New tricyanofuran intramolecular charge transfer dyes comprising the hydrazone group were prepared and fully characterized in order to study their possible solvatochromism, dyeing ability, and antimicrobial activity. The preparation of the hydrazone dyes was achieved in relatively good yields starting from different aromatic amines. The hydrazone functional group was presented via the azo-coupling reaction of the tricyanofuran compound by the properly substituted diazonium chloride. Chemical structures of the prepared hydrazones were confirmed via nuclear magnetic resonance spectroscopy (\(^1\)H- and \(^13\)C-NMR), Fourier-transform infrared spectroscopy (FT-IR), and elemental analysis (C, H, and N). The UV-visible absorption spectra of the produced sensor colorants displayed interesting solvatochromism in solvents with a different polarity, which was found to be affected by the substituents bonded to the aromatic hydrazone moiety. The pH molecular switching was investigated through tuning the intramolecular charge transfer stimulated by the reversible deprotonation/protonation process in acetonitrile solution showing color change from yellow to purple. The produced disperse dyestuffs were employed for dyeing polyester fibers to introduce acceptable color strength and colorfastness properties. Moreover, the dyes verified a weak to moderate antimicrobial activity against some selected pathogens, including \(S.\ aureus\), \(E.\ coli\), and \(Candida\ albicans\).

**1. Introduction**

Solvatochromic materials attract much interest due to their potential applicability as sensors in determining solvent polarity, optical light-emitting diodes, dye sensitized solar cells, and electro- and photoluminescent materials for laser purposes, in molecular electronics for the production of molecular switches, colorimetric chemosensors in detection of explosives, and volatile organic materials [1–8]. Generally, a solvatochromic material can be defined as a chemical substance able to alter its color in solvents of different polarities due to a variation in its absorption or emission spectra in each solvent. There is a variety of solvatochromic dyes that have been discovered recently, such as pyridinium, merocyanine, and stilbazolium dyes [9–15].

Hydrazones are originally a Schiff-base group of materials possessing a secondary amine proton comprising an important category of compounds in a diversity of research fields, such as nonlinear optics, sensors, and a variety of biological and medical applications [16–26]. Thus, many researchers have prepared hydrazone-based materials as main goal molecular structures and investigated their antimicrobial activities. The widespread use of antimicrobial agents results in the growth of strongly resistant pathogens.
Therefore, there has been an important concern in the improvement of diverse inventive collection of pharmacological agents [27–33]. These observations emphasize the necessity for the preparation of novel hydrazones that gain diverse biological activities. Arylhydrazones are characterized by their simple preparation and worth considering in the design of novel molecular switches with solvatochromic and pH sensing performance. A hydrazone functional group has the ability to operate as a bridge in a donor-acceptor molecular structure, or it can act itself as an electron donor moiety, when it is in conjugation with an electron-withdrawing moiety [34–39].

Herein, we present the synthesis, characterization, photophysical properties, dyeing behavior, and antimicrobial assessment of novel tricyanofuran-hydrazone dyes 1–3, in which a number of electron-withdrawing and electron-donating substituents were introduced at the ortho-, meta-, and para-positions of the aromatic moiety of the diazonium chloride. The molecular structures of the novel hydrazone dyes are presented in Figure 1. The molecular switching character of the hydrazone dye was also explored.

2. Experimental

2.1. Materials and Methods. Melting points were measured uncorrected in degree Celsius using Stuart SMP30. FT-IR spectra were investigated by Fourier-transform infrared spectrophotometer (Nexus 670, Nicolet, United States) in the range of 400–4000 cm⁻¹ with a spectral resolution of 4.0 cm⁻¹. Mass spectra were reported on a Shimadzu GCMS-QP 1000 EX mass spectrometer at 70 eV. Elemental analyses (C, H, and N) were studied using PerkinElmer 2400 analyzer (Norwalk, United States). UV-visible absorption spectra were determined at ambient conditions using UNICAM UV-visible 300. Nuclear magnetic resonance (NMR) spectra were measured on a Bruker AVANCE 400 spectrometer at 400 MHz; chemical shift was given in ppm relatively to an internal standard TMS at 295 K. The color strength was examined using UltraScan PRO Spectrophotometer (light source D65/10° observer). The pH values were reported using a Beckman Coulter pH 340 meter with a combined glass-calomel electrode.

Solvents were purchased from Aldrich and Fluka for both of the preparation procedures of dyes and spectroscopic studies (spectroscopic grade). All reactions were observed using Merck aluminum thin layer chromatography plates precoated with silica gel PF254 (20 × 20 mm, 0.25 mm) and monitored by naked eye under an ultraviolet lamp (254 or 365 nm). The tricyanofuran moiety and tricyanofuran hydrazone dyestuffs were prepared according to previously reported literature [26]. A 100% polyester fabric (149 g/m²) was supplied by Misr El-Mahalla Spinning and Weaving Company, El-Mahalla El-Kubra, Egypt. The fabric was scoured according to the literature procedure [40, 41].

2.2. Synthesis of Dyestuffs

2.2.1. General Procedure for the Synthesis of the Hydrazone Dyes 1–3. A mixture of the appropriate substituted arylamine (1.0 mmol) and hydrochloric acid (5.0 mL) in 25 mL glass beaker was stirred on a magnetic stirrer and cooled to 0–5°C in an ice bath. An aqueous solution of sodium nitrite (1.0 mmol) in water (3.0 mL) was then added slowly. The produced diazonium salt was stirred for an extra 30 minutes at 0–5°C. In a separate glass beaker, tricyanofuran (1.0 mmol) was dissolved in acetonitrile (5.0 mL) and cooled to 0–5°C, and sodium acetate (1.0 g) was added. The produced mixture was vigorously stirred in the ice bath at 0–5°C, while the cold diazonium salt solution was added dropwise. After stirring for an extra two hours, the crude product was filtered off under vacuum, washed with distilled water (3 × 5 mL), recrystallized, and air-dried.

(1) 2-(3-Cyano-5,5-dimethyl-4-((2,4-dinitro-5-fluorophenylhydrazono)methyl)furan-2(5H)-ylidene)malononitrile (1). It is prepared from 2,4-dinitro-5-fluoroaniline (200 mg, 1.0 mmol) and tricyanofuran (200 mg, 1.0 mmol). The product was filtered off under vacuum, recrystallized from ethanol, and finally air-dried to give a red solid (260 mg; yield 63%). mp 226–228°C; 1H-NMR (400 MHz, DMSO-d₆) δ (ppm): 12.31 (s, broad, 1H, NH), 8.59 (s, broad, 1H aliphatic, C=H), 8.44 (d, J = 8.0 Hz, 1H aromatic), 7.61 (d, J = 8.0 Hz, 1H aromatic), and 1.78 (s, 6H aliphatic). 13C-NMR (400 MHz, DMSO-d₆) δ (ppm): 177.54, 172.92, 158.50, 136.97, 125.05, 118.45, 115.74, 113.61, 113.76, 112.12, 99.83, 98.00, 55.41, 26.73, and 23.91. IR (neat, cm⁻¹): 3287 (NH), 2206 (CN), 1586 (C=N), and 1507 (NO₂). MS m/z (%): 410 [M+H]+. Elemental analysis calculated for C₁₇H₁₀FN₇O₅ (411.07): C 49.64, H 2.45, F 4.62, and N 23.84; found: C 49.51, H 2.37, F 4.62, and N 23.96.

(2) 2-(3-Cyano-5,5-dimethyl-4-((4-pyridylhydrazono)methyl)furan-2(5H)-ylidene)malononitrile (2). It is prepared from 4-aminopyridine (94 mg, 1.0 mmol) and tricyanofuran (200 mg, 1.0 mmol). The product was filtered off under vacuum, recrystallized from ethanol, and finally air-dried to give a yellow solid (260 mg; yield 63%). mp 218–220°C; 1H-NMR (400 MHz, DMSO-d₆) δ (ppm): 12.82 (s, broad, 1H, NH), 8.57 (d, J = 8.0 Hz, 2H aromatic), 8.31 (s, 1H aliphatic, C=H), 7.48 (d, J = 4.0 Hz, 2H aromatic), and 1.84 (s, 6H aliphatic). 13C-NMR (400 MHz, DMSO-d₆) δ (ppm): 176.91, 171.27, 156.37, 134.89, 132.14, 114.01, 113.28, 112.69, 110.71, 99.41, 56.77, 26.91, and 23.65. IR (neat, cm⁻¹): 3248 (NH), 2223 (CN), and 1575 (C=N). MS m/z (%): 303 [M+H]+. Elemental analysis calculated for C₁₇H₁₂FN₄O₄ (411.07): C 63.15, H 3.97, and N 27.62; found: C 63.28, H 4.09, and N 27.51.

(3) 2-(3-Cyano-5,5-dimethyl-4-((4-nonylphenylhydrazono)methyl)furan-2(5H)-ylidene)malononitrile (3). It is prepared from 4-nonylaniline (220 mg, 1.0 mmol) and tricyanofuran (200 mg, 1.0 mmol). The product was filtered off under vacuum, recrystallized from ethanol, and finally air-dried to give an orange solid (370 mg; yield 86%). mp 214–216°C; 1H-NMR (400 MHz, CDCl₃) δ (ppm): 10.02 (s, broad, 1H, NH), 7.67 (d, J = 8.0 Hz, 2H aromatic), 7.36 (d, J = 8.0 Hz, 2H aromatic), 7.29 (s, 1H aliphatic, C=H), 2.73 (t, J = 3.2 Hz, 2H aliphatic), 1.81 (s, 6H aliphatic), 1.34 (s, 14H aliphatic), and 0.92 (t, J = 2.2 Hz, 3H aliphatic). 13C-NMR (400 MHz, CDCl₃)
2.3. Dyeing Procedure. The dyeing process was applied to the polyester fabric using the high-temperature dyeing technique according to the literature procedure [4]. The dyeing process was carried out using the high-temperature high-pressure coloration technique according to the literature procedure [4]. A dispersion of the dyestuff was prepared by dissolving the proper amount of dye (2 wt.% relative to the weight of fabric) in 1 mL DMF and then added gradually with stirring to the dye bath (liquor ratio 50 : 1) in presence of sodium lignin sulfonate as a dispersing agent (2 wt.% relative to the weight of fabric). The pH of the dye bath was customized to 4.85 using an aqueous solution of acetic acid, and the wetted-out polyester fabric was then added. The dye-bath temperature was maintained at 130°C for 180 minutes under pressure in an infrared dyeing apparatus. The fabric was then washed and subjected to the reduction clearing process at 80°C for 45 minutes in an apparatus. The fabric was then rinsed in cold water and neutralized by an aqueous solution of acetic acid (1 g/L) for 5 minutes at 40°C, followed by rinsing in tap water and allowed to be air-dried. The determination of the dye uptake into polyester fibers was measured applying the absorption process depending on the Beer-Lambert law. This was evaluated by the testing sample from dye bath at different periods of time (15, 40, 55, 75, 90, 105, 120, 135, 150, 160, 170, and 180 minutes) while running the dyeing process, according to the previously reported techniques [4, 7].

2.4. Colorfastness Properties. Colorfastness to perspiration (ISO105-E04:1989), washing (ISO105-C02:1989), rubbing (ISO105-X12:1987), and light (ISO105-B02:1988) was tested according to AATCC standard test methods. Colorfastness to light was assessed employing the blue scale (1–8; where 1 is very poor and 8 is excellent), while colorfastness to washing, perspiration, and rubbing was evaluated according to the grey scale (1–5; where 1 is very poor and 5 is excellent) [7].

2.5. Color Strength Measurement. The color strength ($K/S$) of the dyed polyester samples was assessed by applying the high reflectance technique using the Kobelka–Munk equation $[K/S = (1 - R)^2/2R - (1 - R_0)^2/2R_o]$, where $S$ is the scattering coefficient, $K$ is the absorption coefficient, and $(R, R_o)$ are decimal fractions of the reflectance of both dyed and undyed polyester samples, respectively [7].

2.6. Antimicrobial Evaluation. The antimicrobial testing of the obtained hydrazone dyes was performed against $S. aureus$ (Gram positive), $E. coli$ (Gram negative), and $Candida albicans$ (fungus). The antimicrobial examination was made quantitatively using the standard AATCC microbial count test procedure 100–1999 [42–44].

3. Results and Discussion

3.1. Synthesis of Hydrazone Dyestuffs. The highly electron-deficient tricyanofuran moiety was prepared according to a previously described literature procedure by applying Knoevenagel condensation between 3-methylacetoin and malononitrile in presence of absolute ethanol as a solvent and sodium ethoxide as a strong base [26]. Knoevenagel condensation reaction is an interesting reaction for the synthesis of electron-deficient substances, such as the highly electron-deficient oxygen-containing tricyanofuran heterocycle. The strongly electron-withdrawing CN groups on the tricyanofuran moiety are useful for the stabilization of the tricyanofuran carbanion obtained by proton abstraction from the active methyl substituent in presence on sodium acetate as a weak base [2]. The tricyanofuran carbanion was then subjected to azo coupling by the appropriate aryldiazonium salt to produce the consequent unstable azo dyes that were converted directly into the stable hydrazone dyes 1–3 as shown in Scheme 1.

The molecular structures of prepared hydrazone dyes were verified according to their spectroscopic data. FT-IR spectra displayed absorption peaks at 3287, 3248, and 3386 cm$^{-1}$ which are assigned to the hydrazone NH group, whilst the peaks at 1586, 1575, and 1577 cm$^{-1}$ are due to C= N stretch of the hydrazone functional group for dyes 1, 2, and 3, respectively. The $^1$H-NMR spectra showed singlet peaks at 12.31, 12.82, and 10.02 ppm which are assigned to the proton of the hydrazone NH group of dyes 1, 2, and 3, respectively. The $^1$H-NMR spectra of dyes 1–3 displayed also a singlet peak around 8.59, 8.31, and 7.29 ppm, respectively.

![Figure 1: Hydrazone dyestuffs 1-3.](image-url)
due to the aliphatic vinyl proton (\textunderscore C-H). The downfield shifting of such singlet signal is a result of the strong impact of the electron-withdrawing tricyanofuran moiety.

3.2. Solvatochromic Measurements. The UV-visible absorption bands were monitored in the wavelengths ranges 455–540, 475–497, and 475–485 nm for dyes 1, 2, and 3, respectively (Table 1 and Figure 2). They exhibited colors between yellow and purple in various pure solvents of different polarities. The type of substituents on the aryl-hydrazone moiety was found to affect on the UV-visible absorption maximum band. A distinctive solvatochromic behavior of all dyestuffs in both of protic and aprotic solvents was monitored. Protic environment was anticipated to exhibit partial protonation to the hydrazone dyestuff via hydrogen bonding of the OH proton of the protic solvent to the lone pair of electrons on the hydrazone NH functional group [2]. This leads to reduced charge on the hydrazone NH group to result in the hypsochromic shift. In both of nonpolar and aprotic environment, nonetheless, this phenomenon was negligible and we had to change our thought to other contributions for salvation, such as the dipolar nature of solvents. Solvatochromism monitored in our hydrazone dyes arises from changes in the contribution degree of the lone pair of electrons on the nitrogen atom of the NH functional group, which can partially function as a bridge among partially electron-rich hydrazone donor and highly electron-deficient tricyanofuran acceptor fragment in a semi-donor-acceptor molecular system. This can simply be described as an extended inductive effect as demonstrated in Scheme 2. This results in an interesting positive solvatochromic behavior owing to the generated partial extended conjugation [4]. Therefore, the polarizability due to the donor or acceptor substituents on the arylhydrazone moiety certainly influences the solvatochromic performance of the prepared hydrazone dyes.

<table>
<thead>
<tr>
<th>Solvent</th>
<th>λ_{max} (nm)</th>
<th>1</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methanol</td>
<td>525</td>
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<td>480</td>
<td>480</td>
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<td>Ethanol</td>
<td>530</td>
<td>480</td>
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<tr>
<td>DMSO</td>
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<td>497</td>
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</tr>
<tr>
<td>DMF</td>
<td>538</td>
<td>485</td>
<td>480</td>
<td></td>
</tr>
<tr>
<td>Acetonitrile</td>
<td>455</td>
<td>480</td>
<td>475</td>
<td></td>
</tr>
<tr>
<td>Dichloromethane</td>
<td>455</td>
<td>485</td>
<td>485</td>
<td></td>
</tr>
<tr>
<td>Tetrahydrofuran</td>
<td>455</td>
<td>485</td>
<td>485</td>
<td></td>
</tr>
<tr>
<td>1,4-Dioxane</td>
<td>455</td>
<td>475</td>
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</tr>
<tr>
<td>Toluene</td>
<td>460</td>
<td>485</td>
<td>485</td>
<td></td>
</tr>
</tbody>
</table>

3.3. Assessment of pH Sensing Effect. The electron-withdrawing functional group on arylhydrazone generates an acidic NH hydrazone proton able to create a conjugate base, or so called an arylhydrazone anion, with an electron-donating ability. This arylhydrazone anion acts as an electron-donating moiety in conjugation with the strong electron-deficient tricyanofuran moiety leading to an interesting spectral switch of a donor-acceptor molecular system under pH stimulus [24]. Figure 3 displays the UV-visible absorption spectra and color changes of dye 1 dissolved in acetonitrile (ca. 2.3×10^{-5} mol·L^{-1}), under the deprotonation and protonation reversible process. Upon addition of 1.0 mol·L^{-1} of methanolic solution of tetrabutylammonium hydroxide (TBAH) to compound 1 dissolved in acetonitrile to raise the pH value, the maximum absorption wavelength at 451 nm was bathochromically shifted to 538 nm. On the contrary, the addition of 1.0 mol·L^{-1} methanolic solution of trifluoroacetic acid (TFAA) resulted in the maximum absorption band at 451 nm to reappear, while reducing the pH value.

The existence of an isosbestic point at 489 nm verifies that there are two different molecular species: hydrazone and hydrazone anion that coexist together in equilibrium with...
each others and that the spectral changes are attributed to the acidity of the hydrazone NH. Considering this hydrazone NH prevented resonance among arylhydrazone and tricyanofuran, this proposes that a negatively charged arylhydrazone anion was formed in alkaline environment leading to a considerable increment of the internal charge transfer that can be described as an extended resonance effect (Scheme 3).

To inspect the reversibility and stability of such pH sensing changes, methanolic solutions (1.0 mol·L⁻¹) of TBAH and TFAA were employed to exchange the pH among ~6.62 and ~6.93. The ratios of UV-visible absorption values at 451 nm (~0.8327) and at 538 nm (~0.6650) of compound 1 were recorded, and the results are displayed in Figure 4. It was obvious that this procedure was highly reversible demonstrating that compound 1 was stable at different pH values.

3.4. Evaluation of Dyes Uptake. Polyester fabrics are described by their high affinity toward disperse colorants. The prepared hydrazone colorants are characterized by their nonionic small molecular structures with low solubility in the aqueous medium. Thus, they can be described as disperse dyestuffs with the capability to be applied on hydrophobic fabrics, such as polyester. The prepared dyes 1–3 were applied

Figure 2: UV-visible absorption spectra of dyestuffs 1 (a), 2 (b), and 3 (c) in solvents of different polarities.
by the high-temperature and -pressure approach at 130 °C on polyester samples at 2% shade to afford dyed polyester substrates from yellow, orange, and orange-red. All dyes demonstrated excellent uptake into polyester substrates as displayed in Figure 5. This fact is an indication of their high penetrations through the polyester chains. In addition, the high dye uptake can be attributed to the fabric affinity as a result of the small and planar molecular structures of the prepared hydrazone colorants. Thus, the dyed polyester fabrics showed excellent colorfastness to washing and rubbing. The shades, color strength, and colorfastness properties of dyestuffs 1–3 are displayed in Table 2. The colorfastness against light of the prepared dyes 1–3 was found to rely on the substituent type on the hydrazone moiety. These substituents are able to change the electron density over the entire molecular structure of the dye. Thus, all dyes displayed good
colorfastness to light except for dye 1, which contains highly electron-withdrawing nitrosubstituents. On the contrary, dye 1 showed a better color strength than dyes 2 and 3, which is in agreement with electron-withdrawing nitrosubstituents with the ability to increase the depth of color on dye 1. The shades of the dyed polyester substrates were in accordance with the monitored absorption maximum wavelength of the prepared dyes in solution.

3.5. Antimicrobial Activity. The produced dyestuffs 1–3 were independently examined against *Escherichia coli*, *Staphylococcus aureus*, and *Candida albicans*, using the plate agar counting as the standard procedure. The antimicrobial reduction percentages stimulated by the prepared dyes are summarized in Table 3. Dyestuff 1, comprising higher electron-withdrawing substituents, demonstrated low inhibition effect on the reduction percentages due to poor antimicrobial resistance, while dyestuffs 2 and 3 with electron-donating substituents showed moderate antimicrobial resistance.

4. Conclusions

Some novel tricyanofuran hydrazone derivatives were prepared and applied on polyester fibers as disperse dyes with a variety of substituents at ortho-, meta-, and para-positions of the arylhydrazone moiety. They were prepared using a simple approach via azo coupling of the tricyanofuran starting material with the appropriate diazonium
The molecular structures of dyes were verified by 1H-NMR and 13C-NMR spectra, elemental analysis, and FT-IR spectra. A positive solvatochromism was recorded by UV-visible absorption spectra in a variety of solvents with different polarities. The pH molecular switching effect, accompanied by reversible color changes, was monitored by UV-visible spectra as a result of the variation of the pH value leading to the production of charge delocalization on the hydrazone dye resulting in extended conjugation through a quinoid form. This stimulated planarity of hydrazone dye resulted from the electron-withdrawing substituents on the arylhydrazone moiety producing higher conjugation extent of the hydrazone anion dye than that of the hydrazone dye. Thus, the pH sensory is displayed via modulating the intramolecular charge transfer changed by deprotonation/protonation processing. The prepared dyestuffs were applied on polyester substrates by the high-temperature and-pressure technique to give good depths of shades from yellow, orange, and orange-red. The studied dyestuffs displayed mostly satisfactory colorfastness properties. We also explored the application of the hydrazone dyes as potential substances with antimicrobial efficiency against some pathogenic species. Dyes comprising electron-donating groups on the arylhydrazone moiety demonstrated moderate antimicrobial activities, while dyes with highly electron-withdrawing groups on the arylhydrazone displayed weaker antimicrobial activities.

**Data Availability**

Chemdraw files (Figure 1 and Schemes 1–3), graphical abstract (png. file), and origin files (Figures 2(a)–5) are available.

**Additional Points**

Novel solvatochromic hydrazone disperse dyes were prepared. Colorimetric sensory behavior to detect variations in pH was recorded. Acceptable colorfastness was presented upon dyeing polyester fabric. Satisfactory antimicrobial activity of the hydrazone dye was monitored.

**Conflicts of Interest**

The authors declare that there are no conflicts of interest regarding the publication of this paper.

**Acknowledgments**

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**Supplementary Materials**

Novel solvatochromic and pH sensory molecular switching tricyanofuran hydrazone disperse dyes with antimicrobial activity were prepared to introduce a satisfactory colorfastness on polyester fabric. (Supplementary Materials)

**References**


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**Table 2: Colorfastness, shade, and tinctorial strength of dyed polyester substrates.**

<table>
<thead>
<tr>
<th>Dyes</th>
<th>Wash</th>
<th>Perspiration</th>
<th>Basic</th>
<th>Rubbing</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>Alt.*</td>
<td>St.*</td>
<td>Acidic</td>
<td>Basic</td>
</tr>
<tr>
<td>1</td>
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</tr>
<tr>
<td>3</td>
<td>4-5</td>
<td>4-5</td>
<td>4-5</td>
<td>4-5</td>
</tr>
</tbody>
</table>

*Alt. = alteration in color; St. = staining on cotton.*

**Table 3: Antimicrobial properties of dyestuffs 1–3.**

<table>
<thead>
<tr>
<th>Dyes</th>
<th>Antibacterial (bacterial reduction %)</th>
<th>Antifungal (fungal reduction %)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>E. coli</td>
<td>S. aureus</td>
</tr>
<tr>
<td>1</td>
<td>9 ± 1.3</td>
<td>10 ± 1.4</td>
</tr>
<tr>
<td>2</td>
<td>12 ± 1.6</td>
<td>17 ± 1.8</td>
</tr>
<tr>
<td>3</td>
<td>19 ± 1.1</td>
<td>25 ± 1.5</td>
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</table>


