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# 1. Introduction

Any electro-optical device that utilizes organic dyes requires that they be optimized in a variety of ways relevant to the particular technical application. The immediate applications we are interested in here, including photovoltaics, organic lasers, optical storage and nonlinear optical devices are no exception.<sup>1-8</sup> A pervasive requirement that applies to dyes in virtually any application is stability (photochemical and thermochemical). An exception, of course, is in the area of optical storage wherein dyes may be designed and utilized which are inherently and intentionally photochemically and/or thermochemically labile.<sup>9-12</sup> As such, a very general goal of our effort entails the design and synthesis of dyes that are optimized to provide the appropriate stability required for the specific application. In addition to stability, in the case of photovoltaic devices the extinction coefficients of the dyes are also an important issue as this is a fundamental component in the light energy to electric energy conversion efficiency.<sup>13,14</sup> Therefore, another goal of our effort is to identify and prepare dyes with

# Synthesis of high molar extinction coefficient push-pull tricyanofuran-based disperse dyes: Biological activity and dyeing performance\*

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Several push-pull tricyanofuran-based disperse dyes with high molar extinction coefficient were synthesized and fully characterized. The dyes were prepared *via* Knoevenagel reaction in dry pyridine under ambient conditions employing acetic acid as catalyst. The condensation process was carried out between the reactive methyl-bearing tricyanofuran moiety and tertiary amine-based arylaldehyde. The molecular structures of the synthesized chromophores were proved by Fourier transform infrared spectroscopy (FT-IR), nuclear magnetic resonance spectroscopy (<sup>1</sup>H and <sup>13</sup>C NMR) and elemental analysis (C, H, N). The UV-Vis absorbance spectra of the synthesized dyes demonstrated interesting extinction coefficient values, which were found to be influenced by the aromatic-vinyl bridge and the tertiary amine donor group bonded to this aromatic-vinyl bridge moiety. The prepared tricyanofuran-based disperse colorants were dyed on polyester fabrics to afford satisfactory results. The substituents attached to the tertiary amine moiety influenced the absorption maxima. The fastness properties of the dyed polyester samples were measured. The biological activities of the synthesized dyestuffs against fungus (*Candida Albican*) as well as Gram-positive (*S. Aureus*) and Gram-negative (*E. Coli*) pathogens were also evaluated.

strong and narrow absorbance that are suitable to evaluate physical mechanisms which are proposed to enhance absorption in cavity configurations. The experimental processes and configurations needed to create the cavity devices also create independent demands on the materials that must be dealt with. Organic dyes studied for organic laser applications often suffer from instability. Studies here attempt to take an already stable class of dyes and further enhance their thermal stability and photostability.<sup>15–20</sup>

Chromophores comprised of tricyanofuran (TCF) acceptors and dialkylamine donors coupled by  $\pi$ -conjugated electron linkers have found extensive applications in electro-optics, photorefractives and more recently in single molecule and super-resolution biological imaging.8,21,22 The TCF chromophores developed for these particular applications were pursued due to their demonstrated superior stabilities relative to numerous other structural options and therefore they are prime candidates for extension into other areas, such as for photovoltaics and organic lasers.<sup>23-25</sup> Herein, we report the design, synthesis, and chemical characterization and intial photophysical characterization which concentrate on TCF dyes intended for use in photovoltaic devices, organic lasers and 3D optical data storage. For the photovoltaic studies chromophores with very large extinction coefficients and narrow bandwidths are required and we find that dyes with Ar = phenyl or thiophene

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**Fig. 1** General representative structure of TCF-based chromophores [donor (tertiary amine)-aromatic- $\pi$ -bridge acceptor (TCF)] with Ar = phenyl or thiophene based  $\pi$ -conjugated units. These dipolar push-pull chromophores represent a broad category of organic materials studied for their optical and electronic properties wherein Ar, n, R<sub>1</sub>, R<sub>2</sub>, R<sub>3</sub>, and R<sub>4</sub> are adjusted to tune the ancillary physical properties of the chromophore.

and n = 1 provide nearly the desired function (such systems are the most cyanine like with an intense absorption concentrated in a narrow wavelength range) as shown in Fig. 1. Further modifications thus far in R<sub>1</sub> and R<sub>2</sub> have aided processing requirements and further modifications of these groups are intended to further enhance both photochemical and thermochemical stability. Additional functionality may be attached at R<sub>1-4</sub> such as groups containing hindered amines or phenols which may function in stabilization capacity.

# 2. Experimental

#### 2.1. Materials and methods

Melting points (°C) were recorded uncorrected employing Stuart-SMP30. Fourier-transform infrared (FT-IR) spectra were studied by Nexus-670 (Nicolet, USA). The elemental analysis (C, H, N) was explored employing PerkinElmer-2400 (Norwalk, USA). UV-Vis absorbance spectra were reported UNICAM-300. NMR spectra were determined under ambient conditions utilizing BRUKER-AVANCE-400 at 400 MHz. Solvents (spectroscopic grade) were obatined from Fluka and Aldrich. The progress of reactions was monitored by Merck aluminum TLC PF254 under an UN lamp (254 or 365 nm). The TCF moiety was synthesized according to previously described procedure.<sup>26</sup> Polyester fabrics (100%; 149 g m<sup>-2</sup>) were collected from the local market in Saudi Arabia. The polyester substrates were scoured according to literature procedure.<sup>27</sup>

#### 2.2. Dyeing process

The high temperature dyeing method<sup>28</sup> was applied on the scoured polyester fabrics employing an infra-red dyeing machine. The dye (2 wt% relative to the fabric weight) was dissolved in acetone (1 mL) and then added to the dye-bath (stainless steel cup) with a 50:1 liquor ratio. Dispersing agent (sodium lignin sulfonate; 2 wt% relative to the fabric weight) was added to dye-bath. The pH was adjusted to  $\sim$  5.0 employing an aqueous solution of acetic acid. The polyester fabric (50:1 liquor ratio) was added to the dye-bath which was then closed, heated up and maintained at 130 °C for 3 hours under pressure. The polyester substrates were washed and exposed to reduction clear process at 80 °C for 45 minutes in an aqueous solution of sodium hydrosulphite (Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>; 2 wt% relative to the fabric weight) and sodium hydroxide (NaOH; 2 wt% relative to the fabric weight), followed by washing by nonionic detergent (2 wt% relative to the fabric weight). The polyester samples were rinsed with tap water, neutralized with acetic acid  $(1 \text{ g L}^{-1})$ diluted in tap water for 5 minutes at 40 °C, rinsed with tap water, and finally air-dried. The dye-uptake into fibers was determined using Beer-Lambert law. This was assessed by investigating samples from the dye-bath at different time intervals (20, 40, 60, 80, 100, 120, 140, 160 and 180 minutes) during the dyeing process.<sup>29</sup>

#### 2.3. Colorfastness properties

The fastness against washing (ISO-105-C02:1989), perspiration (ISO-105-E04:1989), rubbing (ISO-105-X12:1987), and light (ISO-105-B02:1988) was examined using AATCC standard tests. The fastness against light was evaluated under the blue-scale from 1 for very poor to 8 for excellent, while fastness against perspiration,



Scheme 1 Synthesis of 3, 4 and 5 TH-TCF-X-V (X = 5, 6 and 8); A one-pot synthetic procedure has been developed for the preparation of chromophores containing thiophene-vinyl in the  $\pi$ -system. Successful synthesis for the thiophene derivatives required formation of the imine as a discrete intermediate.



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Scheme 2 Synthesis of phenyl vinyl-bridged TCF chromophores with a hydroxyl terminal group.

washing and rubbing was evaluated under the grey-scale from 1 for very poor to 5 for excellent.

#### 2.4. Color strength

The color strength (*K*/*S*) was evaluated by Ultra-Scan-PRO spectrometer (light source D65/10 $^{\circ}$  observer).

#### 2.5. Antimicrobial activity

The antimicrobial performance of the synthesized chromophores was evaluated against *Candida Albican* (fungus), *E. Coli* (Gramnegative) and *S. Aureus* (Gram-positive). The antimicrobial activity was examined quantitatively employing AATCC 100-1999 standard microbial counting procedure.

# 3. Results and discussion

### 3.1. Synthesis and characterization

Different approaches have been pursued to design and develop optimized chromophores for the relevant associated applications including photovoltaic devices, organic lasers and 3D optical data storage. The strong electron withdrawing TCF (tricyanofuran) moiety was integrated into a variety of conjugated materials for optoelectronic applications. This oxygen-containing heterocylic intermediate was prepared in a 57% yield according to an early literature method *via* condensation process of malononitrile with 3-hydroxy-3-methylbutan-2-one using ethanol as a solvent and sodium ethoxide as a strong base. The TCF-based chromophores employed in this study were synthesized in different procedure as shown in Schemes 1–6.



Scheme 3 Synthesis of thiophene vinyl-bridged TCF chromophores with a hydroxyl terminal group.



Scheme 4 Synthesis of phenyl vinyl ester-bridged TCF bischromophores.

**3.1.1. One-pot synthesis of thiophene vinyl-bridged TCF chromophores.** This thiophene vinyl-bridged TCF chromophores 6.3, 6.4 and 6.5 (TH-TCF-X-V; X = 5, 6, and 8) contain a vinyl-bridge between TCF moiety and thiophene ring as shown in Scheme 1. In a one-pot synthetic approach with a 28 to 48% overall yield (the yield increases with increasing the alkyl chain length of the secondary amine), 2-bromo-5-formylthiophene and dialkylamine were employed as starting materials followed by condensation with 4-chloroaniline to afford the corresponding imine. Siegrist reaction was performed between this highly reactive imine electrophile and TCF head at room temperature. Siegrist reaction was employed due to the low reactivity of 2-alkylamino-5-formylthiophene.

**3.1.2.** Synthesis of phenyl and thiophene vinyl-bridged TCF chromophores with a hydroxyl terminal group. Phenyl-vinyl and thiophene vinyl-bridged TCF chromophores were prepared directly *via* condensation employing Knoevenagel reaction in dry pyridine under ambient conditions with acetic

acid as catalyst. The reaction was occurred between the active methyl-bearing TCF head and tertiary cyclic amine based arylaldehyde. A higher reaction rate and higher yields were observed for tertiary cyclic amine based aryldehyde that have displayed a high electrophilic reactivity compared to tertiary acyclic amine based arylaldehyde. The 4-(4-hydroxypiperidin-1yl)benzaldehyde displayed a high yield for the final chromophores compared to 2-alkylamino-5-formylthiophene. The 4-(4-hydroxypiperidin-1-yl)benzaldehyde was prepared from a mixture of DMSO and water as shown in Scheme 2. On the other hand, 2-alkylamino-5-formylthiophene was prepared from a mixture of triethylamine and water as shown in Scheme 3. The benzene bridged chromophores **8** and **9** were prepared in high yield of 81-87%; while thiophene the bridged chromophores **12** and **13** were obtained in a low yield of 18–21%.

**3.1.3.** Synthesis of phenyl vinyl-bridged TCF bis-chromophores. Chromophores 8 and 9 reacted with aliphatic dicarboxylic acid 14 *via* an esterification reaction (Steglich reaction) that was performed in dichloromethane employing DCC and DMAP as shown in Scheme 4. Bis-chromophores 15 and 16 were obtained in low yields in the range of 33–39%. Bis-chromophore 18 was prepared employing Knoevenagel reaction in dry pyridine at 40 °C with acetic acid catalyst as shown in Scheme 5. The reaction was performed between the active methyl-containing TCF head and 1,4-bis(4-benzaldehyde)piperazine 17 to afford the corresponding bis-chromophore 18 in a high yield of 92%.

3.1.4. Synthesis of phenyl-bridged TCF chromophores with a 2° amine terminal group. For the synthesis of phenyl bridged TCF chromophore 21 in which the TCF moiety is directly connected to a benzene ring without a vinyl-bridge, compound 2-(dicyanomethylene)-4-(4-fluorophenyl)-2,5-dihydro-5,5-19. dimethylfuran-3-carbonitrile, can be considered as a vital intermediate for a variety of TCF chromophores without a vinyl-bridge. It was found that the intermediate 19 easily reacts with a variety of unhindered secondary amines 20 (added in excess) via a very facile aromatic nucleophilic substitution reaction in dry pydridine at room temperature to afford the corresponding TCF chromophores 21 without a vinyl-bridge as shown in Scheme 6. The phenyl bridged TCF chromophore 21 was prepared in 63% yield at room temperature. The high reactivity of compound 19 results from the activating TCF fragment bearing three electron withdrawing cyano groups.



Scheme 5 Synthesis of phenyl vinyl peprazine-bridged TCF bis-chromophores.



Scheme 6 Synthesis of phenyl bridged TCF chromophores with a 2° amine terminal group.

Compound **19** was previously reported by our research group, *via* efficient condensation reaction of aromatic  $\alpha$ hydroxyketone, 1-(4-fluorophenyl)-2-hydroxy-2-methylpropan-1one, with malononitrile at room temperature using acetic acid catalyst in pyridine. The aromatic  $\alpha$ -hydroxyketone can be synthesized from trimethylsilyl protected 2-hydroxy-2methylpropanenitrile and Grignard reagent of 1-bromo-4fluorobenzene.

#### 3.2. Photophysical properties

In the past,  $\pi$ -conjugated linear push-pull chromophores have been prepared to afford new electroactive architectures for bulk heterojunction solar cells. However, weak acceptors such as malononitrile moieties have been generally utilized, rather than strong and more conjugated electron acceptors such as TCF that has been broadly studied in push-pull molecular systems of second order nonlinear dyes. Consequently, those strong acceptors are linked to electron donating moieties by aromatic/heteroaromatic  $\pi$ -bridges such as thiophenes, fusedthiophenes, and oligothiophenes, increasing the extension coefficient and absorption wavelength to the red wavelengths due to strong intramolecular interaction of the strong electron acceptors and donors.

The molar extinction coefficient of a dye is an important issue related to the power conversion efficiency (PCE) of organic photovoltaic devices. In this work, we report the results of an investigation on the molecular design, preparation, spectral, and photophysical properties of novel TCF dyes, with and without a vinyl-bridge, with strong and narrow absorbance suitable to evaluate physical mechanisms which are proposed to enhance absorption in cavity configurations. We designed and synthesized a series of novel push–pull chromophores *via* Knoevenagel reaction with formyl (hetero)aromatic donating moieties matched with TCF as a very strong electron acceptor. These TCF-based small dyes have afforded potential advantages for nonlinear optics, three dimensional optical data storage and solar cells, as a result of their extremely high absorption coefficient, polarizability, high dipole moments and simple



Fig. 2 TCF-based chromophores with very large molar extinction coefficients;  $\varepsilon_{max}$  (TH-TCF-8V **5** in CH<sub>3</sub>CN) = 216 150 L mol<sup>-1</sup> cm<sup>-1</sup>.

tunability of their energy levels employing a variety of donating substituents and  $\pi$ -bridges. It was found that introduction of a thiophene-vinyl as  $\pi$ -bridge into the conjugation path in TCF-based chromophores provided the extremely high molar extinction coefficients required for the photovoltaics studies (Table 1 and Fig. 2).

From our prior experience we know that generally these TCF chromophores have substantially improved properties relevant to single molecule optical imaging, especially in terms of their photochemical stability. The UV-Vis absorbance established that the coupling of TCF with electron donor fragments resulted in an improved intramolecular charge transfer leading to long wavelength absorption (Fig. 3).

Overall, the structural variations of the TCF chromophores including different aliphatic chains on the donor amine group display a little influence on the absorption spectra. The thiophene bridged chromophores display much sharper absorption

Table 1 TCF-based chromophores and their photophysical properties; the dyes with di-*n*-alkyl tails (5 V; **3**, 6 V; **4** and 8 V; **5**) have particularly large extinction coefficients

Chromophore	Molecular formula (g mol <sup>-1</sup> )	Melting point (°C)	$\lambda_{\max}$ (nm) in CH <sub>2</sub> Cl <sub>2</sub>	ε <sub>max</sub> in CH <sub>2</sub> Cl <sub>2</sub>
TH-TCF-5-V 3	C <sub>26</sub> H <sub>32</sub> N <sub>4</sub> OS; 448.62	214-216	624	166 787
TH-TCF-6-V 4	C <sub>28</sub> H <sub>36</sub> N <sub>4</sub> OS; 476.67	163-165	622	161 908
TH-TCF-8-V 5	$C_{32}H_{44}N_4OS; 532.78$	148-149	624	194 024
TCF-5Cy3MOH-V 9	$C_{24}H_{24}N_4O_2$ ; 400.47	245-247	579	61 200
TH-TCF-5Cy4OH-V 12	$C_{21}H_{20}N_4O_2S$ ;392.47	> 300	621	94 191
TCF-6Cy3NH 21	$C_{21}H_{21}N_5O; 359.42$	208-211	496	87 645

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Fig. 3 Normalized UV-Vis absorbance and fluorescence spectra of TH-TCF-8-V 5.

peaks at longer wavelengths with higher extinction coefficient values compared the phenyl bridged analogs. The UV-Vis absorption spectra in dichloromethane are listed in Tables 1 and 2. The normalized UV-Vis absorption spectra in dichloromethane are shown in Fig. 4. The UV-Vis absorption spectra display a shoulder at the shorter wavelengths of the absorption peak of the produced dyes due to a dimeric aggregate as shown earlier by our research group. This shoulder was not shown in dyes **6.8** and **6.9** with a hydroxyl terminal group on the donor  $2^{\circ}$  amine as a result of the expansion of the absorption peak.

Enhancement of the molecular weight of TCF-based chromophores to facilitate the preparation of thicker films was required for photophysical measurements. These "dimer" chromophores (Fig. 5) have been prepared and evaluated.

## 3.3. Thermal properties

The thermal properties of a range of the obtained TCF-based chromophores were studied *via* differential scanning calorimetry (DSC). The melting points  $T_{\rm m}$  are strongly affected by the substituents on the donor amine groups displaying an increase of the melting points  $T_{\rm m}$  with reducing the aliphatic chain length on the donor amine groups. Incorporating an alkene group affects the melting points  $T_{\rm m}$  as the alkene-containing chromophores display a lower melting points  $T_{\rm m}$  compared to chromophores without an alkene bridge. Many of the TCF chromophores display thermal decomposition around 300 °C. Fig. 6 displays the DSC data evidence of glass formation of TH-TCF-8-V 5.

Neat samples of some of these TCF dyes also have the valuable property that they form relatively stable glasses upon quenching from the isotropic, as in the case of TH-TCF-8-V 5 (Fig. 6). These glasses are of high (linear) optical quality.



Fig. 4 Normalized UV-Vis absorbance spectra of some selected TCF chromophores in dichloromethane.

One shortcoming of these dye monomers involves the fabrication of thin glassy films required for physical studies. Solutions of sufficient viscosity are difficult to achieve to permit reliable spin coating. So, in some refinements higher molecular weight dyes (often just dimers) were made as an approach to dealing with the thin film preparation.

#### 3.4. Assessment of dyes uptake

The polyester fibers are hydrophobic substrates known by their high affinity to disperse dyes, which are sparingly soluble in water and exhibit non-ionic low molecular size. The synthesized chromophores were applied to polyester fibers to introduce shades between orange and blue. As depicted in Fig. 7, all dyes showed high uptake into polyester fibers indicating high dye penetration through the polyester fibers. This can be ascribed to the polyester affinity to the applied dyes which are characterized by small and planar molecular structures. Hence, the dyed polyester demonstrated high colorfastness against washing, perspiration, light and rubbing as displayed in Table 3. The colorfastness to light depended on the aromatic-vinyl bridge and the tertiary amine donor group bonded to those aromatic-vinyl bridge moieties, which are capable to change the electron density over the whole dye molecule. Therefore, all dyes demonstrated good fastness to light except dyes bearing a thiophene moiety. However, the dyes bearing a thiophene moiety displayed improved color strength compared to dyes bearing a phenyl moiety. The color shades of the dyed polyester were in agreement with the observed wavelength absorbance maxima of the synthesized dyes in solution.

Table 2      Some high molecular weight TCF chromophores and their photophysical properties							
Bis-chromop	hore Molecular formula $(g mol^{-1})$	Melting point (°C)	$\lambda_{max}$ (nm) in CH <sub>2</sub> Cl <sub>2</sub>	ε <sub>max</sub> in CH <sub>2</sub> Cl			
15	C <sub>52</sub> H <sub>50</sub> N <sub>8</sub> O <sub>6</sub> ; 883.00	218	550	87 206			
16	C <sub>54</sub> H <sub>54</sub> N <sub>8</sub> O <sub>6</sub> ; 911.05	227	448	114 578			
18	$C_{40}H_{32}N_8O_2$ ; 656.73	>300	624	166 787			





Fig. 5 TCF chromophores for preparation of thicker films.



#### 3.5. Antimicrobial activity

The prepared dyes were tested against *E. coli, S. aureus* and *C. albican*, employing the plate agar count procedure. The antimicrobial reduction percentage motivated by each synthesized colorants is displayed in Table 4. Dyes with s phenyl-vinyl bridge displayed low inhibition to the reduction percentage, while Dyes with a thiophene-vinyl bridge displayed moderate antimicrobial activity.

# 4. Conclusion

Novel multifunctional push-pull architectures of small organic TCF fluorophores have been synthesized, characterized and structure optimized. They have been characterized with different analysis techniques including <sup>1</sup>H and <sup>13</sup>C NMR, FT-IR,



Fig. 7 Dye-uptake on polyester fibers.

elemental analysis, DSC, UV-Vis absorption, and fluorescence emission spectroscopy. TCF chromophores containing a vinylbridge were easily prepared *via* Knoevenagel condensation between TCF moiety and the corresponding aromatic aldehyde. The TCF intermediate containing 4-fluorophenyl bridge can directly react with a variety of unhindered secondary amines by aromatic nucleophilic substitution to afford the corresponding TCF chromophores without vinyl group. Most of the produced TCF chromophores display high extinction coefficient values. The highest extinction coefficient values were observed for the thiophene vinyl-bridged chromophores. The produced materials show promising characteristics suitable for different optoelectronic applications such as photovoltaics, 3D optical data

Table 3 Fastness, shade and color strength of dyed polyester fibers

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Wash			Perspira	tion			Rubbing				
Dyes		St. <sup>a</sup>	Acidic		Basic						
	Alt. <sup>a</sup>		Alt. <sup>a</sup>	St. <sup>a</sup>	Alt. <sup>a</sup>	St. <sup>a</sup>	Dry	Wet	Light	Shade	K/S
3	4-5	4	3-4	3-4	4	4	4	3-4	5	Blue	17.04
4	4	4-5	4	3	4-5	4-5	4-5	4	5	Blue	19.92
5	4	4-5	3-4	3-4	4	4	3-4	3-4	5	Blue	23.57
8	4-5	4	3	3-4	4	4-5	3-4	3-4	6-7	Red	9.78
9	4	4-5	3	3	4-5	4	4-5	4	6	Red	12.27
12	4-5	4-5	3-4	3	4-5	4-5	4-5	3-4	5-6	Blue	15.43
13	4	4	3	3	4	4	4	4	5	Blue	18.71
15	4-5	4-5	4	3-4	4	4-5	4	3	6-7	Red	10.06
16	4-5	4	4	4	4-5	4-5	4-5	3	6	Red	13.43
18	4	4	3-4	3	4	4	4	3-4	6-7	Red	9.76
21	4	4-5	4	3-4	4	4-5	4-5	4	6-7	Orange	8.66

<sup>*a*</sup> Alt. = alteration in color; St. = staining on cotton.

Table 4 Antimicrobial activity of the prepared dyes

	Bacterial reduction	n %	Fungal reduction % <i>C. albican</i>	
Dyes	E. coli	S. aureus		
3	$22 \pm 1.2$	$19\pm1.4$	$9\pm1.1$	
4	$22 \pm 1.6$	$21 \pm 1.1$	$11 \pm 1.3$	
5	$24 \pm 1.1$	$23 \pm 1.5$	$11 \pm 1.4$	
8	0	0	0	
9	0	0	0	
12	$12 \pm 1.3$	$16 \pm 1.8$	$9\pm1.1$	
13	$19 \pm 1.1$	$21\pm1.5$	$8 \pm 1.3$	
15	0	0	0	
16	0	0	0	
18	0	0	0	
21	0	0	0	

storage and laser dyes. Moreover, the prepared colorants were applied onto polyester fabrics as disperse dyes using the high temperature and pressure method to provide good color depth of shades ranging between orange to blue. The studied colorants demonstrated mostly acceptable colorfastness properties. We also studied the synthesized dyes as potential antimicrobial compounds against some pathogenic species. Thiophene vinylbridged TCF based dyes displayed moderate antimicrobial activity, while phenyl vinyl-bridged TCF based dyes showed weaker antimicrobial activity.

# Conflicts of interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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