



Eco-Friendly Dyeing and Functional Finishing of PET Fabric at Mild Conditions

Nabil A. Ibrahim^a, Enas M. El-Zairy^b, Soliman R. Barakat^{b*}

^a Textile Research and Technology Institute, National Research Centre, 33 El Bohouth st, Dokki, Giza, Egypt.

^b Faculty of Applied Arts, Textile Printing, Dyeing and Finishing Department, Helwan University, Cairo, Egypt.



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In Loving Memory of Late Professor Doctor "Mohamed Refaat Hussein Mahran"

Abstract

An eco-friendly single step process for free carrier dyeing and anti-UV/antibacterial functional finishing of alkali-treated PET fabric using TiO₂ NPs as safe carrier and environmentally benign functional additive along with disperse and cationic dyes at mild temperature is successfully developed. The obtained data demonstrate that increasing TiO₂ NPs concentration up to 8 g/L, dyeing temperature up to 100°C for 1h, and dye concentration up to 3% (owf) results in a significant improve in: color strength, anti-UV blocking ability as well as antibacterial efficacy against the tested *S. aureus* and *E. coli* pathogenic bacteria. On the other hand, the variation in color strength, fastness properties as well as in the imparted anti-UV and antibacterial functionalities is governed by type of the used dye. SEM and EDX analysis for selected cationic dyed/functional finished samples confirmed the presence of Ti-element in addition to carbon, nitrogen and oxygen onto the simultaneously dyed/functional finished fabric.

Keywords: TiO₂ NPs, Modified PET, Disperse and cationic dyes, Eco-friendly single step, Functional properties.

1. Introduction

Polyester (PET) is one of the most widely used synthetic fabric in textile industry as a direct consequence of its many favorable performance properties such as high mechanical properties, dimensional stability, very good antcrease and washing durability as well as antichemicals [1].

Because of the aforementioned favorable features, PET is widely used for home textile products, apparels and automotive interior fabrics [2, 3]. However, PET is hydrophobic in nature, its high crystallinity, as well as lack of chemically active sites and functional groups negatively affect its disperse dyeing in conventional dyeing under atmospheric conditions [4].

Disperse dyeing of PET may be achieved and enhanced either by using high temperature (≈130°C) or by dyeing at boil in the presence of carrier to accelerate exhaust of its dyeing, or by padding technique using an appropriate dispersing agent followed by dyeing and then curing at 190-220°C [5, 6]. Disperse dyeing of PET in the presence of carrier has significant negative impacts such as toxicity, unpleasant odor as well as environmental pollution and contaminations [7].

More R&D efforts are still required to develop free carrier disperse dyeing of PET at mild conditions, surface modification, as well as to enhance its performance and functional properties [2, 3, 8-10]. Therefore, the main task of the present study is adopting and

implementing cleaner production opportunities for developing an environmentally sound single step method for fabrication of disperse and basic dyed/multifunctionalized of pre deweighted PET fabrics through the inclusion of TiO₂NPs in the dyeing bath at mild conditions using the exhaustion technique [11-13].

2. Experimental

2.1. Materials

A plain weave 100% polyester fabric (PET, mass per unit area of 220 g/m²) was used throughout this study.

The Commercially disperse dyes: C.I. Disperse Red 60, C.I. Disperse Yellow 42 and C.I. Disperse Blue 77, as well as commercially basic dyes: C.I. Basic Red 29, C.I. Basic Blue 3 and C.I. Basic Red 46, used for this investigation were kindly supplied by Sonochem® Ningbo, Ching, with the chemical structures shown in Table 1.

Nano-Sized TiO₂ (mixture of anatase and rutile, particle size 10-30 nm, Skypring Nanomaterials Inc. USA) was also utilized. Other chemicals used were all reagent grade.

*Corresponding author: Soliman R. Barakat, e-mail: soliman_eng@yahoo.com

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2.2. Materials

2.2.1. Surface modification of PET

To modify the PET-fabric surface as well as to enhance its hydrophilicity and functionality, the PET-fabric was padded twice in a deweighting bath containing 30 g/L NaOH to a wet-pick up of 85%, at speed of 20 m/min, with a pressure of 2 bars, followed by steam fixation at 110°C using a Korean weight reduction equipment. The treated PET-fabric was then washed, neutralized using acetic acid, thoroughly rinsed with warm water, and finally dried/thermosetted at 200°C for 30s [14].

2.2.2. Combined coloration and functionalization

Unless otherwise stated, post coloration/functionalization of the pre-modified PET-fabric sample was carried out using Lab IR dyeing machine. Factors affecting the extent of coloration and functionalization of the pre-modified PET-fabric samples namely TiO₂ NPs concentration, the liquor ratio, type of dye and its concentration (% owf), as well as dyeing temperature were varied to examine their impacts on the extent of coloration, expressed as K/S and fastness properties, as well as degree of functionalization, expressed as antibacterial functionality and UV-shielding capacity. Removal of excess and unfixed dyes and/or TiO₂ NPs was carried out by after-washing with 2g/L nonionic wetting agent out 45°C for 20 min in a lab IR dyeing machine, then thoroughly rinsed and finally dried at 100°C for 3 min.

2.3. Measurements

The following tests were carried out on alkali-treated and untreated PET-fabrics.

- The loss percentage of weight (R) due to alkali-pretreatment was calculated using the following formula: $R (\%) = (W_o - W_i) / W_o \times 100$ where W_o and W_i are the weights of PET-samples before and after the alkali surface modification.
- Breaking strength (BS) of alkali-treated and untreated PET-fabric samples in warp direction, was measured according to (ASTM D5034-21)
- Air permeability of the treated and untreated PET-fabric samples were conducted by using KES-F8-API, and was evaluated by windage resistance (ASTM D737-18(2023))
- The color strength of dyed samples, expressed as K/S Value, was calculated using the Kubelka - Munk equation [15].

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

where K is the absorption coefficient, S is the scattering coefficient, and R represents the reflectance at wavelength of maximum absorption.

- Fastness properties to washing (WF), perspiration and light fastness (LF) of treated fabric samples were evaluated according to AATCC Test

methods: (61-2020), (AATCC15-2021) and (16.1-2023) respectively.

- UV protection factor, UPF, was determined according to the Australian/New Zealand Standard (AS/NZS 4399:2017).
- Antibacterial activity of the obtained dyeings against both Gram-positive (*S. aureus*) and Gram-negative (*E. coli*) pathogenic bacteria was evaluated qualitatively according to AATCC Test Method (AATCC TM147-2011 (2016e), and expressed as zone of growth inhibition (ZI, nm).
- The surface morphology of selected dyed fabric samples with and without TiO₂ NPs was examined using Quanta SEM 250 FEG (Field Emission Gun) equipped with energy dispersive X-ray spectroscopy (EDS) with accelerating voltage-30 KV FEI Co., Netherland for the surface compositions analysis.
- All determinations were done in triplicate and the average was taken as final results.

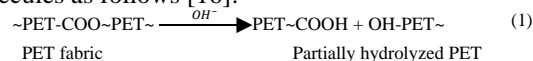
3. Results and Discussion

Herein, an eco-friendly single treatment step for coloration and functionalization of pre-alkali treated PET fabric using TiO₂ NPs as an environmentally benign carrier, inorganic UV blocking agent and pathogenic bacteria protector along with various disperse and cationic dyes at mild conditions was developed to meet the ever-growing consumer health and safety awareness as well as environmental concerns. Factors affecting the combined dyeing and protecting process, expressed as extent of coloration and functionalization, of the developed substrates as well as full discussion of the obtained results follow.

3.1. Effect of alkali treatment

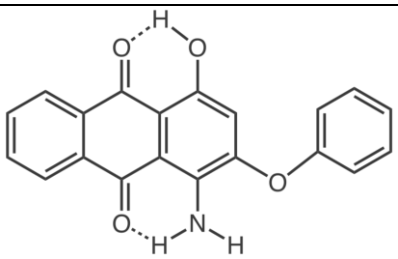
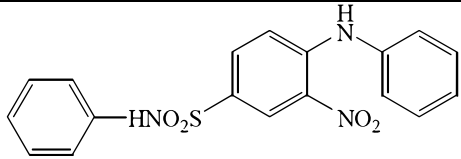
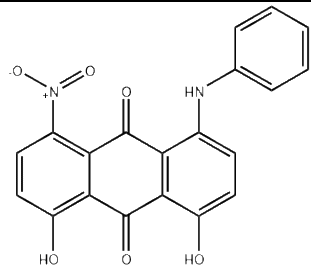
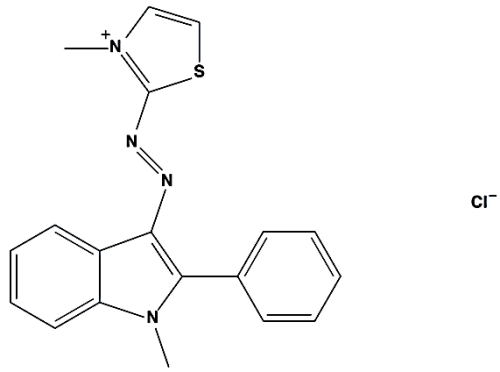
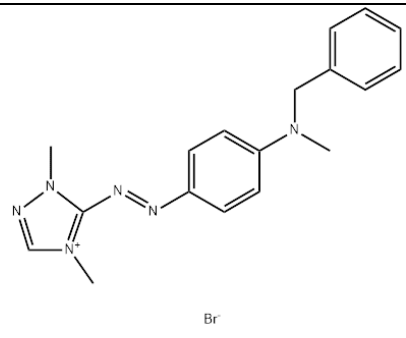
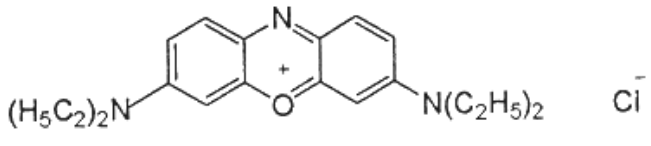
The data in Table 2 demonstrate that alkali-treatment of PET fabrics under the given conditions results in i) a loss in weight (14.15 %), in warp tensile strength (4.95%) and elongation at break (10.94%), ii) an increase in air permeability (51.63%), and a marginal improve in antibacterial activity against both *S. aureus* and *E-Coli* bacteria, and iii) a noticeable decrease in UV-protection ability, expressed as UPF values, (73.39%).

The changes in the aforementioned properties of pre-dewighted fabric compared with the untreated one reflects the impact of alkali-treatment on promoting partial hydrolysis of ester-linkages of PET macromolecules as follows [16].



which in turn negatively affects the fabric strength, density, UV-shielding capacity, and positively affects both air permeability, to a large extent, and antibacterial activity to a marginal extent.

Table 1 Chemical structures of the used dyes

Type	Dye Name	Dye Structure
	C.I. Disperse Red 60	
Disperse Dyes	C.I. Disperse Yellow 42	
	C.I. Disperse Blue 77	
Basic Dyes	C. I. Basic Red 29	
	C. I. Basic Red 46	
	C. I. Basic Blue 3	

The decrease in fabric thickness as well as peeling off of the copolymerization additives, especially inorganic ones, negatively affect the UV-protection ability of the deweighted fabric and positively affect its air permeability [17]. On the other hand, the marginal improve in antibacterial activity of alkali-treated PET is a direct consequence of generation of free active groups, i.e. –COOH and -OH groups.

Consequently, the modified PET fabric structure could be exploited as an appropriate substrate for combined disperse or basic dyeing along with functional finishing, i.e. antibacterial and UV- blocking properties, in the presence of TiO₂ NPs as eco-friendly functional / textile auxiliary agent at mild conditions.

3.2. Effect of TiO₂ NPs concentration

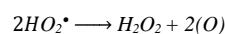
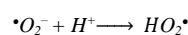
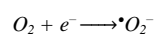
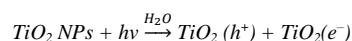
As far as the change in color strength (K/S, Fig. 1a), UV- shielding property (UPF, Fig. 1b), and antibacterial activity against *S. aureus* (ZI, Fig. 2C) and *E. coli* (ZI, Fig. 2d) as a function of dye type (disperse or basic) and TiO₂NPs, as multifunctional auxiliary, concentration, it is clear that increasing TiO₂ NPs concentration up to 8 g/L in the dyeing bath at 100°C for 60 min is accompanied by a gradual increase in K/S values of the obtained dyeings. The increase in the K/S values reflects the positive role of the inclusion of TiO₂ NPs as a safe carrier in disperse dyeing as well as in enhancing and improving the fabric hydrophilicity and wettability along with the generated active sites i.e. -COOH and -OH groups, onto the alkali- treated polyester which in turn positively affects its dyeability and dye-fibre affinity, regardless of the used dye [18, 19]. The extent of dye fixation onto/ within the fabric structure is governed by type of dye, its molecular and chemical structure, affinity and functionality as well as degree of fixation under the given dyeing conditions [20, 21].

On the other hand, it could be observed from Fig. 1b that increasing TiO₂ NPs up to 8g/L in the dyeing / functional finishing formulation brings about a sharp increase in UPF value (> 50+) which reflects the positive blocking/shielding role of loaded TiO₂ NPs onto the modified PET-fabric structure via its active sites i.e. -COOH and -OH groups, thereby leading to higher anti - UV functionality along with the fixed dye absorption ability compared with TiO₂ NPs-free samples [22]. The variation in the imparted UV protection functionality is governed by type of dye, extent of its fixation along with degree of immobilization of TiO₂ NPs, as well as the synergetic effect of TiO₂ NPs / dye under the given dyeing/ functional finishing conditions.

From Fig's 1c and 1d, it is clear that increasing TiO₂ NPs concentration within the range examined (0- 8 g/L) results in a significant improve in the imparted antibacterial activity against the tested pathogenic bacteria, *S. aureus* (Fig. 1C) and *E-Coli* (Fig. 1d). The extent of improvement in antibacterial functionality is

determined by type of the used dye, Basic dye > Disperse dye, as well as kind of pathogenic bacteria, antibacterial against *S. aureus* > *E-Coli*, keeping other parameters constant.

The higher enhancement in the imparted antibacterial activity of basic dyed fabric compared with the disperse dyed reflects the positive role of cationic active sites (quaternary ammonium groups), on damaging of cell membrane, inhibiting DNA and hindering multiplication [23], along with the negative impacts of generated ROS, e.g. •OH, •O₂, H₂O₂, etc (scheme 1) on Cell membrane viability growth, which in turn causes bacterial death [24].



Scheme 1. Generation of ROS

On the other hand, the imparted antibacterial activity follows the decreasing order *S.aureus* > *E. Coli*, reflecting their differences in cell wall structure, degree of damaging cell wall, extent of inhibition of the enzyme activity along with subsequent destruction of the cell wall and death of Pathogenic microbial cell [25, 26].

3.3. Effect of treatment temperature

As far as the change in antibacterial activity, ZI, UV-shielding property, UPF, as well as in color strength, K/S, of the obtained functionalized dyeings as a function of dyeing temperature, the obtained data, Fig. 2, demonstrate that increasing dyeing temperature up to 100°C for 60 min results in a remarkable improve in K/S value of the obtained dyeings, regardless of the used dye (Fig. 2a), along with a significant improve in the UV-shielding capability, expressed as UPF value (Fig. 2b). On the other hand, the data so obtained demonstrate that, the higher the dyeing temperature, the better the imparted antibacterial activity of the developed PET dyeings against both *S. aureus* (Fig. 2c) and *E. coli* (Fig. 2d), keeping other parameters constant.

The variation in the extent of dyeing as well as degree of protecting against the harmful UV-radiation and the pathogenic bacteria is determined by kind of dye as well as type of pathogenic bacteria respectively [27, 28].

The positive role of raising dyeing temperatures from 40 to 100°C could be interpreted in terms of: opening the PET structure, enhancing the extent of swellability and mobility of the macromolecular

chains in PET structure as well as solubility of the used dye, improving the extent of the interaction of the modified substrate with the used dyes and TiO₂ NPs constituents through its active sites, i.e. -COOH and -OH groups [29]. The net effect of the positive impacts of raising dyeing temperatures up to 100°C for 60 min would be expected to lead to higher extent of simultaneous dyeing and functional finishing.

The experimental results so obtained demonstrate that creation of active sites like -COOH and -OH groups onto/within PET structure as well as fixation of TiO₂ NPs onto the alkali-treated PET enhance wettability, improve dyeability, increase the extent of adhesion and fixation of disperse and basic dyes, which in turn facilitates surface modification, coloration and functionalization to meet various potential demands [30, 31].

On the other hand, the positive role of TiO₂ NPs on enhancing the extent of adhesion and fixation of disperse and basic dyes, as well as generation of powerful ROS, especially [•]OH- scheme 1, can further participate in creation of free active sites on both the PET structure as well as the dye molecule, thereby enhancing the extent of dye-fibre affinity and fixation along with the traditional Van der Waals dipols forces, hydrogen bonding as well as ionic bonding as in case of using the basic dye [32], along with imparting antibacterial activity to the dyed substrate, as discussed earlier.

Table 2 Effect of alkali-treatment on some performance properties of treated polyester

Substrate	WL (%)	B. S (warp) (Kg)	EB (%)	AP Cm ³ /cm ² .sec	UPF	ZI (mm)	
						<i>S. aureus</i>	<i>E. coli</i>
Untreated	0.00	87.0	64	10.42	30	1.5	1.0
Alkali-treated	14.15	76.6	57	15.80	22	2.0	1.5

Alkali-treatment: Na OH (30g/L), wet-pick up (85%); speed (20 m/min), pressure (2 bars); followed by steaming at 110°C using a Korean weight reduction machine. Alkali treated fabric polyester fabric was then washed with hot water, neutralized with acetic, thoroughly rinsed with warm water to remove excess reactants & hydrolyzed by- products, and Finally, dried/thermosetted at 200°C for 30 sec.

WL: Wight loss, BS: breaking strength; EB: elongation at break, AB: air permeability UPF: UV-protection factor; ZI: zone of inhibition.

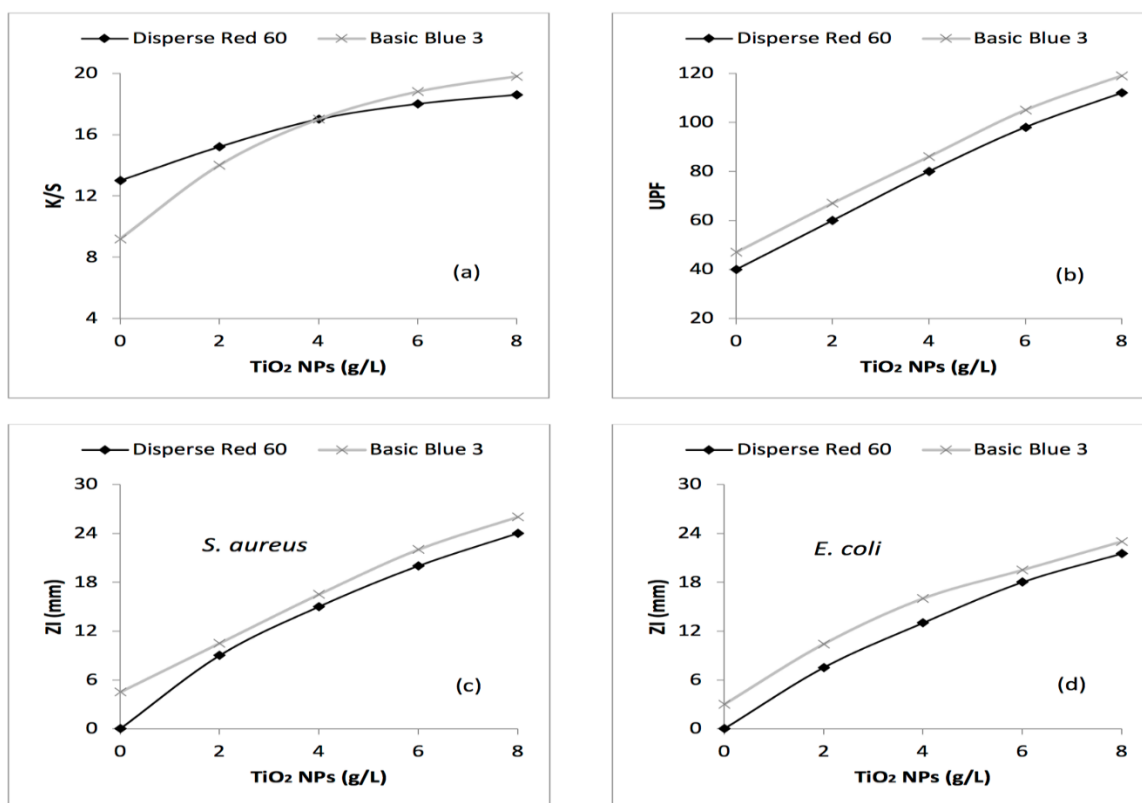


Fig. 1 Effect of TiO₂ NPs concentration on K/S (a), UPF (b), and antibacterial (c & d) properties of alkali treated PET fabric samples. Dye (2%), MLR (1:20), Temp. 100°C, Time 60 min.

3.4. Effect of dye concentration

Fig. 3 shows the effect of dye concentration, % owf, on the K/S (Fig. 3a), UV-protection efficacy (UPF-Fig. 3b), as well as on the imparted antibacterial activity, ZI, against *S.aureus* (Fig. 3c) and *E.coli* (Fig. 3d) pathogenic bacteria. For a given set of simultaneous dyeing and functional finishing of alkali treated PET, it is clear that increasing dye concentration up to 3% owf results in an increase in K/S and UPF values along with a noticeable improve in antibacterial functionality against the tested pathogenic bacteria.

The enhancement in the imparted coloration and functionalization properties, expressed as K/S, UPF and ZI values respectively, could be discussed in greater availability of the dye molecules and dye free radicals in vicinity of the immobilized PET active sites, -OH and -COOH and macroradicals, thereby enhancing the extent of dye fixation as well as TiO₂ NPs

immobilization, which in turn positively affects the color strength of the obtained dyeings and conferring the modified PET fabric samples a remarkable anti-UV and anti-bacterial protection properties.

The extent of improve in coloration and functionalization properties is governed by type of dye: Cationic > disperse taking in consideration their differences in chemical structure, functional groups, affinity to the modified substrate, ability to interact with and/or facilitate fixation of the TiO₂ NPs onto / within the pre-alkali treated substrate during simultaneous dyeing / functional finishing step at 100°C for 1h [33-35].

Additionally, the extent of improvement in the imparted antibacterial functionality to the pre modified PET fabric against pathogenic bacteria *S.aureus* & *E. coli* affected by: i) type of dye, cationic > disperse, most probably due to the electrostatic interactions between the positive groups of dye and the negatively charged cell membrane of bacteria, thereby leading to membrane disruption, cytoplasmic leakage and subsequent death of the pathogenic bacterial cell [33, 35],

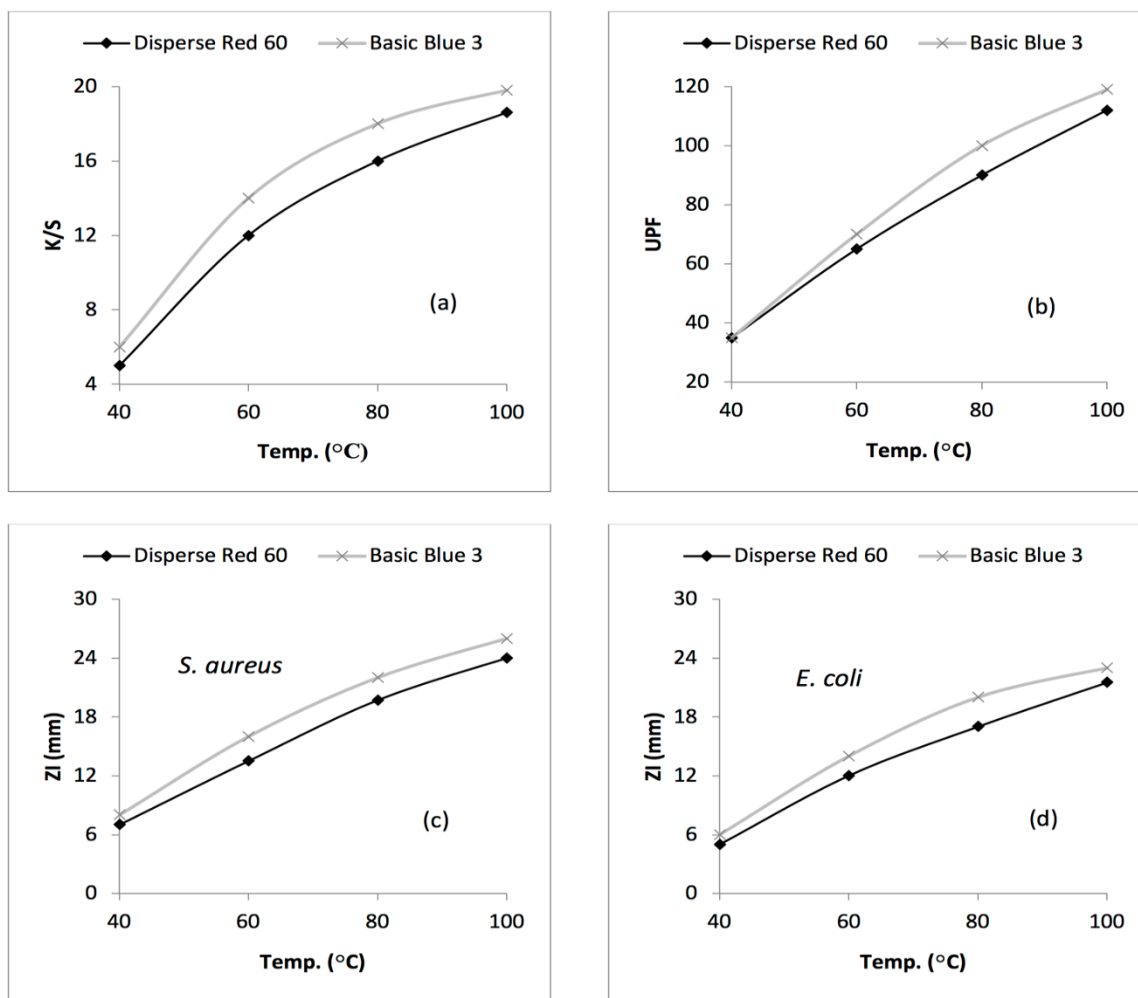


Fig. 2 Effect of dyeing temperature on K/S (a), UPF (b), and antibacterial (c & d) properties of alkali treated PET fabric samples. Dye (2%), TiO₂ NPs (8 g/L), MLR (1:20), Time (60 min).

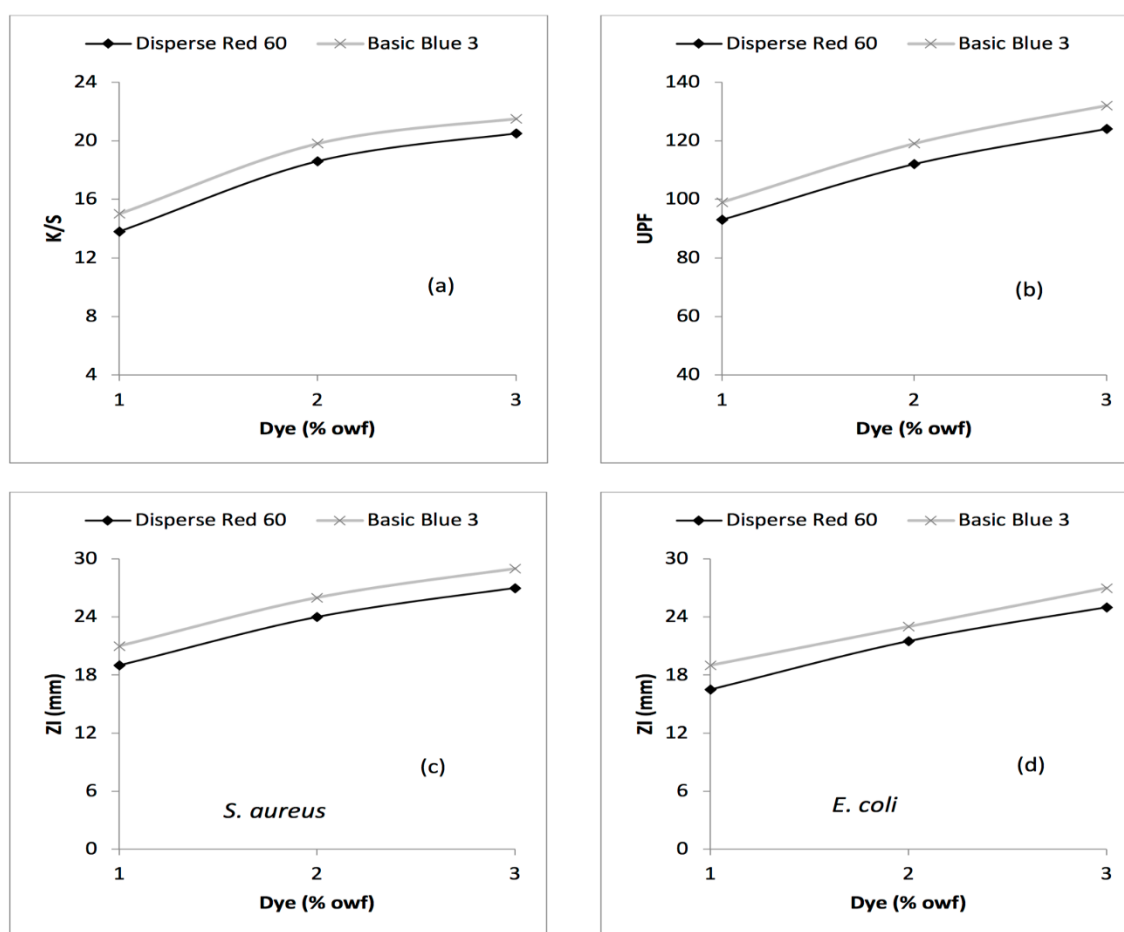


Fig. 3 Effect of dye concentration on K/S (a), UPF (b), and antibacterial (c,d) properties of alkali treated PET fabric samples. TiO₂ NPs (8 g/L), MLR (1:20), Temperature (100°C), Time (60 min).

ii) extent of loading TiO₂ NPs onto and/ or within the treated substrate, which in turn affects generation of ROS like: $\cdot\text{OH}$, $\cdot\text{O}_2$, H₂O₂ etc. and their negative impacts on bacterial cell membrane, cell manipulation and subsequently bacterial demising [36], and iii) type of pathogenic bacteria, *S. aureus* > *E. coli* as a direct consequence of their differences in cell wall structure, arrangement and subsequent amenability to inhibition and/or Killing [37].

3.5. Surface analysis (SEM) and elemental analysis (EDX) of selected samples

The change in surface morphology of **C.I. Basic Blue 3** fabric samples in the absence (Fig. 4a) and presence of TiO₂ NPs (Fig.4c) was examined. A close view of both figures clearly demonstrates the remarkable change in surface morphology of TiO₂ NPs - loaded fabric samples (Fig.4c) compared with the unloaded smooth surface one (Fig. 4a).

On the other hand, EDX analysis confirms the presence of Ti element along with C, N and O on the surface of dyed / functionalized fabric sample (Fig. 4d),

while the only dyed fabric surface (Fig.4b) consisted of C, N and O elements.

Of course, the presence of -COOH and -OH groups in alkali-pretreated PET structure facilitates and helps to uptake and immobilize / fix both the basic dye molecules and TiO₂ NPs onto / within the modified substrates.

3.6. Effect of using different dyes

As far as the variation in coloration properties, expressed as K/S value and fastness properties, as well as in the imparted functional properties, expressed as UPF and ZI values, of the simultaneously dyed and functionalized PET fabric samples as a function of dye type, the data in Table 3 demonstrate that: i) using different disperse and cationic dyes in combined coloration/functionalization step results in a noticeable variation in K/S, antibacterial efficacy along with a change in surfaces properties without affecting the imparted anti-UV property (50+),

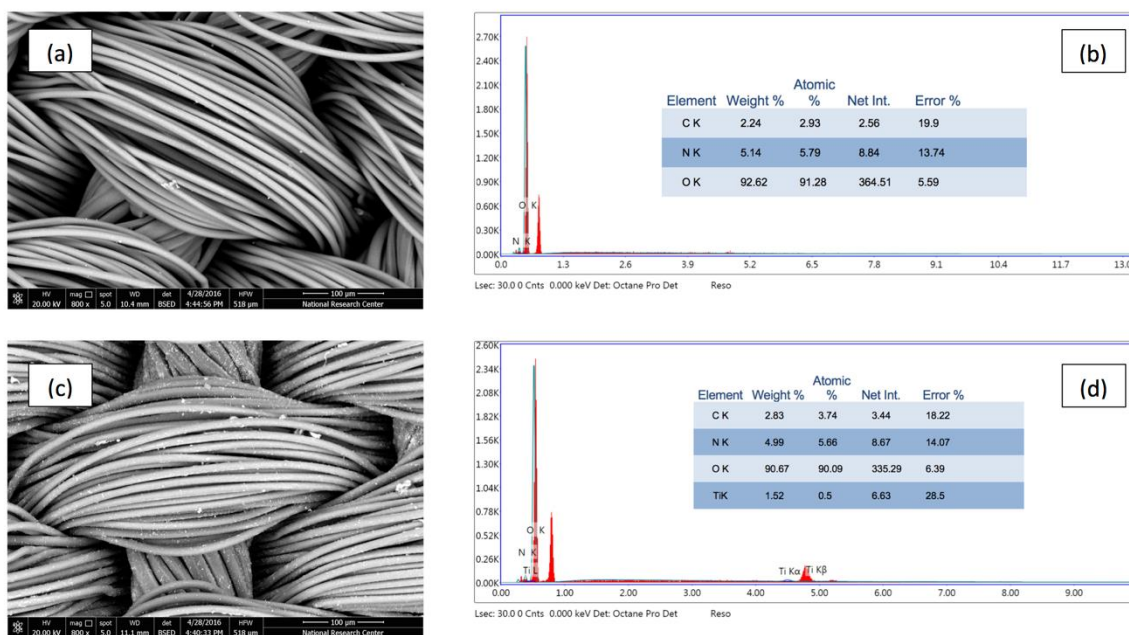


Fig 4. SEM & EDX , a) SEM of PET dyed with C.I. Basic Blue 3, b) EDX of PET dyed with Basic Blue 3, C) SEM of TiO₂ PET treated fabric, d) EDX of TiO₂ PET treated fabric.

Table 3 Effect of Using Different Dyes

Type of dye (2% OWF)	K/S	UPF	ZI (mm)		W. F.		L. F.	P. F.		
			<i>S. aureus</i> (G+ve)	<i>E. coli</i> (G-ve)	St.	Alt.		Aci.	Alk.	
Basic	Basic Red 29	18.90	50 ⁺	24.0	22.0	4-5	4-5	4-5	4	4
	Basic Red 46	11.83	50 ⁺	20.0	19.0	4-5	4-5	4-5	4	4
	Basic Blue 3	21.5	50 ⁺	29.0	27.0	4-5	4-5	4-5	4	4
Disperse	Disperse Red 60	20.5	50 ⁺	27.0	25.0	4	4	3-4	3-4	3-4
	Disperse Yellow 42	16.82	50 ⁺	22.5	20.5	3-4	3-4	4	4	4
	Disperse Blue 77	19.19	50 ⁺	22.0	18.5	4-5	4-5	4-5	4-5	4-5

Dye 3% owf, TiO₂ 8 g/L, MLR 1:20, temp. 100°C for 60 min

K/S: colour strength, UPF: UV-Protection Factor, ZI: Zone of Inhibition, W. F: Washing Fastness, L. F: Light Fastness, P. F: Perspiration Fastness

ii) the extent of coloration and functionalization of the modified PET is governed by kind of the used dye, i.e. chemical composition, molecular structure, active sites, mode of interaction among the dye, TiO₂ NPs and the alkali-pretreated PET, UV-blocking ability and antibacterial efficacy as well as extent of fixation and immobilization onto and/or within the modified PET [13, 38], iii) all developed products acquired antibacterial activity against the tested pathogens, an outstanding UV-protection efficacy and demonstrated good to high fastness properties as a direct consequence of appropriate and high fixation of the used dyes.

4. Conclusion

A facile and green single step process for disperse or cationic dyeing and functional finishing of alkali-hydrolyzed PET at mild temperature was developed

using TiO₂ NPs as a safe carrier and functional auxiliary. The developed dyed/functional finished PET fabric samples showed improved coloration properties and enhanced antimicrobial functionality against *S. aureus* and *E. coli* pathogenic bacteria along with outstanding anti-UV protection efficacy. The best improvement in both coloration and functionalization was achieved upon using 8g/L TiO₂ NPs, at 100°C dyeing temperature and 3% owf dye concentration. The developed dyeings demonstrated mostly good to very good fastness properties. Inclusion of TiO₂ NPs into the dyeing bath enhanced the extent of dyeing as well as upgrade the imparted functional properties. SEM and EDX analysis confirmed both the change in surface morphology as well as the deposition of Ti element. From the above, the suggested method for developing colored/multifunctionalized PET products showed to be a promising approach to produce

colored, antibacterial and anti-UV textiles with vast potential applications at mild conditions.

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