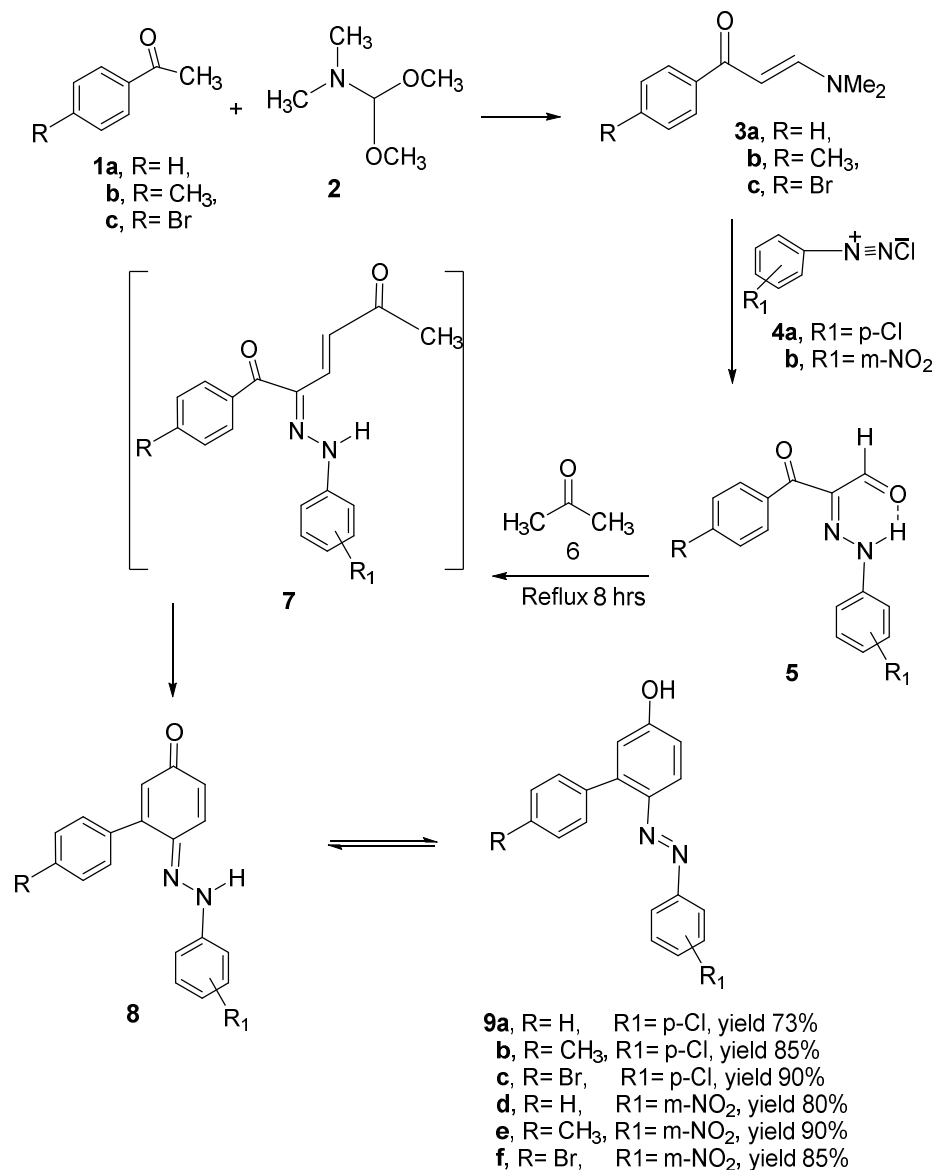
Chemistry

It is worth mentioning here that Scheme 1 shows the reaction of substituted acetophenones **1a-c** in xylene with dimethylformamide dimethyl acetal to synthesize enaminones **3a-c** in excellent

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yields. Conditions for efficient synthesis of enaminones from substituted acetophenones and dimethylformamide dimethylacetals, including microwave heating, have been previously investigated by us [10]. Next, we investigated the coupling reaction of enaminones **3a-c** with diazonium chloride. This procedure yielded the azo dyes **5a-f**, which exist in the anti-form rather than the syn-form in the solid state. Compounds **5a-f** were reacted with acetone to give new disperse dyes **9a-f**. Mass spectra, FT-IR, elemental analysis and ^1H NMR spectra were used to confirm the chemical structures of the new dyes (Scheme 1).



Scheme 1. Syntheses of azo disperse dyes **9a-f**.

Low temperature dyeing process (carrier dyeing)

Carrier dyeing considered an important effective techniques of polyester dyeing as the presence of carrier helping to increase the uptake value of the dyes on the polyester fabric, it also play an important role in dyeing of sensitive fabrics that does not need much heat in their dyeing process as much heat above 100 °C harmed these types of fabrics. The dyeing process gives us different yellow brown colors for the six dyes (pale yellowish brown to dark yellowish brown color). The color strength of the dyes has the descending order according to the K/S value as follow **9f** > **9a** > **9d** > **9c** > **9e** > **9b** (Figure 1).

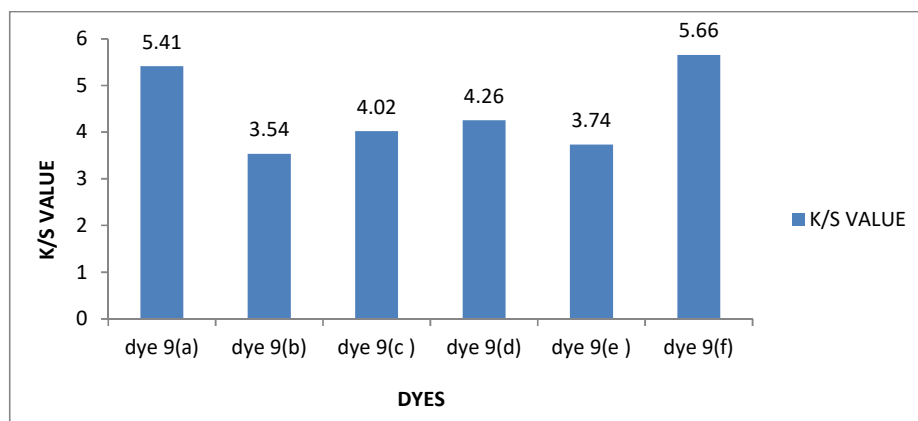


Figure 1. The color strength K/S of the synthesized disperse dyes **9a-f**.

Dyeing baths reuse impact

In our innovative strategy towards the synthesis, characterization and application of new disperse dyes in the dyeing of polyester fabrics, we take into account the non-pollution of the environment by dumping colored waste that harms the environment, but rather we treat it [12-22]. In this study, we use dyeing baths effluents after the first dyeing process as a kind of waste treatment that harms the environment. The dyeing baths solutions of innovative dyes **9a-f** are analyzed after the dyeing process and reconstituted with the amount of fresh water required to maintain a constant ratio of the solution to the original volume.

To keep the pH at 5.5, the pH of the remaining dye solution is monitored. In order to improve washing fastness, the reduction was clarified using caustic soda (1 g/L), sodium hydrosulfite (1 g/L), soap with 2% non-ionic detergent (pH 8), and 50°C for 15 minutes.

It is of value to reveal here, from the data obtained in table 1 that represented in figure 2 we observed that color strength K/S value of the dye bath reuse process in 1st, 2nd and third re-dyeing process at 100 °C vary from (70, 50 and 15) % from its original value obtained in the dyeing process for all the dyes which prove that dyeing reuse is an effective method of reduction costs, water, energy and chemicals also water pollution prevention. Also, from the data obtained about the fastness properties of the disperse dyes in tables 2 at concentration of dye shades (3%) and by using of the dyeing process at 100 °C, we observed that, the six dyes showed from good to excellent washing, and perspiration fastness properties according to grey scale, while showed moderate light fastness properties according to the blue scale. We can say that, the better washing fastness of these disperse dyes are come back to the partly insolubility of the disperse dyes and partly to the hydrophobic nature of the textile fibers. The washing fastness ratings range between

(4-5) according to grey scale. The light fastness shows moderate to good light fastness rating which is found in the range of (3-4) to (4-5). And that is may be due to attributed partly to the disperse dyes non-ionic nature, which will not easily attract water molecules and other polar molecules that may have an accelerate effect on light fading.

Table 1. Effect of dyeing method and dyeing baths reuse at 100 °C using shade 3%.

Dye No	Polyester dyed fabrics	L^*	a^*	b^*	C^*	h^*	K/S
9a	Dyeing	81.08	-0.91	4.12	4.22	102.43	5.41
	1st dye reuse	68.71	5.08	23.44	23.99	77.77	3.73
	2nd dye reuse	73.72	1.12	27.39	27.41	87.66	2.94
	3rd dye reuse	72.25	3.76	44.58	44.47	85.19	0.69
9b	Dyeing	82.12	-0.22	0.20	0.29	137.75	3.54
	1st dye reuse	80.49	-0.33	4.37	4.38	94.35	2.16
	2nd dye reuse	73.90	2.27	17.14	17.29	82.44	0.73
	3rd dye reuse	70.32	4.02	23.76	24.09	80.39	0.45
9c	Dyeing	81.07	-0.70	3.12	3.20	102.67	4.02
	1st dye reuse	77.32	-0.09	13.86	13.86	90.83	2.36
	2nd dye reuse	72.69	1.43	24.40	24.44	86.65	1.31
	3rd dye reuse	69.72	1.74	37.34	37.38	78.33	0.54
9d	Dyeing	80.84	-0.26	3.50	3.51	94.21	4.26
	1st dye reuse	69.72	3.67	18.86	19.21	78.99	2.27
	2nd dye reuse	68.63	6.23	34.06	34.63	79.63	0.63
9e	Dyeing	79.44	-0.25	6.42	6.43	92.27	3.74
	1st dye reuse	74.68	1.07	17.43	17.47	86.48	2.10
	2nd dye reuse	71.00	3.21	26.39	26.59	83.07	0.89
9f	Dyeing	81.58	-0.46	2.41	2.45	100.87	5.66
	1st dye reuse	75.02	1.16	19.84	19.87	86.85	2.13
	2nd dye reuse	73.07	2.95	35.60	35.72	85.26	0.55

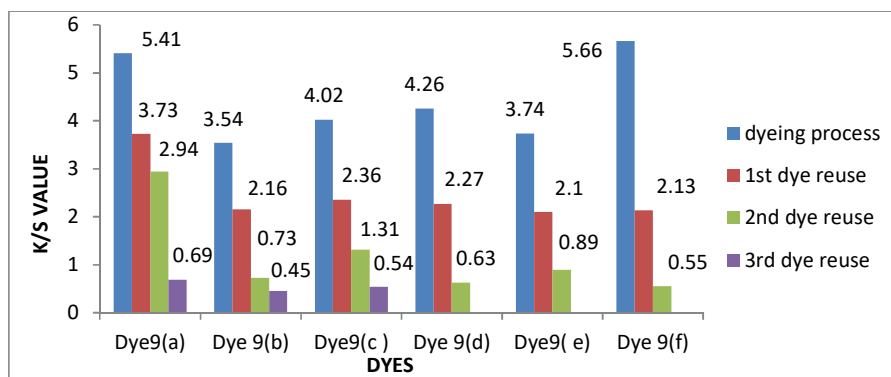


Figure 2. Use of dyeing bath effluents of disperse dyes **9a-f**.

Finally we can observe from Table 2 that fastness to perspiration of both alkaline and acidic nature are excellent for the all dyes at both low temperature dyeing process where its value approximately 5 degree and this is satisfied result for our dyes.

Table 2. The fastness properties of the new dyes with shade 3% at 100°C dyeing process.

Dye No.	Washing			Perspiration fastness						Light fastness
				Acidic			Alkaline			
	SC	SP	ALT	SC	SP	ALT	SC	SP	ALT	
9a	5	4	4-5	5	5	5	5	5	5	3-4
9b	5	4	4-5	5	5	5	5	5	5	4
9c	5	4	4-5	5	5	5	5	5	5	3-4
9d	4-5	4	4-5	5	5	5	5	5	5	4
9e	4-5	4	4-5	5	5	5	5	5	5	4
9f	5	4	4-5	5	5	5	5	5	5	4-5

Antimicrobial activity of the dyes 9a-f

Compounds with electron-withdrawing or electron-donating groups at the *ortho*, *meta*, and *para* positions of the hydrophobic aryl ring are more reactive than other derivatives according to structure-activity relationship studies. The acceptor side; therefore, substitutions in the hydrophobic domain could be the reason for the compound's promising properties. Using the diffusion agar technique with a well diameter of 6.0 mm (tested volume: 100 L), six dyes' biological activity were assessed against four different microbiological cultures. Pure cultures of Gram-negative bacteria *Escherichia coli*, Gram-positive bacteria *Staphylococcus aureus*, as well as fungi *Aspergillus fumigatus* and *Candida albicans*, were used. Ketoconazole was used as a positive control for the fungi (gentamycin).

Table 3. Antimicrobial activity of the dyes 9a-f.

Tested microorganisms	Inhibition zone diameter (nearest mm)						
	Dye 9a	Dye 9b	Dye 9c	Dye 9d	Dye 9e	Dye 9f	
Fungi							Ketoconazole
<i>Aspergillus fumigatus</i>	16	15	19	NA	17	NA	20
<i>Candida albicans</i> RCMP 005003(1) ATCC 10231	28	13	18	16	15	26	
Bacteria							Gentamycin
<i>Staphylococcus aureus</i> (RCMP010010)	24	12	16	14	14	23	24
<i>Escherichia coli</i> (RCMP 010052) ATCC 25955	15	12	12	NA	NA	NA	16

The materials were evaluated at a concentration of 10 mg/mL, with the mean zone of inhibition measured in millimetres beyond the well diameter at the National Research Center. The results are shown in the Table 3. According to the inhibitory zones shown in that table, the dyes 9a-f have effective antibacterial properties against at least two of the examined pathogens. Dye 9a shows strong activities with significant inhibition zone equal 16 mm against *Aspergillus flavus* (RCMP0022002) and equal 28 mm against *Candida albicans* (RCMP 005003(1)ATCC 10231) fungi and strong activities against gram positive *Staphylococcus aureus* (RCMP010010) equal 24 mm and *Escherichia coli* (RCMP 010052) ATCC 25955 gram positive bacteria equal 15 mm.

Dye 9b shows strong activities with significant inhibition zone equal 15 mm against *Aspergillus flavus* (RCMP0022002) and moderate activities equal 13 mm against *Candida albicans* (RCMP 005003(1)ATCC 10231) fungi and moderate activities against gram positive *Staphylococcus aureus* (RCMP010010) equal 12 mm and strong activities against *Escherichia coli* (RCMP 010052) ATCC 25955 gram positive bacteria equal 12 mm.

Dye **9c** shows strong activities with significant inhibition zone equal 19 mm against *Aspiggillus flavus* (RCMP0022002) and moderate activities equal 18 mm against *Candida albicans* (RCMP 005003(1) ATCC 10231) fungi and moderate activities against gram positive *Staphylococcus aureus* (RCMP010010) equal 16 mm and strong activities against *Escherichia coli* (RCMP 010052) ATCC 25955 gram positive bacteria equal 12 mm.

Dye **9d** shows no activities against *Aspiggillus flavus* (RCMP0022002) and moderate activities equal 16 mm against *Candida albicans* (RCMP 005003(1)ATCC 10231) fungi and moderate activities against gram positive *Staphylococcus aureus* (RCMP010010) equal 14 mm and no activities against *Escherichia coli* (RCMP 010052) ATCC 25955 gram positive bacteria.

Dye **9e** shows strong activities with significant inhibition zone equal 15 mm against *Aspiggillus flavus* (RCMP0022002) and moderate activities equal 13 mm against *Candida albicans* (RCMP 005003(1)ATCC 10231) fungi and moderate activities against gram positive *Staphylococcus Aureus* (RCMP010010) equal 12 mm and no effect against *Escherichia coli* (RCMP 010052) ATCC 25955 gram positive bacteria. Dye **9f** shows no activities against *Aspiggillus flavus* (RCMP0022002) and strong activities equal 26 mm against *Candida albicans* (RCMP 005003(1)ATCC 10231) fungi and strong activities against gram positive *Staphylococcus aureus* (RCMP010010) equal 23 mm and no activities against *Escherichia coli* (RCMP 010052) ATCC 25955 gram positive bacteria.

EXPERIMENTAL

Electron impact mass spectra were performed at the regional center for mycology and biotechnology Azhar University, IR spectra were regulated by a Jasco FT/IR 4700 spectrophotometer, and all reactions were followed by thin layer chromatography (TLC) using Merck aluminum plates, ¹H NMR–spectra were accounted for by a Bruker Avance 400 spectrophotometer at 400 MHz and Elemental analysis were made utilizing PerkinElmer 2400 analyzer (Perkin Elmer, Norwalk, CT, United States). Solvent utilized in this exploration study were gotten from Fluka and Aldrich for both of the synthesis process and spectroscopic estimation.

Preparation of compounds **3a-c** and dyes **5a-f**

Compounds **3a-c** and dyes **5a-f** were prepared according to our published recipe [10].

Synthesis of disperse dyes **9a-f**

Compounds **5a-f** (10 mmol) were refluxed in acetone (0.58 g, 10 mmol) in the presence of triethylamine (3 mL) for 8 hours. Solvent was reduced under vacuum. The remaining product was poured into water and neutralized with dilute hydrochloric acid. The solid product was collected by filtration and crystallized from a mixture of ethanol/dioxane 3/1.

6-[(*E*)-(4-Chlorophenyl) diazenyl] [1, 1'-biphenyl]-3-ol **9a**. Formed brown crystals, yield: (73%), m.p. 160 °C yield: (73%); IR (cm⁻¹) 3448 (OH); MS m/z (M⁺) = 308; anal. calcd. For C₁₈H₁₃ClN₂O (308.76): C, 70.12; H, 4.22; N, 9.09. Found: C, 70.05; H, 4.50; N, 9.31. ¹H NMR (DMSO-d₆): δ = 7.33-8.49 (m, 12H, arom-H), 10.02 (s, 1H, OH).

6-[(*E*)-(4-Chlorophenyl) diazenyl]-4'-methyl [1, 1'-biphenyl]-3-ol **9b**. Formed brown crystals, yield (85%); m.p. 135 °C; anal. calcd. for C₁₉H₁₅ClN₂O: (322.09), C, 70.80; H, 4.65; N, 8.69. Found: C, 71.09, H, 5.12, N, 8.85; MS m/z (M⁺) = 322; IR: 3300, (OH); ¹H NMR (DMSO-d₆): δ = 3.65 (s, 3H, CH₃), 6.65-7.83 (m, 11H, arom-H), 8.82 (s, 1H, OH).

4'-Bromo-6-[(E)-(4-chlorophenyl) diazenyl] [1, 1'-biphenyl]-3-ol 9c. Formed brown crystals, yield (90%); m.p. 145 °C; anal. calcd. for C₁₈H₁₂BrClN₂O: (387.66), C, 55.77; H, 3.12%; N, 7.23. Found: C, 55.86, H, 3.17, N, 7.45; MS m/z (M⁺) = 387; IR: 3350, (OH); ¹H NMR (DMSO-d₆): δ = 6.61-7.82 (m, 11H, arom-H), 10 (s, 1H, OH).

6-[(E)-(3-Nitrophenyl) diazenyl][1, 1'-biphenyl]-3-ol 9d. Formed brown crystals, yield (80%); m.p. 135 °C; anal. calcd. for C₁₈H₁₃N₃O₃: (319.314), C, 67.71; H, 4.10; N, 13.16. Found: C, 67.68, H, 4.59, N, 13.40; MS m/z (M⁺) = 319; IR: 3300, (OH); ¹H NMR (DMSO-d₆): δ = 7.53-7.63 (m, 12H, arom-H), 10 (s, 1H, OH).

4'-Methyl-6-[(E)-(3-nitrophenyl) diazenyl] [1, 1'-biphenyl]-3-ol 9e. Formed brown crystals, yield (90%); m.p. 150 °C; anal. calcd. for C₁₉H₁₅N₃O₃: (333.34), C, 68.64; H, 4.54; N, 12.61. Found: C, 68.45, H, 5.02, N, 12.47; MS m/z (M⁺+1) = 334; IR: 3450, (OH); ¹H NMR (DMSO-d₆): δ = 3.65 (s, 3H, CH₃) 7.15-8.65 (m, 11H, arom-H), 9.51 (s, 1H, OH).

4'-Bromo-6-[(E)-(3-nitrophenyl) diazenyl] [1, 1'-biphenyl]-3-ol 9f. Formed brown crystals, yield (85%); m.p. 155 °C; anal. calcd. for C₁₈H₁₂BrN₃O₃: (398.21), C, 54.29%; H, 3.04%; N, 10.55%. Found: C, 53.86%, H, 3.17%, N, 10.76%; MS m/z (M⁺+1) = 399; IR: 3450, (OH); ¹H NMR (DMSO-d₆): δ = 7.37-7.91 (m, 7H, arom-H), 8.00-8.32 (m, 4H, arom-H), 9.52 (s, 1H, OH).

Low temperature dyeing process at 100 °C (carrier dyeing)

Disperse dyes **9a-f** were created by dissolving the necessary amount of dye (3% colours) in 2 mL of DMF, adding drops at a time, and agitating the dye solution. In the event of dyeing at 100 °C, solution ratio 1:30 contains (1.5%) MDL leveal as an anionic dispersion (TANATEX Chemical) and (1.0%) Tanaval EP 2007 as an anionic eco-carrier (TANATEX Chemical). A moist polyester fabric (3 g) was added after the PH value of the dye solution was changed using acetic acid in water to 5.5. The temperature of the dye bath solution is raised to 100 °C and kept there for an hour in order to begin dyeing. After washing the colored fabrics in fresh water and cleaning them with a decontamination clearing solution (1 g/L caustic soda, 1 g/L sodium hydrosulphite, 10 min. and 80 °C), the dyeing path is cooled to 50 °C. After being washed in both hot and cold water and neutralised with acetic acid, the samples were dried by air.

Color measurements

On the machine reflectance spectrometer, colorimetric characteristics of polyester fabrics treated with disperse dyes were discovered. Utilizing the Ultra Scan PRO D65 UV/VIS spectrophotometer's light reflection technique, the color performance of the dyed samples was determined. The colour intensity was measured using Kubelka-Mink equation and given as a K/S value.

$$K/S = [(1 - R)^2 / 2R] - [(1 - R_0)^2 / 2R_0]$$

where *K* = absorption coefficient, *R* = decimal fraction of the reflectance of the dyed fabric, *R*₀ = decimal fraction of the reflectance of the undyed fabric, and *S* = scattering coefficient.

Color fastness to washing

The ISO 105-C02 technique from 1989 was used to determine the washing fastness [10]. Two samples of bleached cotton and wool fabric were sandwiched between mixed test pieces, which were then submerged in an aqueous solution with a solution ratio of 5 g/L of non-ionic detergent (1:50) the tank for 30 min at 60 °C After a predetermined amount of time, the samples were taken

out, twice washed while periodically applying hand pressure, and then dried. Ratings of washing fastness are established [10].

Color fastness to perspiration

According to test method ISO 105-E041988, two solutions (acid and alkaline) were created. The following procedure was used to test for resistance. The composite design was created by stitching a 5 cm × 4 cm dyed piece between two uncolored swatches. In order to guarantee full soaking, mixed samples were submerged in both solutions for 15 to 30 min while being vigorously agitated and compressed. The sample is held between two glass or plastic plates by a force of about 4-5 kg. The combined sample plates were then maintained upright in an oven at 37.2°C for 4 hours. The impact on the test sample's color is demonstrated and evaluated using the grey scale for color alteration.

Color fastness to light

Light fastness test carried out according to ISO 105-B02. A carbon arc lamp was used in the test, which was being run nonstop for 35 hours. One can finally conclude that the nature of the fabric into which the dye has been dispersed was the cause of the colour of the dyed fabric increasing with increasing dye concentration. Different fabrics contain different chemical groups, and these substituents can significantly affect the light fastness index of a dye on a given fabric. The incoming radiation's wavelength distribution; not all absorptions begin the bleaching process equally well. The humidity and chemistry of the atmosphere can significantly affect the rate at which certain colorants fade. The blue scale was used to record the tested materials' colour changes.

Biological activity (disc diffusion method)

For bacteria or malt agar, the disc diffusion method had been utilised to screen samples of polyester fabric for antibacterial activity (for bacteria) [11]. Yeast was inoculated with 0.1 mL of a suitable diluent for the inoculum to be evaluated. A layer of test tissue samples, each measuring 1 cm in diameter, was applied to the surface of the culture plate. Plates were incubated at the right temperature for 24 hours. The diameter of the zone of inhibition (mm), which includes the disc's diameter [11], was calculated for each treatment. In this study, the antibacterial efficacy of recently synthesised disperse dyes was investigated using the agar plate diffusion method. The four different types of bacteria tested were: *Staphylococcus aureus* (G+ve), *Candida albicans* RCMP 005003(1) ATCC 10231 and *Aspergillus fumigatus* (a representative fungal species), and *Escherichia coli* (RCMP 010052) ATCC 25955 (G-ve) were used. For bacteria and yeast, 0.1 mL of a density of 10⁵–10⁶ cells/mL was added to the nutrient agar plates as an inoculum. On top of the plates, the treated plates (1 cm) are positioned. Following that, the plates were kept at a low temperature (4 °C) for 2-4 hours. To ensure the best possible microbial development, plates were then incubated for 24 hours in an upright position at 30 °C for yeast and 37 °C for bacteria. The antibacterial activity of the test substance was identified by measuring the diameter of the inhibition zone, which is specified in millimetres (mm). The investigation was repeated many times, and the mean was reported [11].

CONCLUSION

Complementing our strategy towards the synthesis and characterization of innovative disperse dyes, we have synthesized a novel disperse dyes and verified the chemical composition of these innovative dyes, so we applied them in dyeing polyester fabrics at a temperature of 100 degrees

Celsius. Color strength values were evaluated for textiles colored with these disperse dyes at 3% shade. The fastness properties of polyester fabrics dyed with disperse dyes were measured according to known procedures, giving us an acceptable light fastness and very good wash and perspiration fastness. The antimicrobial activity of the new dispersed dyes against different types of microorganisms was also discussed, which gave promising results for the use of these innovative dyes in medical and pharmaceutical purposes.

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