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Dyeing of Polyester Fabrics with Eco-Friendly Modified Natural Dyes Using IR and Ultrasonic Methods without Mordants or Dispersion Agents

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Abstract

This study aims to synthesize and characterize new disperse dyes based on modification of curcumin natural dye. When these dyes were used on polyester fabric, the resulting dyed fabrics had good colour strength. Without the use of a mordant or dispersion agent, they also demonstrated good light fastness and other fastness characteristics. Two steps were involved in the dyes' synthesis. The initial p-amino phenol was diazotized (0.01 mole, 1. 09 g) or 2-chloro-p-nitroaniline (0.01mol, 1.72 g) in hydrochloric acid with sodium nitrite. The Second step is the coupling with curcumin (0.01mol, 3. 68 g) to give two disperses dyes D1 and D2 respectively. Dyeing polyester fabric take place in IR machine and also, Ultrasonic dyeing with dispersing agent and without dispersing are performed. Dyed polyester fabrics without dispersing agent gave higher exhaustion and fixation values than polyester dyed with dispersing agent. Also, they showed higher fastness properties against washing, rubbing, perspiration and light fastness. Modified curcumin dyes also, impart dark yellow colour without using mordant. In this work, analytical and spectral data were used to confirm the structures of the new disperse dyes that were created by modifying the naturally occurring color curcumin.

Keywords: Curcumin, disperse dyes, IR machine, ultrasonic dyeing, fastness properties

1. Introduction

A perennial herb called turmeric has been used for ages throughout Asia as a natural colour. Curcumin, which gives it a yellow to orange tint, is present. Using a straightforward extraction method, the natural colour curcumin was recovered from Curcuma longa L. Due to the fact that curcumin loses some of its colour over time, it is regrettably categorised as a fugitive dye. To enhance the hue of curcumin, certain chemicals are included in the dye composition. No organic solvents were used in the extraction of the turmeric juice. The juice was mixed with a mordant and an emulsifier to make the dye solution. Alum and lime juice were utilised as artificial and natural mordants, respectively [1-3]. The dye was used as a sensitizer in a dye-sensitized solar cell (DSSC) without further purification [4, 5].

A well-known healing herb, turmeric has a wide range of pharmacological benefits, including antioxidant, antiprotozoal, anti-inflammatory, anti-proliferative, anti-tumor, and anti-aging qualities. Curcuminoids have gained potential therapeutic interest for the treatment of immune-related metabolic disorders and cancer due to their wide spectrum of biological targets and absence of adverse effects. [6-15]. We looked at the antibacterial, anticancer, and antioxidant characteristics of thaizolyl dispersion dyes. To ascertain their dyeing capabilities, they were tested on polyester materials [16-18]. Disperse dyes with a hydrophobic character are frequently used to colour polyester [19-21]. Using UV/ozone pretreatment to activate fibre and enhance polyester and nylon's dyeability when dying synthetic materials with natural colours [22, 23]. As a

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result, some researchers have created emulsion by mixing natural colours with additions like surfactants. A water-in-oil (W/O) natural dye made from emulsion that is a striking shade of red [5, 24, 25]. During the dyeing process, sonication can also enhance the dye's adhesion and fastness capabilities on cotton cloth [26]. Wool and silk were dyed by using a combination of synthetic and natural dyes [21, 27-29], Also, they were treated with nano chitosan to increase its dyeability and antibacterial properties [2, 17, 19, 20, 22, 27, 30].

This study is being conducted to better understand the effect of azo dye coupling with curcumin on colour strength, colour fastness, colour gradation, or curcumin coordination on polyester without mordant or dispersing agents

2. Experimental

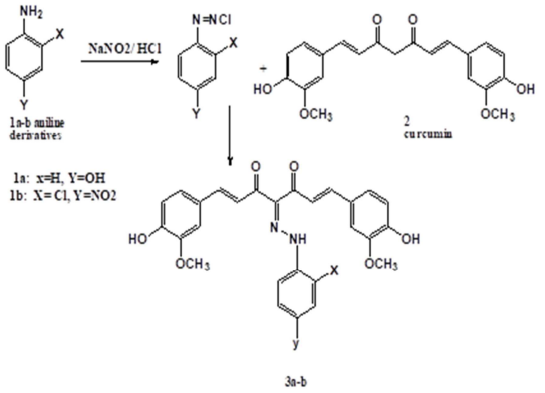
Materials and method

The "Gallenkamp melting point apparatus" assigned all melting points without making any corrections. The infrared spectrophotometer PyeUnicam SP-3-300 and potassium bromide discs were used to determine the wave number. Tetramethylsilane was used as an internal standard in deuterated dimethyl sulphoxide (DMSO-d_6), and ^{1}H-NMR spectra were obtained at "400MHz, on a Varian Mercury VX-300 NMR spectrometer" at the Main Chemical Laboratory. Chemical shifts were indicated in ppm. Mass spectra were gathered at the Micro Analytical Centre using Shimadzu GCMS-QP-1000EX mass spectrometers set at 70eV.

100% polyester was employed in the study's materials, which were bought in the local textile market. At a local market, fresh turmeric (Curcuma domestica Val) was bought.

General procedure for 2-(phenyl-1-yl) hydrazono curcumin (3a-b)

The solution of different phenyl amine was cooled to 0-5°C and then treated with 10 ml HCl/0.69 g $NaNO_2$ to produce the diazonium salt. Dropwise additions of diazonium salt solution (3.68 g in 20 ml) were made while stirring continuously to a cold solution of curcumin 2 in NaOH (0.5M). After 4 hours of stirring at 0-5°C, the precipitate was filtered, rinsed with water, dried, and recrystallized from ethanol. **Scheme 1.**



Scheme 1: Synthesis of 2-(phenyl-1-yl) hydrazono curcumin (3a-b)

1,7-bis(4-hydroxy-3-methoxyphenyl)-4-(2-(4-hydroxyphenyl-2yl)hydrazono)hepta-1,6-diene-3,5-dione **(3a)**

yellow solid, yield: 80 per cent, mp > 300°, λ_{max} (H_2O) 400 nm; IR(KBr, λ_{max} cm^1): 3410, 3502,3312 (3OH), 3318 (NH),1691 (C=O), 1660 (C=N), 1510(C=C conjugated). ^{1}HNMR (400MHz, DMSO-d6, ϭ/ppm): 3.62 (s, 6H, 2OCH3), 5.61-5.51(s, 3 H, 3OH), 6.40 (d, 4H, vinylic H), 6.910-7.40 (m, 10H, ArH), 7.98 (s, 1H, NH). MS, m/z (per cent): Anal. Calcd. For C27H24N2O7 (488): C66.39; H, 4.92; N, 5.74 per cent, Found C, 66.09; H, 4.79; N, 5.69 percent.

1,7-bis(4-hydroxy-3-methoxyphenyl)-4-(2-(2-chloro-4-nitro-phenyl-2 yl) hydrazono) hepta-1,6-diene-3,5-dione **(3b)**

Yellowish brown solid, yield: 77 per cent, mp > 300°, λ_{max} (H_2O) 390 nm; IR(KBr, λ_{max} cm^1): 3410, 3502, (2OH), 3318 (NH),1691 (C=O), 1660 (C=N), 1510(C=C conjugated). ^{1}HNMR (400MHz, DMSO-d6, ϭ/ppm): 3.62 (s, 6H, 2OCH3), 5.61-5.51(s, 3 H, 3OH), 6.40 (d, 4H, vinylic H), 6.90-7.40(m, 9H, ArH), 7.98 (s, 1H, NH). MS, m/z (per cent): Anal. Calcd. For C27H22N3O8CL (551.5): C58.75; H, 3.99; N, 7.62 per cent, Found C, 58.09; H, 3.79; N, 7.59 percent.

Dyeing procedures

IR dyeing

Disperse dyes **1** and **2** were applied on polyester at LR 50:1 using an infrared mechanism. Dyeing was carried out at pH 5 with a concentration of 2% of the

dye without dispersing agent for comparison, we add dispersing agent. To the dyeing bath, add 2 g/L of anionic (Sera Gal P-LP, DyStar, Egypt). Starting at 40°C, the dyeing process was gradually raised to 120°C. At the optimum temperature of 120 ° C, the dyeing procedure was then repeated at intervals of 20, 40, and 60 minutes. After being dyed, all samples were rinsed with water and allowed to air dry.

Ultrasonic dyeing

Disperse dyes 1 and 2 were applied at LR 50:1 using an ultrasonic mechanism. The dye bath, prepared at pH 5 with a concentration of 2% of the dye concentration. Both dispersion agents and none were used during the dyeing process, which started at 40°C and subsequently escalated to 80°C. At the desired temperature of 80 ° C, the dyeing process was then continued for another 15 minutes. After being dyed, all stained samples were rinsed with water and allowed to air dry.

Measurements and testing

Dye exhaustion

Tests and Measurements

Exhaustion of the dyes

By taking samples from the dyebath both before and after dying, the dye concentration was calculated. Spectrophotometric measurements were made of the dyebath's concentration (g/l) at the maximum of each dye. Equation (1) was used to calculate the percentage of dye exhaustion (% E)

$\%E = C_1\text{-}C_2/C_1 \times 100$ (1)

where C_1 and C_2 are the concentrations of dye in the dye bath before and after dyeing, respectively.

Dye fixation.

Colour fixation In order to extract the unfixed dye from the dyed samples, the samples were refluxed in 50% aqueous DMF (liquor ratio: 20:1) for 15 minutes. This measurement of dye fixation (%F) is the percentage of the exhausted dye that chemically binds to the fibre. Until the extract was colourless, this process was repeated. The dye fixation ratio was then determined using equation 2 after measuring the concentration of the extract spectrophotometrically at the maximum of each dye:

(2) %F = C1-C2-C3 /C1-C2 100

where C3 is the amount of extracted dye.

Equation 3 was used to compute the total dye fixation (%T), or the proportion of dye chemically attached to the total amount of dye employed, for the manufactured dyes from dyebath exhaustion (%E) and dye fixation (%F).

$\%F = C_1\text{-}C_2\text{-}C_3/C_1\text{-}C_2 \times 100$ (2)

where C_3 is the amount of extracted dye.

Equation 3 was used to compute the total dye fixation (%T), or the proportion of dye chemically attached to the total amount of dye employed, for the manufactured dyes from dyebath exhaustion (%E) and dye fixation (%F).

$\%T = \%E \times \%F / 100$ (3)

Measures of colours

Additionally, the dyed fabrics' relative colour strength (K/S) and CIELAB coordinates (L*a*b* c* h*E*) were assessed using a Hunter Lab Ultra Scan PRO spectrophotometer (USA) with an illumination of D65 and a 10-degree standard observer. Using Kubelka-Munk equation 4, the light reflectance approach was used to determine the K/S value of coloured fabrics. The following equation was used to calculate the reflectance (R) of the coloured fabrics.

$K/S = (1\text{-}R)2 / 2R$ (4),

where R is the decimal fraction of the coloured fabric's reflection.

K = Absorption coefficient, and S for the scattering factor

Testing of fastness

Following removal with 2 g non-ionic detergents at 80 ^oC for 15 min, dyed polyester samples with 2% shade (o.w.f.) were assessed according to conventional ISO procedures. Using the visual ISO greyscale, wash fastness [ISO 105-C02 (1989)] and fastness to perspiration [ISO 105-E04 (1989)] were assessed for both colour change. Xenon arc light fastness was assessed using ISO 105-B02 [31, 32].

3. Results & Discussions

Applications of dyes

Under certain exhaust dyeing conditions that are detailed in the experimental section, the disperse dyes 1 and 2 were produced.

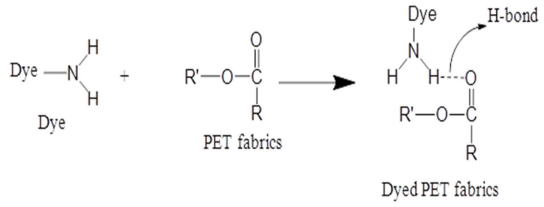


Figure I: reaction mechanism between dyes and treated fabric

The reaction mechanism of disperse dyes based on modification of curcumin natural dye and polyester fabrics is a complex process that involves several steps. The first step is the synthesis of the disperses dye. This is typically done by reacting curcumin with a diazo compound to form an azo dye. The azo dye is then dispersed in a carrier medium, such as water or an organic solvent. The next step is the dyeing of the polyester fabric. The disperse dye is applied to the fabric in a dyeing bath. The dye molecules then diffuse into the polyester fibres and become trapped there. The final step is the fixation of the dye. This is typically done by heating the dyed fabric to a high temperature. This helps to lock the dye molecules into the fibres and prevent them from bleeding (see figure I).

IR dyeing

The effect of the time change on exhaustion and fixation value on polyester fabric

In this research, the effect of the time change on the fixing and exhaustion values of polyester fabric coloured with disperse dyes 1 and 2 was examined. The dyeing was done in a L: R (50:1) ratio for 20 to 40 to 60 minutes at 120 degrees Celsius, 2% (owf), and pH (5). Using an IR dyeing equipment, dyeing was done both with and without a dispersion agent. In the absence of the dispersing agent in the dyeing solution, the dyeing of polyester fabric with the prepared dyes showed very good exhaustion and total fixation as well as the K/S values on the fibre, recording exhaustion and total fixation of 90.06% and 86.21%, respectively, at 60 minutes, while recording exhaustion and total fixation of 80.66% and 78.34%, respectively, at the same time. From the results shown in (Figures 1 and 2), dye exhaustion and total fixation for dye 1 are significantly higher than those for dye 2 and that the extent of dye exhaustion and total fixation increases with increasing time to give the highest value at 60 minutes. Due to dye 1's free donating group (OH) in the structure and the increased dispersion of dispersed dyes on fabric, dye 1 has a higher affinity for the fabric and experiences higher levels of exhaustion and total fixation than dye 2, which contains (NO_2), which results in lower values for exhaustion and fixation.

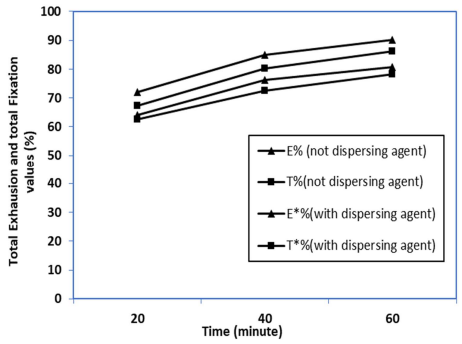


Figure 1: The total Exhaustion and total Fixation values of dye 1 at different time intervals

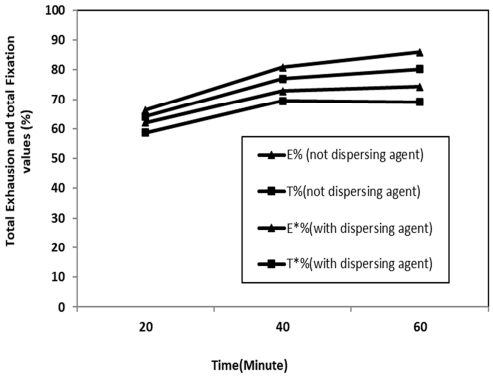


Figure 2: the total Fixation and Exhaustion values of dye 2 throughout various time periods

Ultrasonic dyeing

The Result showed that dyeing polyester with disperse dyes (1,2) in ultrasonic machine at 80 ˚C for 15 minute as in (**Figure 3**) with prepared dyes exhibited has very high exhaustion and fixation with decreasing time of dyeing . Also, the exhaustion and total fixation values on the fibre without dispersing agent is recorded 80.06 %, 76.21 % respectively, while it recorded 73.96, 70.44 respectively with dispersing agent so it decreased the uses of auxiliaries . Additionally, due to the presence of the hydroxyl group, dye 1 exhibits far higher levels of total fixation and dye exhaustion than dye 2 does

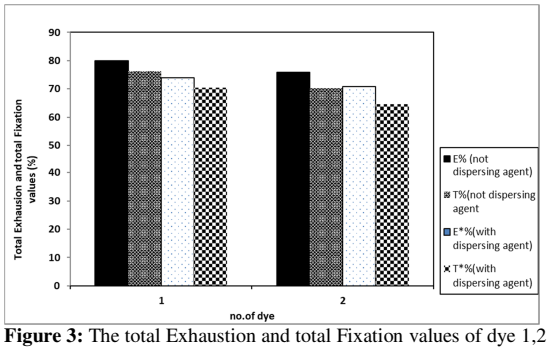


Figure 3: The total Exhaustion and total Fixation values of dye 1,2 with and without dispersing agent using ultrasonic dyeing technique 80°C for 15 min.

Colourimetric data and Fastness properties

Both the CIE lab and the modified CIE L* C* h0 (D65/100) systems were used to assess colour parameters. The colorimetric information from Table I for the dyed polyester samples with dyes 1 and 2 at 20, 40, and 60 C with and without dispersing agents. Additionally, Table II displays the colorimetric results of fabric that was dyed using dyes 1 and 2 at 80 °C with and without dispersing agents. Using the digital CIE lab system, the coloured samples were given the following colour parameters: Lightness (L*), a* colour (red if positive coordinate, green if negative), b* colour (yellow if positive coordinate, blue if negative), C*-Chromaticity, h0 colour hue. Also, Table III shows that the dyed samples had excellent rubbing, washing, and perspiration fastness without dispersing agents at IR and ultrasonic techniques.

Table I: Colorimetric data of the dyed polyester with dye **1** and **2** at 20, 40, 60 ˚C with and without dispersing agents using IR technique

ID	L*	a*	b*	dE*	C*	h◦
Dye**1** at 20◦C Without dispersing	66.49	13.75	38.40	45.54	40.78	70.30
Dye**1** at 20◦C with dispersing	74.84	6.63	26.57	29.83	27.38	76.00
Dye**2** at 20◦C without dispersing	72.09	5.39	23.41	28.03	24.03	77.04
Dye**2** at 20◦C with dispersing	80.67	-0.01	21.45	22.18	21.45	90.02
Dye**1** at 40◦C Not dispersing	57.97	20.77	41.68	54.75	46.57	63.51
Dye**1** at 40◦C with dispersing	73.29	1.57	17.94	22.23	18.00	85.00
Dye**2** at 40◦C without dispersing	63.98	15.94	38.76	47.69	41.91	67.64
Dye**2** at 40◦C with dispersing	75.23	4.35	22.24	25.33	22.66	78.93
Dye**1** at 60◦C Without dispersing	56.36	18.19	38.74	52.45	42.80	64.85
Dye**1** at 60◦C with dispersing	74.43	6.70	24.89	28.53	25.77	74.94
Dye**2** at 60◦C without dispersing	68.44	14.66	36.34	43.29	39.19	68.03
Dye**2** at 40◦C with dispersing	73.85	6.89	26.89	30.57	27.76	75.63

Table II. Shows the colorimetric data of dyed polyester with dye **1** and **2** at 80 ˚C with and without dispersing agents using Ultrasonic techniques

ID	L*	a*	b*	dE*	C*	h
Dye**1** without dispersing	74.52	5.38	24.96	28.24	25.53	77.84
Dye**1** with dispersing	81.37	1.76	13.11	14.21	13.11	82.35
Dye**2** without dispersing	81.30	0.91	11.02	12.19	11.06	85.26
Dye**2** with dispersing	78.60	3.70	13.51	16.14	14.01	74.68

Table III. indicate the fastness properties of dyed polyester at 60 ˚C the dye **1, 2** without dispersing agent at I.R technique and dye **1**, **2** with and without dispersing agents at 80 ˚C using Ultrasonic techniques

Dyes	**techniques**	**Washing fastness**			**Perspiration fastness**						**Fastness to Rubbing**		**Light**
					Acidic			**Alkaline**					
		SC	**SW**	**Alt**	**SC**	**SW**	**Alt**	**SC**	**SW**	**Alt**	**Dry**	**Wet**	
1	**IR**	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
Without agents	**Ultrasonic**	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4
1	**IR**	4	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
With agents	**Ultrasonic**	4	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4
2	**IR**	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
Without agents	**Ultrasonic**	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4
2	**IR**	4	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
With agents	**Ultrasonic**	4	4-	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4

SC, staining on cotton**; SW,** staining on wool**; Alt,** color change of the dyed sample

Figures 4,5 and 6 indicates the relation between wavelength and K/S of dyed sample with dye **1** and **2** with and without dispersing agents at 20 , 40 and 60 ˚C respectively in I.R technique . At 60 ˚C the dye **1** without dispersing agent gave K/S 7.56 but with dispersing it recorded 1.16 values. Also, the dye **2** without dispersing agent gave K/S 4.04 but with dispersing it recorded 1.37 values. From this data we find that the dye **1** gives higher K/S than dye **2** due to Prescence of OH group which cause highly dispersion of the dye on the surface of the fabric. Also, K/S increases by increase the temperature.

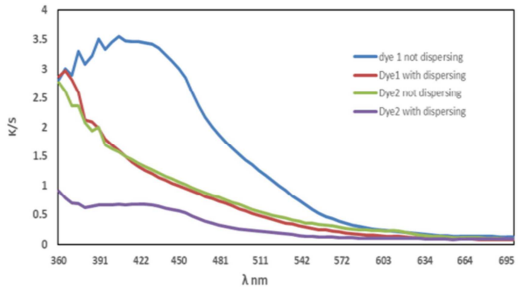


Figure 4: The relation between the wavelength and K/S of polyester fabrics with dye **1** and **2** at 20 ˚C in I.R technique

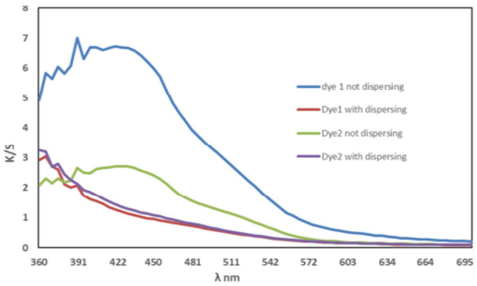


Figure 5: The relation between the wavelength and K/S of polyester fabric with dye **1** and **2** at 40 ˚C in I.R technique.

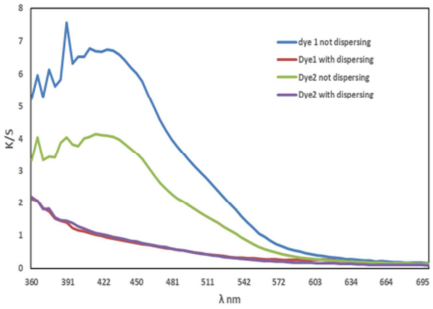


Figure 6: The relation between the wavelength and K/S of polyester fabric with dye **1** and **2** at 60 ˚C in I.R technique.

Figure 7 indicates the relation between wavelength and K/S of dyed sample with dye **1**and **2** with and without dispersing agents at 80 ˚C using Ultrasonic techniques indicates the relation between wavelength and K/S of dyed sample with dye **1** and **2** with and without dispersing agents at 80 ˚C respectively . The dye **1** without dispersing agent gave K/S 1.02 but with dispersing it recorded 0.47 values. Also, the dye **2** without dispersing agent gave K/S 0.59 but with dispersing it recorded 0.37 values.

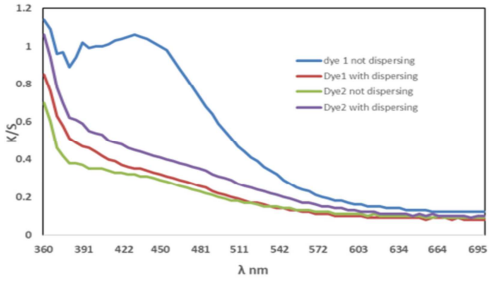


Figure 7: The relation between the wavelength and K/S of polyester fabric with dye **1** and **2** at 80 ˚C in ultrasonic technique.

4. Conclusion

Disperse dyes comprise between 30 and 60 percent dispersion agents, and perfect dyeing of polyester with disperse dyes requires between 2 and 5 g/L of dispersing agent. Dispersing agents don't get absorbed by fibres, therefore a lot of them provide an unavoidable load to the effluent treatment system. This study aims to synthesise and analyse two novel disperse dyes based on dye modification of curcumin. There are two methods for applying these dyes to polyester fabric: infrared (IR) procedures and ultrasonic techniques. In the absence of a mordant or a dispersion agent, all coloured materials displayed good colour strength, light fastness, and other fastness characteristics, protecting the environment from pollution.

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