



Diazenyl Chalcones Incorporating Thiazole moiety: Synthesis, Spectroscopic Characterizations, Dyeing Performance, and Antimicrobial Efficacy



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Abstract

Numerous polyfunctionalized heterocyclic compounds containing azo groups have been synthesized utilizing thiazolidine derivatives **1a-e** as precursors. Mass, NMR, and infrared spectra are among the spectrum analyses used to verify the structures of the generated molecules. Following the application of a variety of dyestuffs to polyester fabrics, the dyed samples showed suitable levels of light fastness, rubbing, washing, and thermal fixation fastness, as well as highly excellent sweat resistance. Fabrics dyed with the dyestuffs also offer adequate UV protection properties, according to the UPF measurements of the fabric samples **2-3a, b, d, e**. The dyed fabric has a higher UPF value (50+). Gram-positive and Gram-negative microorganisms, as well as *Candida albicans*, were tested for antimicrobial activity in the fabric samples. All evaluated fabrics demonstrated measurable antibacterial efficacy.

Keywords: Azo chalcones, heterocycles, Polyester fabric, antimicrobial activity.

1. Introduction

The global rise in melanoma [1] heightens concerns about public health. Melanoma, a kind of skin cancer, is caused by an abnormal transformation of melanocytes. Numerous studies investigating the impacts of UV radiation on various animals have been conducted, with humans serving as the principal subjects due to the robust association between UV exposure and skin cancer. Sunlight, and especially UV light, is toxic to all forms of life. Reddening, tanning, and a reduction in the synthesis of collagen and elastin, two macromolecules essential to the structure of living organisms, are all effects of ultraviolet light [2]. Textiles, as an effective broadband screen against solar radiation, have lately been the focus of educational endeavors. These advancements have highlighted the significance of using protective gear to mitigate the negative impacts of UV radiation. If more people become aware of these steps, it could significantly reduce the number of skin-related health problems [3]. A large family of chemicals with diverse applications in various scientific disciplines, azo-function-containing molecules are pigmented. The presence of azo groups in the primary structural components characterizes a class of organic dyes known as azochromophores. Their adaptability allows them to produce a wide range of colors, making them ideal for use in advanced manufacturing processes. Azo dyes share the characteristic of creating covalent bonds with textile substrates [4,5]. Azo dyes make up over 70% of the dye supply in the textile industry. Leather, plastic, food, cosmetics, printing, medicine, paint, lacquer, textiles, and a multitude of other products use azo dyes as colorants. Changing the coupling components from electron donors to electron-withdrawing azo donors allows you to modify the color [6,7]. Protons can be transferred within molecules through a process called azo/hydrazone tautomerism [8]. Azo compounds show stereoisomerism when light hits them. The trans isomer can undergo a transition to become the cis isomer. Some compounds can undergo isomerization independently under certain conditions. Several researchers have lately taken an interest in developing heterocycle-integrating dispersion azo dyes [9,10]. The colorfastness, strength, and brightness of azo compounds containing heterocycles are far better than those of benzene-based dyes. Consequently, these compounds have become very popular in the market for industrial dyes. Dyeing and printing are the two main methods of adding color to textiles. One great example of a new chromophore system is the use of heterocyclic dyes in the dyeing of polyamides and polyesters. A lot of azo-dispersing heterocyclic dyes are used when printing and dyeing polyester. They react at light speed, have the ideal dye thickness, and

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produce colors that pop [11,12]. Synthesis of azo dyes, particularly heterocycles and their derivatives, is attracting a lot of interest due to their chemical sensing capabilities and biological activities [13–15].

This work focuses on creating new chalcones with mono- and diazo-linkages that include a thiazole moiety. The novel azo compounds are subjected to additional examinations using spectroscopic methods. Their potential as dyes for polyester fabrics is examined, and their effectiveness against germs and fungi is evaluated.

2. Materials and methods

All melting point ranges were taken on a Gallen Kamp electric melting point apparatus using the one-end open capillary method and were uncorrected. The reactions progress was followed up by the TLC technique using silica gel G60 F (Merck) plates that were precoated to a thickness of 0.2 mm, visualizing the spots under ultraviolet light and dichloromethane/PE (1: 2) as mobile phases. The IR spectra of the compounds were recorded on the Bruker ALPHA II Fourier Transform Infrared Spectrometer at a wavenumber of (4000–400 cm^{-1}) (Central Lab, Faculty of Science/ A S U). The ^1H and ^{13}C -NMR spectra were recorded on a Bruker AV 300 and JEOL 500-MHz spectrometer operating at 300 and 500 MHz for ^1H and 75, 125 MHz for ^{13}C using dimethyl sulfoxide as solvent and tetramethyl silane (TMS) as an internal reference (Center for Drug Discovery Research and Development/ Faculty of Pharmacy/ A S U, and Central Laboratory & Service, N R C). Mass spectra (MS) were recorded on the Thermo Scientific GCMS model (ISQ LT) using Thermo X-Calibur software (Bioscience laboratory /Cairo Egypt).

2.1. Synthesis and characterization of the target compounds

2.1.1. Synthesis of 2-(4-substituted-phenyl) diazenyl)-2-hydroxybenzylidene) hydrazineylidene)-5-(2-hydroxybenzylidene) thiazolidin-4-one derivatives (2a-e).

A stirring solution of salicylaldehyde (0.01 mol) in ethanol was supplemented with an ethanolic solution of **1a-e** (0.01 mol) combined with 10% NaOH and 10 mL of DMSO. Petroleum ether/ethyl acetate thin-layer chromatography (TLC) in a ratio of 2:1 allowed for the monitoring of reaction progress. The reaction mixture was refluxed for six hours, filtered, washed with ether and ethanol, and recrystallised from an ethanol and water solution in a 1:1 ratio to produce the desired product, **2a-e**.

2-(2-Hydroxy-5-(phenyldiazenyl) benzylidene) hydrazineylidene) -5-(-2-hydroxybenzylidene) thiazolidin-4-one(2a).

Yellow crystals, yield 71%, m.p.257-260°C, FTIR (cm^{-1}): 3464 (OH), 3039, 2938 (CH) Ar, 2778 (CH) Al, 1687 (C=O), 1620 (C=N), 1594 (C=C), 1491 (C=C alkenyl), 1452 (N=N), 454 (OH, Enol); ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ (ppm): 10.72, 9.14 (s, 2H, 2OH), 8.72 (s, 1H, CH), 8.29 (s, 1H, CH=N), 7.91 (s, 1H, CH=C), 7.68-6.54 (m, 11H, Ar-H); MS (m/z, %): 442.40 (M^{-1} , 12.83).

2-(2-Hydroxy-5-(4-chlorophenyldiazenyl) benzylidene) hydrazine -ylidene)-5-(-2-hydroxy benzylidene) thiazolidin-4-one(2b).

Green crystals, yield 99%, m.p.279-280°C, FTIR (cm^{-1}): 3358(OH), 3254(NH), 2945(CH)Ar, 2776(CH)Al, 1712(C=O), 1635(C=N), 1599(C=C), 1505(C=C alkenyl), 1477(N=N), 451(OH, Enol); ^1H NMR (500 MHz, $\text{DMSO}-d_6$): δ (ppm): 12.58 (s, 1H, NHCO/ N=C-OH), 11.67 (s, 1H, NH), 11.44, 10.33 (s, 2H, OH), 8.77 (s, 1H, CH), 8.72 (d, 1H, CH), 8.60 (s, 1H, CH=N), 8.13 (s, 1H,CH=C),7.88-7.22 (m,9H, Ar-H) ; MS (m/z, %): 476.01 (M^{-1} , 37.61).

2-(2-Hydroxy-5-(4-bromophenyldiazenyl) benzylidene) hydrazineylidene)-5-(-2-hydroxy benzylidene) thiazolidin-4-one(2c).

Red crystals, yield 44%, m.p.238-240°C, FTIR (cm^{-1}): 3400(OH), 3168(NH), 3048-2944(CH)Ar, 2778(CH)Al, 1683(C=O), 1622(C=N), 1594(C=C), 1498(C=C alkenyl), 1455(N=N), 457(OH, Enol); ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ (ppm): 12.58 (s, 1H, NHCO / N=C-OH), 11.44 (s, 1H, NH), 10.65 (s, 1H, OH), 8.95 (s, 1H, CH), 8.76 (d, 1H, CH), 8.31(s, 1H, CH=N), 8.09 (s, 1H,CH=C),7.92-6.55 (m,9H, Ar-H), 4.55(s, 1H, OH) ; MS (m/z, %): 521.73 (M^+ , 15.08).

2-(2-Hydroxy-5-(4-tolyphenyldiazenyl) benzylidene) hydrazineylidene)-5-(-2-hydroxy benzylidene) thiazolidin-4-one(2d).

Pale yellow crystals, yield 73%, m.p.248-250°C, FTIR (cm^{-1}): 3430(OH), 3232(NH), 3056-2943(CH)Ar, 2779(CH)Al, 1681(C=O), 1636(C=N), 1595(C=C), 1482(C=C alkenyl), 1460(N=N), 456(OH, Enol); ^1H NMR (500 MHz, $\text{DMSO}-d_6$): δ (ppm): 12.58 (s, 1H, NHCO/N=C-OH), 10.64 10.34 (s, 2H, OH), 8.80 (s, 1H, CH), 8.69 (d, 1H, CH), 8.26(s, 1H, CH=N), 8.13 (s, 1H,CH=C),7.87-6.97 (m,9H, Ar-H), 2.36(s, 3H, CH_3) ; ^{13}C -NMR (125MHz, $\text{DMSO}-d_6$): δ (ppm): 168.05(C=O), 163.66 (C-O), 160.93 (C=N), 158.63 (CH=N), 158.04 (C-O), 150.51(C-N), 145.78(C-N), 145.38 (CH-chalcone), 141.88, 133.07, 130.48(2 X C),128.88,125.57, 123.14(2 X C), 122.93, 121.47, 119.31, 118.93, 117.92, 116.93, 116.63(C-Ar), 21.00(CH_3) ; MS (m/z, %): 556.45 (M^+ , 44.69).

2-(2-Hydroxy-5-(4-methoxyphenyldiazenyl) benzylidene) hydrazineylidene)-5-(-2-hydroxy benzylidene) thiazolidin-4-one(2e).

Green crystals, yield 99%, m.p.270-272°C, FTIR (cm^{-1}): 3400(OH), 3219(NH), 3056-2943(CH)Ar, 2776(CH)Al, 1681(C=O), 1636(C=N), 1597(C=C), 1502(C=C alkenyl), 1457(N=N), 458(OH, Enol); ^1H NMR (500 MHz, $\text{DMSO}-d_6$): δ (ppm): 12.58 (s,

1H, NHCO/N=C-OH), 10.63, 10.33 (s, 2H, OH), 8.69 (s, 1H, CH), 8.10 (d, 1H, CH), 7.88 (s, 1H, CH=C), 7.65-6.94 (m, 9H, Ar-H), 3.82 (s, 3H, OCH₃); ¹³C-NMR (125MHz, DMSO-d₆): δ ppm; 167.98(C=O), 163.31 (C=O), 162.28 (C=N), 159.35 (C-O), 158.41 (CH=N), 157.54 (C-O), 146.60(C-N), 145.42(C-N), 133.10 (CH- chalcone), 128.88, 125.40, 124.87(2 X C), 123.72, 123.10, 121.39, 120.98, 120.21(2 X C), 119.31, 116.93, 116.63, 115.14(C-Ar), 56.16(OCH₃); MS (m/z, %): 472.40 (M⁺, 18.40).

2.1.2. Synthesis of 5-(5-(4-Substituted phenyl) diazenyl)-2-hydroxy benzylidene)-2-(5-(4-substituted-phenyl) diazenyl)-2-hydroxybenzylidene) hydrazineylidene) thiazolidin-4-one derivatives (3a-e).

A solution of substituted aniline (0.01 mol) in hydrochloric acid (5 mL) and water (5 mL) was heated to 70°C. The clear solutions were added to an ice-water mixture, and diazotization was carried out at 0–5°C by adding sodium nitrite (0.05 mol) dissolved in 10 mL of water. The cold diazo solutions were added gradually to compounds **2a-e** (0.01 mol) solution in 50 mL of water, which included sodium hydroxide (0.005 mol) and sodium carbonate (0.03 mol). The reaction mixture was vigorously stirred for 30 minutes at 0°C during the addition process. The precipitated products were collected via filtration, washed with 250 mL of water and diethyl ether to obtain the pure products **3a-e**.

5-(5-(Phenyl) diazenyl)-2-hydroxybenzylidene)-2-(5-(phenyl) -diazenyl)-2-hydroxy benzylidene) hydrazineylidene) thiazolidin-4-one (3a).

Yellowish brown crystals, yield 96%, m.p.287-290°C, FTIR (cm⁻¹): 3465(OH), 3058-2959 (CH)Ar, 2780(CH)Al, 1660(C=O), 1627(C=N), 1589(C=C), 1447(N=N), 459(OH, Enol); ¹HNMR (500 MHz, DMSO-d₆): δ ppm; 11.45 (s, 1H, NH), 10.77, 10.34 (s, 2H, OH), 8.86 (s, 1H, CH), 8.74 (d, 1H, CH), 8.33(s, 1H, CH=N), 7.96 (s, 1H, CH=C), 7.91-7.17 (m, 13H, Ar-H); ¹³C-NMR (125MHz, DMSO-d₆): δ ppm; 163.85(C=O), 158.63(C-O), 152.44(2 X C), 145.61, 145.38, 133.27, 131.68, 131.48, 131.31, 130.72, 130.28, 129.98(4 X C), 124.32, 123.94, 123.59, 123.19, 122.91(4 X C), 120.20, 119.18, 118.98, 118.61, 117.00; MS (m/z, %): 547.48 (M⁺, 10.88).

5-(5-(4-Chlorophenyl) diazenyl)-2-hydroxybenzylidene)-2-(5-(4-chloro phenyl) diazenyl)-2-hydroxy benzylidene) hydrazineylidene)thiazolidin-4-one (3b).

Yellow crystals, yield 77%, m.p.187-190°C, FTIR (cm⁻¹): 3318(OH), 3053-2951(CH)Ar, 2752(CH)Al, 1669(C=O), 1620(C=N), 1575(C=C), 1478(N=N), 453(OH, Enol); ¹HNMR (500 MHz, DMSO-d₆): δ ppm; 12.53 (s, 1H, NHCO), 11.65 (s, 1H, NH), 10.32 (s, 1H, OH), 8.75 (s, 1H, CH), 8.60(s, 1H, CH=N), 8.17 (d, 1H, CH), 8.13 (s, 1H, CH=C), 7.88-6.96 (d, 4H, Ar-H), 7.58(s, 1H, CH), 7.43-6.96(m, 7H, Ar-H), 5.00(s, 1H, OH); MS (m/z, %): 616.33 (M⁺, 25.40).

5-(5-(4-Bromophenyl) diazenyl)-2-hydroxybenzylidene)-2-(5-(4-chloro phenyl) diazenyl)-2-hydroxy benzylidene) hydrazineylidene)thiazolidin-4-one (3c).

Dark red crystals, yield 79%, m.p.267-270°C, FTIR (cm⁻¹): 3456(OH), 3161(NH), 3070(CH)Ar, 2750(CH)Al, 1702(C=O), 1622(C=N), 1590(C=C), 1456(N=N), 461(OH, Enol); ¹HNMR (400 MHz, DMSO-d₆): δ ppm; 10.86(s, 1H, NH), 10.36 (s, 1H, OH), 8.74 (s, 2H, CH), 8.32 (s, 1H, CH=N), 8.19 – 8.05 (d, 4H, CH), 7.91 (s, 1H, CH=C), 7.79 (d, 1H, CH), 7.76 (d, 4H, CH), 7.68 (s, 1H, CH), 7.19 (d, 1H, CH), 7.05 (d, 1H, CH), 5.21 (s, 1H, OH); ¹³C-NMR (100MHz, DMSO-d₆): δ ppm; 174.42(C=O), 164.53(2 X C-O), 161.09(C=N), 158.61(C=NH), 151.34(2 X C), 145.40, 145.08(2 X C), 133.01, 132.95, 132.86, 130.23(4 X C), 126.58, 125.55, 124.88, 124.75, 123.58, 123.23, 120.95, 120.19, 119.20, 118.43, 118.61, 116.85; MS (m/z, %): 704.91(M⁺, 34.11), 705.67 (M⁺, 37.20).

5-(5-(4-Tolyldiazenyl)-2-hydroxybenzylidene)-2-(5-(4-chlorophenyl) diazenyl)-2-hydroxy benzylidene) hydrazineylidene) thiazolidin-4-one(3d).

Reddish brown crystals, yield 99%, m.p.238-240°C, FTIR (cm⁻¹): 3487(OH), 3029-2947(CH)Ar, 2787(CH)Al, 1713(C=O), 1628(C=N), 1599(C=C), 1458(N=N), 460(OH, Enol); ¹HNMR (500 MHz, DMSO-d₆): δ ppm; 11.45 (s, 1H, NH), 10.90, 10.33 (s, 2H, OH), 8.83 (s, 1H, CH), 8.72 (d, 1H, CH), 8.33(s, 1H, CH=N), 8.29-8.28(m, 4H, Ar-H), 8.18 (s, 1H, CH=C), 8.13(d, 1H, CH), 7.80-7.15 (m, 7H, Ar-H), 2.36(s, 6H, 2CH₃); ¹³C-NMR (125MHz, DMSO-d₆): δ ppm; 163.67(C=O), 157.99(C-O), 157.32(C-O), 156.22(C=N), 150.42(2 X C), 147.85, 146.59, 145.24(2 X C), 143.6, 135.32, 130.39(4 X C), 127.25, 126.73, 126.54, 125.89, 124.04, 123.64, 123.05, 122.84(4 X C), 118.88; MS (m/z, %): 775.82 (M⁺, 48.41).

5-(5-(4-Methoxyphenyl) diazenyl)-2-hydroxybenzylidene)-2-(5-(4-chloro phenyl) diazenyl)-2-hydroxy benzylidene) hydrazineylidene) thiazolidin-4-one(3e).

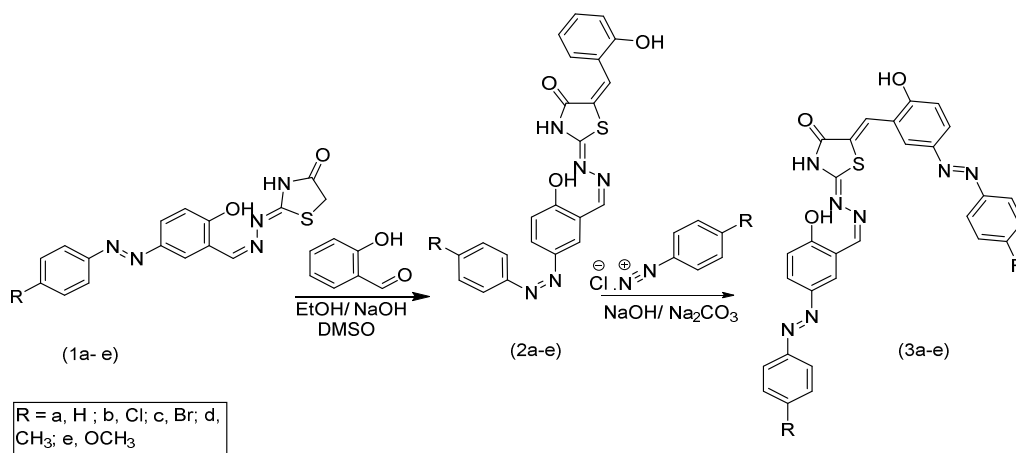
Brown crystal, yield 86%, m.p.393-294°C, FTIR (cm⁻¹): 3480(OH), 3061-2946(CH)Ar, 2837(CH)Al, 1709(C=O), 1624(C=N), 1596(C=C), 1503(N=N), 458(OH, Enol); ¹HNMR (500 MHz, DMSO-d₆): δ ppm; 12.62 (s, 1H, NHCO), 11.33 (s, 1H, NH), 10.82, 10.36 (s, 2H, OH), 8.75 (d, 1H, CH), 8.13 (s, 1H, CH), 8.05(s, 1H, CH=N), 7.98 (s, 1H, CH=C), 7.91-6.96 (m, 12H, CH), 3.89(s, 3H, OCH₃), 3.85(s, 3H, OCH₃); MS (m/z, %): 607.13 (M⁺, 19.13).

3. Results and Discussion

3.1. Chemistry

It seems that molecular hybridization is a good way to make new structures with better biological activity since it combines the active parts of many different substances. The chemical sequences that were employed to synthesize a novel class of chalcones linked by mono- and diazo bonds are shown in Scheme 1. A series of compounds, designated as 2-(4-substituted-phenyl) diazenyl)-2-hydroxybenzylidene) hydrazineylidene) thiazolidin-4-one derivatives (**1a-e**), were synthesized using the methods described according to [16–18]. These compounds underwent condensation by reacting with salicylaldehyde in ethanol and sodium hydroxide. The process was further demonstrated to produce 5-benzylidenethiazol-4(5*H*)-ones **2a-e** [19–23]. Use of infrared, hydrogen nuclear magnetic resonance, carbon nuclear magnetic resonance, and mass spectroscopy allowed for the confirmation of the structures of the newly synthesized chalcones **2a-e**. The 5-arylidene derivatives **2a-e** were found to have a *Z*-configuration, as shown by the NMR signal for the C=CH methine proton. This signal was a single peak at a higher value between 7.88 and 8.13 ppm, and another peak around 12.58 ppm, which was attributed to the keto-enol tautomerism (NHCO/N=C-OH) of the thiazolone ring. The absence of a signal for the methylene protons of the thiazolidinone component also lend credence to the proposed structure.

The reaction involved chalcones **2a-e** being combined with diazonium salts of aryl amines (aniline, *p*-chloroaniline, *p*-methyl aniline, and *p*-methoxy aniline) in modest quantities of ethanol, NaOH, and Na₂CO₃ while being stirred uniformly [14]. This resulted in the formation of diazenyl chalcones, each containing two azo linkages **3a-e**. Scheme (1) delineates the preparation technique. ¹H-NMR, ¹³C-NMR, and mass spectrometry confirms the structures of the target derivatives. The ¹H-NMR proton of compounds **3a-e** revealed two singlets observed at about 11.65 and 10.82 ppm and at 10.36 and 10.32 ppm, corresponding to the hydroxyl protons of salicylaldehyde rings, while a multiplet signal was detected between 7.92 and 6.69 ppm, comprising aromatic protons.



Scheme 1: Route strategy for the synthesis of chalcones derivatives (**2a-e**), and (**3a-e**).

Dyeing process and dye-fabric interaction

In this study, the relationship between the fabric and the dyeing process was discussed. Scrubbed polyester fabrics from Egypt's El-Mahalla El-Kobra Company were treated with 2% of eight dyestuffs **2a, b, d, e** and **3a, b, d, e** using the high temperature (HT) method. Using the IR environmental infrared textile dyeing process, a 4-gram sample of polyester fabric was exposed to a pH of 4 and 130°C for 60 minutes. A liquor-to-fabric-weight ratio of 1:50 was used, with a dye concentration of 2%. Sodium bisulphite and sodium hydroxide were dissolved in water in a 1:40 liquor-to-goods ratio. After rinsing the fabrics in warm water, they were reduced-cleared at 60°C for 10 minutes. The sample was rinsed with water after the reduction process. After a five-minute neutralization with 1 g/l of acetic acid, the dyed materials were rinsed in cold water. After that, they were washed under running water and allowed to air dry at room temperature [24]. Pale yellow to dark yellow hues were attained.

Color measurements

This study examines the colorimetric properties of polyester fabrics dyed with azo dyes **2-3a, b, d, e**, measured in the CIE Lab* color space using a Hunter Lab DP-9000 Color-Spectrophotometer with pulsed xenon lamps as the light source. The instrument was designed with a 10° observer, D65 illuminate, d/2 viewing geometry, and a 2 mm measurement area. Sourced from Hunter Lab, USA. The λ values at which each measurement was taken varied. The comparable color strength value (K/S) was calculated using Kubelka-Munk [25].

In the CIE Lab* color space, the total difference CIE (L^* , a^* , b^*) was measured between two colors each given in terms of L^* , a^* , b^* is calculated from.

$$\Delta E = L^*_2 + (a^*_2 + b^*_2)^{1/2}$$

where ΔE is the total difference between the sample and the standard, L^* the lightness from black (0) to white (100), a^* is a red (+)/green (–) ratio and b^* is yellow (+)/blue (–) ratio. L^* value indicates lightness, (+) if sample is lighter than standard, (–) if darker. a^* & b^* values: indicate the relative positions in CIE Lab space of the sample and the standard, from which some indication of the nature of the difference can be seen [26].

Results for the dyed fabrics are shown in Table 1, which shows that the lightness levels vary according to the type of substituents, L^* , ranging from 80.69 (dye **2b**, 4-Cl) to 74.39 (dye **3d**, 4-CH₃). The polyester textiles displayed brighter colors with dyes **2b**, **d**, and **e** ($R = 4\text{-Cl}$, 4-CH₃ and OCH₃) and **3b** ($R = 4\text{-Cl}$), in contrast to the dyes **3d**, **a**, and **e** ($R = 4\text{-CH}_3$, H, and OCH₃), which had the lowest L^* values and indicated deeper shades (Figure 1). a^* values ranging from -4.41 to -0.76 were observed for most of the dyes, **2a-e** with one azo-linkage and **3a, b, e** with two azo-linkages ($R = \text{H}$, Cl, CH₃, OCH₃); a^* value of 3.31 was observed for dye **3d** with two azo-linkages (4-CH₃).

On the other hand, the K/S values are largely adequate, and Table 1 shows that most of the dyes have a strong affinity for polyester materials. The presence of substitutions in the chromophore explains the variation in color intensity. For instance, the color strength of diazenyl chalcones (**2b**, **2d**, and **2e**) with monoazo linkage was higher, so the results were better compared to the **2a** dye. This decrease may be a result of the presence of electron-withdrawing groups (-Cl, **2b**) and electron-donating groups (CH₃ and OCH₃, **2d** and **2e**) substituents. A hypsochromic shift, brought about by electron withdrawal in nature, causes a reduction in color intensity. Dye **3a**, which does not contain any substitution, displays a stronger hue than dyes **3b**, **d**, and **3e**, which have two azo linkages. Also, from the results, the intensity of color may decrease with the increasing of the number of azo groups **2b-e** or the number of substituent groups in comparison with dyes **3b-e**.

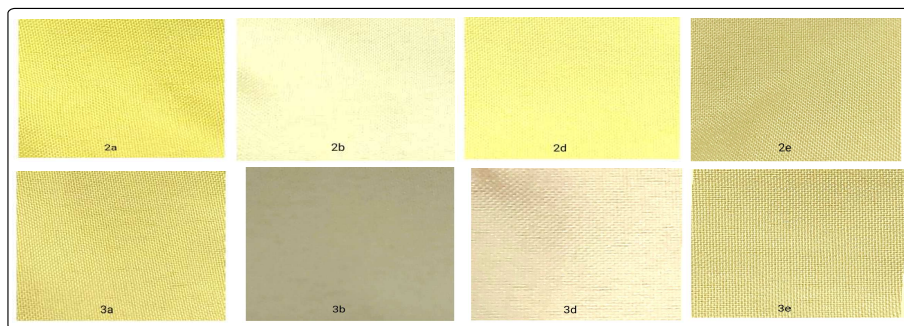


Figure 1: Color of dyeing on polyester fabrics.

Table 1: The intensity of the coloration achieved on the fabrics and the colorimetric data in the CIE Lab space

Dyes	R	Max λ	K/S	L^*	a^*	b^*	ΔE
2a	H	360	1.58	78.41	-3.80	29.87	39.12
2b	Cl	360	8.64	80.69	-2.99	13.57	22.81
2d	CH ₃	360	8.55	79.79	-4.41	27.35	36.61
2e	OCH ₃	365	7.93	77.38	-3.81	23.60	33.18
3a	H	365	6.23	77.14	-0.76	21.33	30.58
3b	Cl	360	4.69	79.61	-1.17	14.32	23.35
3d	CH ₃	365	7.53	74.39	3.31	23.15	32.20
3e	OCH ₃	360	4.88	77.32	-1.45	20.90	30.20

Color-staple assays

The fastness properties of dyed textiles are crucial factors that determine the quality, durability, and commercial viability of the dyeing process. In this study, we evaluated the fastness properties of polyester fabrics dyed with a series of novel azo dyes **2-3a, b, d, e**, as summarized in Table 2. The fastness properties assessed include wash fastness, perspiration fastness (acidic and alkaline on cotton, wool, and polyester, and light fastness with the standard technique [27]. These properties were evaluated using standard techniques and rated on a scale from 1 (poor) to 5 (excellent) with light fastness rated on a separate scale from 1 to 7.

Table 2: Fastness characteristics of fabrics dyed with azo dyestuff (**2 -3a, b, d, e**).

Dyed fabrics	R	Rubbing		Washing		Perspiration				Sublimation				light			
		Dry	wet	St.	st.*	St.**	alt.	Acid		Alkaline		At 180 °C		At 210°C			
								St.	st.*	St**	alt.	St.	st.*	St**	alt.		
2a	H	4	4	4	4	4	4	4	4	4	4	4	4-3	4-3	4-3	4-3	6
2b	Cl	4	4	4	4	4-	4	4	4	4	4	4	4-3	4-3	4-3	4-3	6
2d	CH ₃	4	4	4	4	4	4	4-	4	4	4	4	4-3	4-3	4-3	4-3	5-6
2e	OCH ₃	4	4	4	4	4	4	4	4	4	4	4	4-3	4-3	4-3	4-3	6
3a	H	4	4	4	4	4	4	4	4	4	4	4	4-3	4-3	4-3	4-3	6
3b	Cl	4	-4	4	4	4	4	4	4	4	4	4	4-3	4-3	4-3	4-3	6
3d	CH ₃	4-3-	3-4	4	4	4	4	4	4	4	4	4	4-3	4-3	4-3	4-3	6
3e	OCH ₃	4	4	4	4	4	4	4	4	4	4	4	4-3	4-3	4-3	4-3	6

• Staining on cotton, wool, and polyester is referred to as St., St.*, and St.**, respectively.

• A light fastness rating from 1 (poor), 2 (slight), 3 (moderate), 4 (fair), 5 (good), 6 (very good), and 7 (excellent).

• Priority of wash and sublimation: 1 for poor, 2 for fair, 3 for outstanding, 4 for very acceptable, and 5 for exceptional.

Washability and color retention

The color fastness of polyester to washing was determined using the ISO 105-C02 technique [28]. The typical procedure involved sewing together pieces of bleached wool and cotton fabric and then immersing the resulting composite specimens in a water solution containing 5 g/l of non-ionic detergents in a 1:50 liquor ratio. Once the bath was heated to 50°C, which took around 30 minutes, the samples were removed, rinsed twice using intermittent hand squeezing, and then dried. To test how quickly the wash dried, we employed a color-changing greyscale.

Color durability during rubbing

The ISO 105-X12 test method was used to assess the rubbing resistance of polyester [29]. The goal of this experiment is to determine how much color can be transferred from one surface to another by rubbing differently colored fibers.

A test for dry crocking

The crock meter's base was used to lay the specimen flat for testing. A white calibration cloth rested on top of the mount. The covered finger was dropped onto the specimen and then spun around ten times at a speed of one turn per second, causing it to slide twenty times. The white test sample was then removed and stained in greyscale for analysis.

Wet crocking experiment

The white sample was withheld from further testing after being immersed in water up to a 65% level. Just like the last dry crocking test, this one was also conducted. The white samples for testing were allowed to air dry before analysis.

Resistance of dyes to perspiration

Two solutions of synthetic sweat were prepared in accordance with the criteria laid out by ISO 105-E04 (1989). In the first step, we create an acidic solution by combining 0.5 g of L-histidine monohydrochloride monohydrate, 5 g of sodium chloride, and 2.2 g of sodium dihydrogen phosphate in 1 liter of distilled water. The next step was to add 0.1 N NaOH until the pH reached 5.5. In 1 liter of distilled water, 0.5 g of L-histidine monohydrochloride monohydrate, 5 g of sodium chloride, and 2.5 g of disodium hydrogen phosphate were diluted to make the alkaline solution. We used 0.1 N NaOH to get the pH up to 8 to create a composite specimen; the colored specimen, which had dimensions of 5 x 4 cm, was stitched onto two sections of the uncolored specimens. Soaking the composite specimen in solutions of various acids and bases for 15–30 minutes while it was vigorously shaken and compressed was the next step in the fastness test. d. The samples that were going to be tested were placed in an oven set at 37°C (±2°C) for four hours with a force of about 4-5 kg given to every plate. To show how this affected the color of the object, the word "greyscale" was used.

Color fastness to light

Following the test method ISO 105-B02 (1988), the lightfastness test was conducted under continuous exposure to a carbon arc lamp for 35 hours. The impact on the colors of the examined objects was analyzed with a blue scale for color variation [30].

Sublimation fastness

The purpose of sublimation fastness is to determine the color of different kinds of garment materials, as well as their capacity to withstand high temperatures and retain color when subjected to heat pressure or heat drum processing. Dry pressing: In a pressure apparatus set to 180 and 210°C, the dry specimen is exposed to a predetermined temperature and pressure for 30 seconds. Following testing, the specimen's color is assessed using a grey sample card [31]. **Table 2** demonstrates that all dyes had positive outcomes. High washing and perspiration fastness were anticipated, and these colors are often hydrophobic in nature.

Evaluation of the UPF values of the synthetic dyes.

The synthesized dyes were checked using UPF measurements. The more effective the colored fabric is at blocking UV light, the higher the UPF rating. A computerized spectrophotometer (3101PC) that can detect both ultraviolet light and visible light was used to find the UPF values. The sphere had fabrics with wavelengths ranging from 290 nm to 400 nm, with each wavelength spaced 10 nm apart. The UV-penetration capabilities of the fabrics were assessed in the 290-400 nm range using a method for assessing light absorption and protection. The textiles were permitted to cure at NTP for a full day prior to the measurements being taken. Four scans of the fabric sample were averaged at 10-nanometre intervals from 290 nm to collect the spectral data. Four photos were obtained by rotating the sample 90 degrees at regular intervals throughout the testing. The software determines the UPF rating of a flat [32], tensionless, dry fabric using the UPF value calculation equation.

$$UPF = \frac{ED}{ED_m} = \frac{\int_{290\text{ nm}}^{400\text{ nm}} E_{\lambda} S_{\lambda} \Delta\lambda}{\int_{290\text{ nm}}^{400\text{ nm}} E_{\lambda} S_{\lambda} T_{\lambda} \Delta\lambda}$$

Where: (UPF)-ultraviolet protection factor value through fabrics, E_{λ} -relative erythral spectral effectiveness ($\text{W/m}^2 \text{ nm}^{-1}$), S_{λ} -solar spectral irradiance (Melbourne), $\Delta\lambda$ -measured wavelength interval (nm), and T_{λ} -spectral transmittance of the sample (%). The percentage blocking of UVA range (315-400 nm) and UVB range (290 -315nm) was calculated from the transmittance data.

UV radiation exposure links to numerous skin issues, including sunburns, skin cancers, premature ageing, and immune system suppression [24]. There is proof that UV light reduces the effectiveness of the immune system. The UPF is frequently used for textiles and clothing since it is the most common and natural method of shielding the human body from the elements. The three primary defenses against the damaging effects of UV radiation are: wearing protective clothes, applying sunscreen, and limiting sun exposure. Ultraviolet Protection Factor is what the UPF stands for. It is defined as the ratio of the average UV irradiance for skin protected by the test fabric to the average effective UV irradiance computed for skin that is not protected [33]. The polyester fabric's UV protection factor results for samples **2-3a, b, d, and e** are shown in Table 3. With a score between 123.2 and 78.1, dyed polyester has a favorable UPF rating. This indicates that the synthetic dyes **2-3a, b, d, e** offer superior UV protection for the textile material.

Table 3: UV Protection Grade.

Sample	UPF Range	Protection category	UV-B 290-315 λ_{max}	Transmittance % UV-B	UV-A 315-400 λ_{max}	Transmittance % UV-A
2a	114.7	50+ Excellent	254.7	9.79	249.1	3.11
2b	78.1	50+ Excellent	176.8	6.8	172.8	2.01
2d	88.1	50+ Excellent	188.5	7.25	183.6	2.29
2e	115.1	50+ Excellent	255.6	9.83	249.4	3.11
3a	123.2	50+ Excellent	277.2	10.66	108.1	1.35
3b	93.9	50+ Excellent	194.5	7.48	190.2	2.37
3d	91.3	50+ Excellent	209.1	8.04	203.7	2.54
3e	106.2	50+ Excellent	223.4	8.59	218.3	2.72

UV-B^a Transmittance (mean transmittance percentage in the range (290–315 nm))

UV-A^b Transmittance (mean transmittance percentage in the range (315–400 nm))

3.2. Screening Antimicrobial Activity

Antimicrobial testing was carried out using an agar well diffusion experiment. The *Bacillus subtilis* (ATCC 6633), *Staph. Aureus* (ATCC 6538), *K. pneumonia* (ATCC13883), *Salmonella typhi* (ATCC 6539), and *Candida albicans* (ATCC 10221) were incubated at 37 °C for 24 h and every test was repeated 3 times. As positive controls, standard solutions Gentamicin and Fluconazole were chosen as an antibacterial agent and as an antifungal agent, respectively. The recorded results for each tested sample were represented as the average diameter of inhibition zones (IZ) of bacterial or fungal growth around the discs in mm [33,34].

The antibacterial properties of four fabric samples **2-3b, d** are demonstrated against a common yeast and both Gram-positive and Gram-negative bacteria [table. 4, fig. 2]. It is very desirable for textiles used in medical or hygienic applications to have this wide range of action. With frequently higher antibacterial activity than the control antibiotics/antifungals, Fabrics **2d** and **3b** are consistently the most effective. This effectiveness is especially remarkable because the controls are well-known for therapeutic purposes. When tested against *B. subtilis*, all four cloth samples exhibit strong antibacterial action. Notably, the inhibition of fabric **2d** (27±1 mm) and Fabric **3b** (28±1 mm) is stronger than that of the Gentamicin control (25±2 mm). The result indicates that these textiles are highly successful in stopping the spread of this gram-positive bacteria. Every fabric exhibits outstanding anti-*S. aureus* action. Fabrics **2d** (31±1 mm) and **3b** (33±1 mm) in particular exhibit better antibacterial properties than the Gentamicin control (30±1 mm). Strong anti-staphylococcal fabrics could be very helpful in hospital or personal hygiene settings, and *S. aureus* is a common source of skin and soft tissue infections, so our finding is a significant result. The outer membrane of gram-negative bacteria, such as *K. pneumoniae*, frequently makes them more difficult to deal with. Although the activity of fabrics **3d**, **2b**, and **2d** is similar to that of the Gentamicin control, fabric **3b** (25±1 mm) exhibits noticeably more inhibition against *K. pneumoniae* than the control (21±2 mm). This suggests that fabric **3b** has a high potential for controlling resistant Gram-negative bacteria. Like *K. pneumoniae*, fabrics **2d** and **3b** have greater action against *S. typhi* (25±1 mm and 25±2 mm, respectively) in comparison to the Gentamicin control (22±1 mm). The activity of fabrics **3d** and **2b** is likely similar to or slightly less than that of the control. This indicates that fabrics **2d** and **3b** are effective against additional significant Gram-negative bacteria. Furthermore, compared to the common antifungal Fluconazole (27±1 mm), fabric **2d** (31±1 mm) and fabric **3b** (29±1 mm) show stronger antifungal action against *Candida albicans*. This conclusion implies that these textiles might be very successful in stopping the growth of fungi, which is advantageous for textiles used in settings where fungus contamination is a common problem.

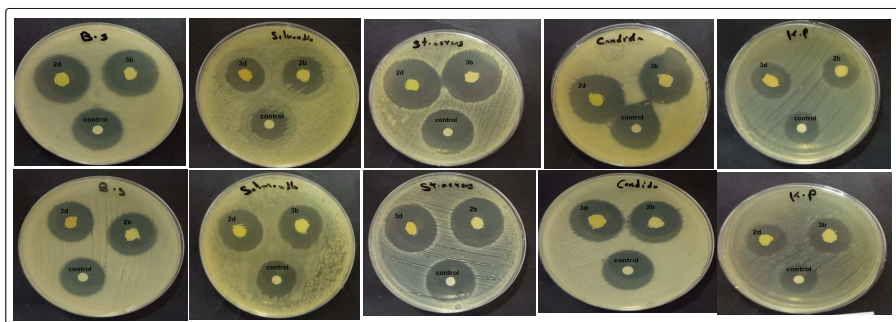


Figure 2: The anti-microbial activity of the fabric samples (2-3b, d).

Table 4: Antimicrobial Activity for fabric samples (2-3b, d).

Samples / Control drugs	Microorganism				
	<i>Bacillus subtilis</i> (ATCC 6633)	<i>Staph. Aureus</i> (ATCC 6538)	<i>K. pneumonia</i> (ATCC13883)	<i>Salmonella typhi</i> (ATCC 6539)	<i>Candida albicans</i> (ATCC 10221)
2b	25±1	30±1	20±2	22±1	25±1
2d	27±1	31±1	22±2	25±1	31±1
3b	28±1	33±1	25±1	25±2	29±1
3d	24±1	29±1	21±1	20±2	25±1
Gentamicin	25±2	30±1	21±2	22±1	-
Fluconazole	-	-	-	-	27±1

*** Inhibitions zones were represented as mm.

4. Conclusion

Based on 2-(4-substituted-phenyl) diazenyl-2-hydroxybenzylidene) hydrazineylidene) thiazolidin-4-one derivatives (**1a-e**), the synthesized monoazo and diazo dispersion dyes were made. Applying them to polyester fabric allowed us to study their different characteristics. For polyester fibers, the dyes showed good coloring properties. On the polyester materials, every dye showed excellent dyeing performance. Depending on how many electron-donating or electron-withdrawing groups were

connected to the chromophore component, the dyed polyester fabrics displayed a spectrum of pale to dark yellow hues. The dyed samples showed strong sweat fastness and excellent fastness levels to rubbing, washing, heat fixation, and light exposure. On the other hand, all samples showed excellent antifungal activity against *Candida albicans* in comparison to the standard drug fluconazole and noteworthy antibacterial activity against *Bacillus subtilis*, *Staphylococcus aureus*, and *Klebsiella pneumoniae* in comparison to the standard drug gentamicin. Compounds **2d** and **3b** showed exceptional antibacterial and antifungal activity, according to the results of the biological activities of the investigated compounds.

Conflicts of interest

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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Data availability

All data generated or analysed during this study are included in this published article as Supplementary material and supplementary information files.

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