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In situ anchoring iron oxide nanoparticles onto polyester/disperse dye for production of multifunctional fabrics

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Abstract

Medical textiles, including surgical gowns, masks are used as obstacles to prevent the risk of infection for both doctor and patient. The widespread of bacteria and viruses, e.g. chronic hepatitis B, hepatitis C and currently Covid-19 viruses in the patient population is very common. In this work, multifunctional eco-friendly polyester fabrics have been produced by in situ impregnation of 2-((E)-(2-(2,4-dinitrophenyl)hydrazono)methyl)-4-((E)-phenyldiazenyl)phenol disperse dye onto magnetic iron oxide nanoparticles. The technique endowed polyester fabrics with a new color as well as magnetic and antibacterial functionalities. The colored magnetic nanoparticles showed high affinity toward fabrics. Besides, the unbound dye could be easily collected from wastewater by a magnet, significantly facilitate the wastewater treatment. The treated fabrics were analyzed by energy dispersive X-ray spectroscopy, scanning electron microscopy, X-ray diffraction and vibrating sample magnetometry. Colorimetric values, tensile properties and fastness of the composite fabrics were also measured. The tensile properties of the composite were increased after functionalization. The wettability features of the fabric were investigated and showed a significant improvement. Also, the toxicity of the resulted fabric was exhibited low toxicity against wi-38 cell line. These results indicate the potentiality of the suggested technique in producing multifunctional fabrics with various applications, especially as medical textiles.

Keywords: Polyester fabric, Disperse dye, Magnetic iron oxide nanoparticles, In situ coloration and functionalization, Multifunctional product

Introduction

Nanomaterials of magnetic elements are known as magnetic nanoparticles (MNPs). It has received an extensive consideration owing to their impressive merits. They have high surface area, superparamagnetic properties, great ability for modification and lower biotoxicity (Zhang et al., 2017a, b). The most important of this category are iron (Fe), cobalt (Co) and nickel (Ni) due to its high magnetic properties and high ability to control and regulate the size and composition as well as the shape compared to magnetic metal oxide nanoparticles (Hedayatnasab et al., 2017). Recently, iron and iron oxide, especially in its nanosized forms have received significant interest because of their excellent magnetic

properties (Jadoun et al., 2020; McCafferty et al., 2015; Sharouf & Saffour, 2023). Moreover, magnetic iron oxide nanoparticles (MIONPs) has been highlighted due to their utilization in adsorption of metal aqua ions like Cu^{2+}, Hg^{2+}, Cr^{6+}, Au^{3+} As^{3+} and organic compounds from aqueous solution (Shahidi, 2019; Zhang et al., 2017a, b). Furthermore, iron-based NPs have their own unique properties such as antimicrobial and/or antiviral properties, self-clean, anti UV, wettability which further improve the material finishing (Tan et al., 2019). Accordingly, researchers and scientists have used iron oxide NPs in protective textiles and medical applications for the prevention of microbial and viral infections due to its delightful performance (Abou Elmaaty et al., 2018a, b; Elmaaty et al., 2018b; Hebeish et al., 2015; Ibrahim et al., 2013a, b, c, 2015, 2017a, b, 2018; Shahidi, 2016).

Harifi et al. reported on the utilization of ferric chloride under a sonochemical method to in situ synthesize the hematite and magnetite NPs which further deposited on polyester fabric (Harifi & Montazer, 2014). The resulted fabrics exhibited durable biological activity and magnetic properties which are useful for use in antibacterial and electromagnetic shielding (EMS) applications. Rastgoo et al. (2016) used ultrasound mediation for preparing Fe_3O_4 on cotton/polyester fabric that showed sonocatalytic, photocatalytic, antibacterial and magnetic characteristics (Rastgoo et al., 2016). As for the removal of dye or other organic compounds; Wen et al. reported that cotton fabrics were dyed with (disperse red 1) with the coloured (MIONPs) suspension by a dip-pad-dry method and after dyeing; wastewater was exposed to magnets. The unattached dyes were collected by magnets and was reused in the next dyeing processes (Wen & Sun, 2018). All previous methods utilized multiple steps to implement the fabrics with the combination of colour and functional properties and consequently suffered from the disadvantages of concomitant consumption of water, chemicals and energy. The present research was directed to develop a novel in situ method to overcome the disadvantages mentioned above by using one-step MIONPs anchoring and dyeing of PET fabric with multifunctional properties. This research also aims to improve the properties of water absorption and dyeing by hydrophobic polyester, in addition to improving the resistance to bacteria. The resulted multifunctional PET fabric is a promising candidate for using as medical textile and in dye removal.

Methods

Materials

A 100% plain-weave polyester (PET) fabric with wight of 108 g/m^2 and it was 70 denier warps and 150 denier weftswas purchased from Omar Afandy Co., Egypt. As for chemicals; ferrous sulfate ($FeSO_4 \cdot 7H_2O$), ferric trichloride ($FeCl_3$), sodium hydroxide (NaOH) and dispersing agent were used. The dyestuff used in this study was synthesized according to the method previously reported (Fig. 1) (Dehspande et al., 2012).

In situ dyeing and synthesis of (IONPs) on polyester fabric

The IONPs were synthesized on the polyester fabric by treating the fabric in a bath containing: $FeCl_3$, $FeSO_4 \cdot 7H_2O$ (Fe^{2+}/Fe^{3+} molar ratio = 2), NaOH (pH = 12), dispersing agent and azo disperse dye. The liquor ratio was kept at 1:100. The synthesis was

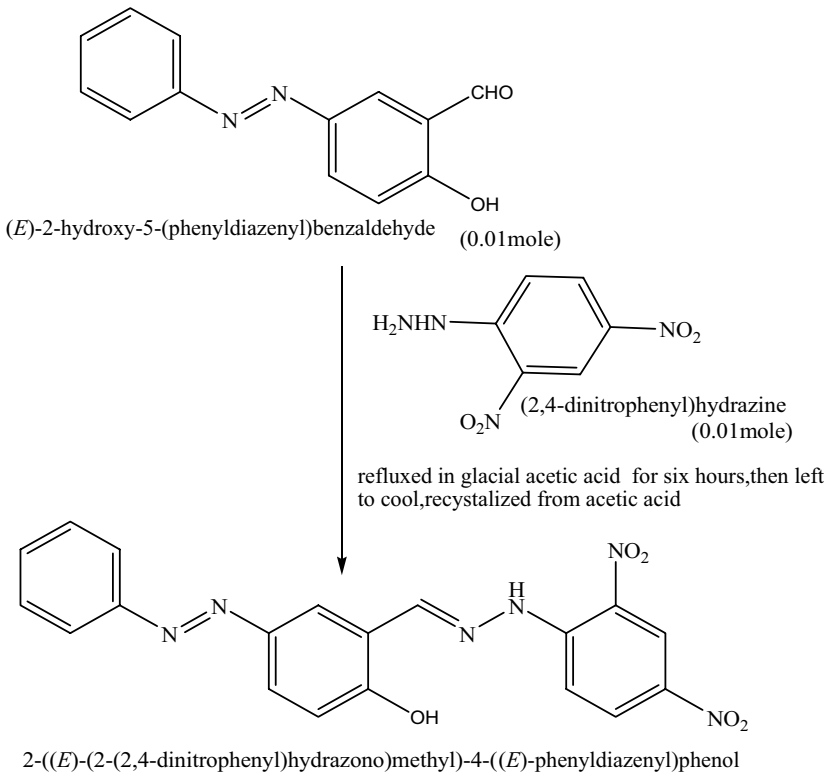

Fig. 1 Synthesis of 2-((E)-(2-(2,4-dinitrophenyl)hydrazono) methyl)-4-((E)-phenyldiazenyl)phenol

conducted at a temperature of 100 °C for 60 min. Finally, the resulted fabrics were rinsed with water for 15 min. at 60 °C and dried at around 20–22 °C. The resulted fabric coded with D/T.

Characterization

XRD was performed with a Bruker D8 ADVANCE, Karlsruhe, Germany using a Cu radiation source (30 mA) running at 40 kV to explore the crystalline phases and size of IONPs on the polyester fabric. The surface morphology of the D/T fabric and particle size of IONPs were analyzed by using Jeol-Japan-JSM-6510LVscanning electron microscope (SEM). The elemental composition of resulted fabrics was analyzed by using Oxford-x max 20 Energy Dispersive Spectrometer (EDX). The fastness properties of D/T fabric were measured by using methods of AATCC TM 61-1996 ("American Association of Textile Chemists and Colorists, AATTCC 61-1996: Colorfastness to Laundering: Accelerated, Research Triangle Park, NC, USA", 1996), AATCC TM 8-1996 ("American Association of Textile Chemists and Colorists, AATCC Test Method 8-1996, Colorfastness to Crocking, Research Triangle Park, NC, USA," 1996) and AATCC TM 16 .2-2022 ("American Association of Textile Chemists and Colorists. AATCC TM16.2-2022, Test Method for Colorfastness to Light: Carbon-Arc," 2022) for washing, dry/wet rubbing, and lightfastness, respectively. The antibacterial activity of the dyed and D/T polyester fabrics were assessed quantitatively according to AATCC TM 147 -2004 ("American Association of Textile Chemists and Colorists, AATTCC 147-2004: Antibacterial Activity Assessment of Textile Materials, Research

Triangle Park, NC, USA," 2010) method against *Staphylococcus aureus and Bacillus cereus* as a gram-positive bacterium and gram-negative bacterium (*E. coli*). The magnetization was measured using vibrating sample magnetometry at room temperature. The color strength (*K/S*) of treated fabric with IONPS was analyzed by using Konica-Minolta-CM-3600A spectrophotometer. Wettability of untreated and D/T fabric was evaluated by calculating the time that takes to absorb one drop of distilled water dropped on the fabric. Moreover, the tensile strength of the untreated and D/T fabrics was measured by using Mecmesin-AFG-1000N instrument for three repeated measurements, and the mean value was taken. AATCC TM 61(2A)-1996 ("American Association of Textile Chemists and Colorists, AATCC 61(2A): Colorfastness to laundering, Research Triangle Park, NC, USA ", 1996) method was used to evaluate the durability after five washing cycles. The uptake of the dye by the polyester fabric was measured by using UV/VIS spectrophotometer-model: Alpha -1860. The toxicity of D/T fabrics was tested according to the MTT assay protocol.

Results and Discussion

Synthesis of iron oxide nanoparticles

As reported previously (Harifi & Montazer, 2014); IONPs were formed after blending the iron salts with sodium hydroxide solution. Where the reactions occurring to form IONPs are as follows (Harifi & Montazer, 2014):

$$Fe^{2+} + 2OH^{-} \rightarrow Fe(OH)_2 \tag{1}$$

$$Fe^{3+} + 3OH^{-} \rightarrow Fe(OH)_3 \rightarrow FeOOH \tag{2}$$

$$Fe(OH)_2 + 2FeOOH \rightarrow Fe_3O_4 + 2H_2O \tag{3}$$

$$\text{Polyester} - COO - \text{Polyester} + OH^{-} \rightarrow \text{Polyester} - COO^{-} + HO - \text{Polyester} \tag{4}$$

$$2\text{Polyester} - COO^{-} + Fe^{2+} \rightarrow \text{Polyester} - COO^{-}Fe^{2+^{-}}OOC - \text{Polyester} \tag{5}$$

The synthesis procedures take place at high pH value, during which hydroxylation of Fe^{2+} and Fe^{3+} species was obtained simultaneously [reactions (1) and (2)] (Hribernik et al., 2012) carrying out the process at boiling for 60 min. This process allowed enough time for the evolution of magnetite (Fe_3O_4) nucleus to complete. Whereas the average pH for the synthesis of magnetite NPs is ~11, (Faiyas et al., 2010). We adjusted the pH at 12, as reported in Faiyas et al. (2010); the evolution of magnetite was carried out at a pH higher than 11. Also, the carboxyl ($-COO^{-}$) functional group on polymeric chains of polyester fibers was generated in alkaline solution as in the Eq. (4). The iron ions with positive charge can subsequently absorb on negative groups created on polyester fabric by ionic interactions [reaction (5)]. The ionic interactions and hydrogen bonding with functional groups of polymeric substrates were mainly remarked for adsorption of nanoparticles on the surface in nano-functionalization of polyester fabrics (Rezaie & Montazer, 2019).

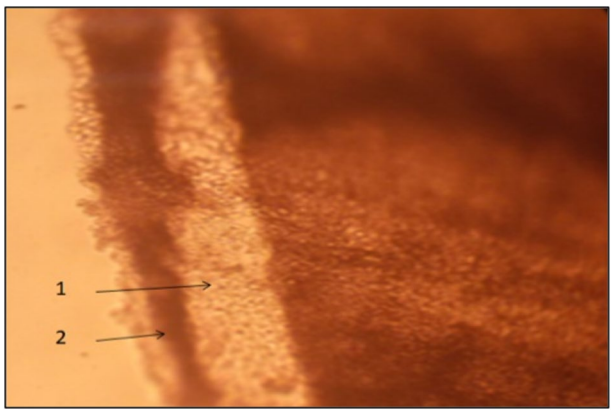

Fig. 2 Viability of normal human cell on wi38: **1**; localization of cells on D/T fabric in circular morphological shape without shrinkage or granulation, **2**; D/T fabric

Table 1 O.D, toxicity and viability of normal human cell on D/T fabric

ID	Mean of optical denisity (O.D)	Standard error (ST.E)	Viability %	Toxicity %
wi-38	0.25	0.00	100	0
D/T fabric	0.25	0.01	99.73	0.26

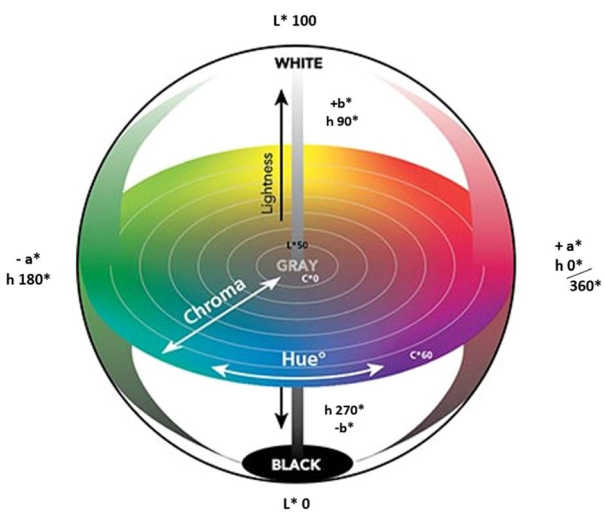

Fig. 3 L*a*b* and L*C*h colour space

Cytotoxicity test on D/T fabric

The cytotoxicity of D/T fabric was evaluated against healthy human cells (wi-38) by the MTT assay. Figure 2 and Table 1 show that the viability of cells of D/T fabric was 99.7% of the negative control. However, the average relative cell viability was over 70%, as mentioned in (Kangwansupamonkon et al., 2009), and it was concluded that the D/T fabrics have low toxicity toward human skin. Which leads to the use of these fabrics in the medical fields.

Table 2 Optical measurements of dyed PET and D/T PET fabrics

Type	Sample	Colour parameters					
		L*	a*	b*	C*	h	*K/S*
Dyed PET fabric		80.2	8.9	77.2	77.7	83.4	14.0
D/T PET fabric		37.9	5.9	19.1	19.9	72.9	16.9

Where *K/S* is refere to color strength

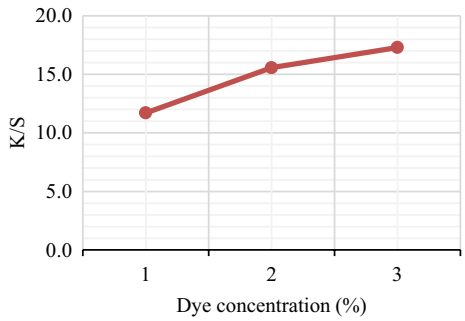

Fig. 4 Effect of in situ application with different dye concentrations on colour strength (*K/S*)

Colorimetric study

The colour parameters of the dyed and D/T polyester fabrics were analyzed by using the (Konica Minolta spectrophotometer CM-3600A). Figure 3 and Table 2 exhibit the L*a*b* values for each fabric, where (L*) values represent colour lightness, (a*) is the red/green coordinate, and (b*) is the yellow/blue coordinate (Khalifa et al., 2015), we can objectively determine that the fabrics do not match in colour. These values tell us that D/T fabric is darker according to the colour lightness values L*, greener and less blue according to a*, b* values respectively than Dyed fabrics. If we put the values of $\Delta L^* = +42.28$, $\Delta a^* = +3.11$, and $\Delta b^* = +58.09$ into the colour difference equation, it can be determined that the total colour difference between the two fabrics is ~ 71.9.

$$71.9 = \sqrt{(42.28)^2 + (3.11)^2 + (58.09)^2}$$

Effect of in situ application with different dye concentrations on color strength (K/S)

Figure 4 shows that increasing dye concentration from 1 to 3% (owf) lead to an increase in dye uptake. The maximum *K/S* value was reached at 3% concentration of the dye. This result referred that by increasing the dye concentration, the dye diffusion into fabrics increased (Elmaaty et al., 2018a). Further increase in dye concentration has no effect on the *K/S* value. This may be attributed to over saturation of the fabric by dye and NPs (Abou Elmaaty et al., 2020).

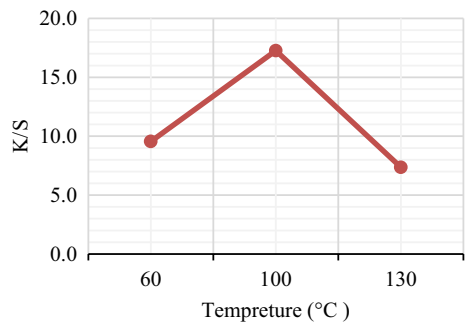

Fig. 5 Effect of temperature of in situ application on colour strength (*K/S*)

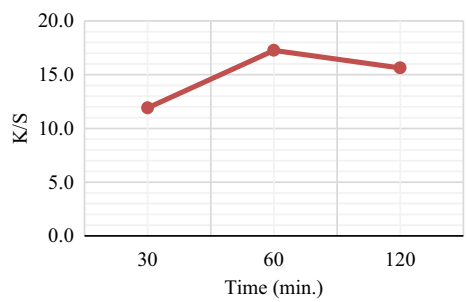

Fig. 6 Effect of time of in situ application on colour strength (*K/S*)

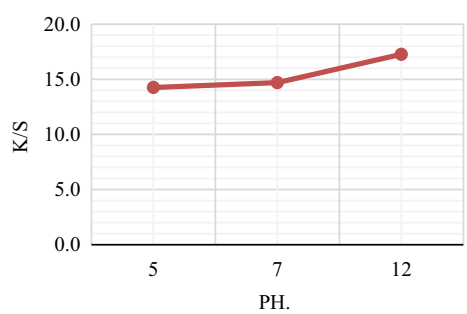

Fig. 7 Effect of pH on colour strength (K/S)

Effect of temperature and time of in situ application on color strength (K/S)

The effect of in situ application settings on (*K/S* values) was investigated. Various temperatures (60, 100, and 130 °C) and durations (30, 60 and 120 min.) were applied on polyester fabrics with dye and IONPs, and the results were shown in Figs. 5 and 6. It can be concluded from Fig. 5 that; raising the temperature from 60 to 100 °C increased the *K/S* from (9.6 to 17.3), this may be referred to the high distribution rate and the raise of the kinetic energy of the dye molecules. Thus 100 °C was selected as an optimum temperature for the application. Above optimum temperature, the rate of dyeing is reversed, and the dye may not stabilize onto fabrics resulting in reducing the *K/S* and reducing the fastness properties (Adeel et al., 2018). Otherwise, the maximum *K/S* value, 17.3 has been recorded after 60 min in Fig. 6. Increasing the application time above 60 min caused a reduction in the *K/S* values. This result may be referred to the fact that the

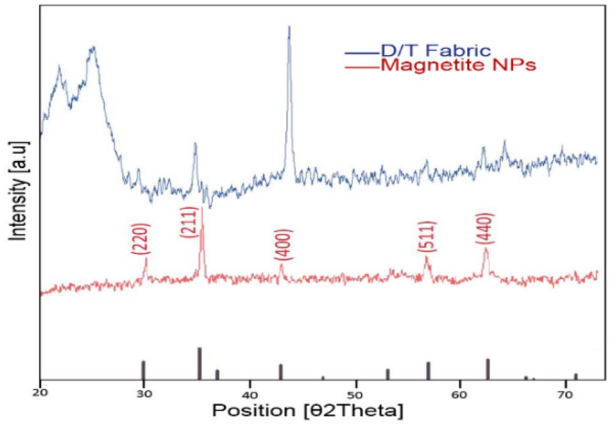

Fig. 8 The XRD pattern for the obtained magnetite on fabric

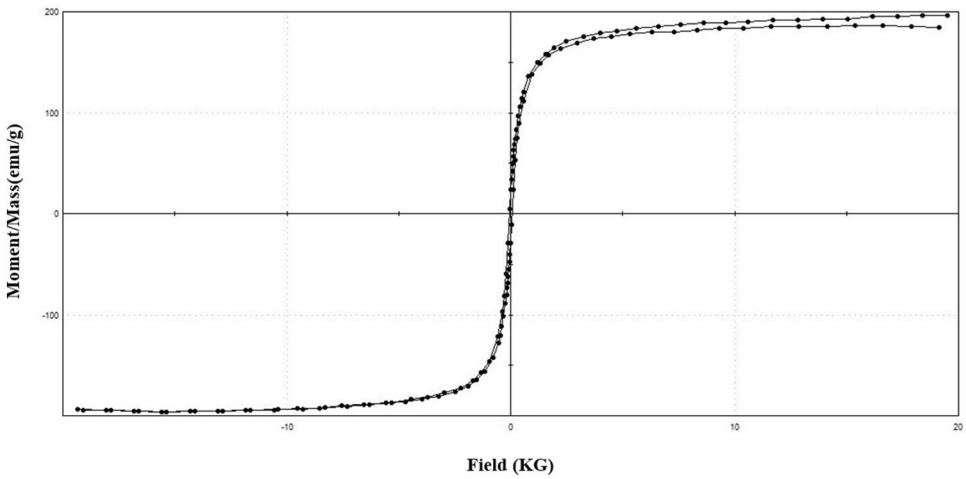

Fig. 9 Magnetization curve of D/T polyester fabric

heating for a long time may cause stripping and equilibrium are favoured towards the treatment bath (Adeel et al., 2018).

Effect of pH on color strength (K/S)

Figure 7 showed that the (*K/S*) of D/T polyester fabric was increased by increasing the pH value of the treatment bath from pH 5 to 12. The better result and the maximum uptake of dye and IONPs were gained at pH 12. This may be referred to the growth of Fe_3O_4 nuclei which was occurred at pH higher than 11 (Faiyas et al., 2010). In addition; it can be suggested that the alkaline hydrolysis of polyester causes the formation of polyethylene glycol and through the oxidation of hydroxyl groups to aldehyde groups in ethylene glycol, carboxylate ions was produced. This increases the tendency of fabric surface toward IONPs (Allahyarzadeh et al., 2013).

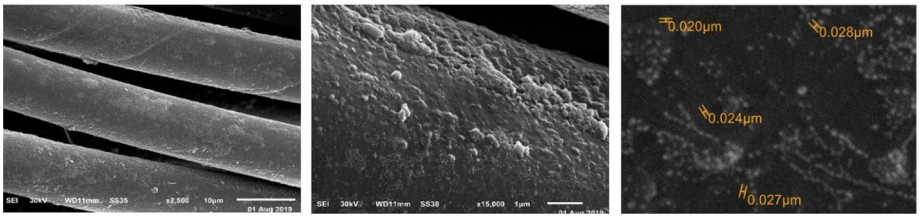

Fig. 10 SEM images of D/T fabric

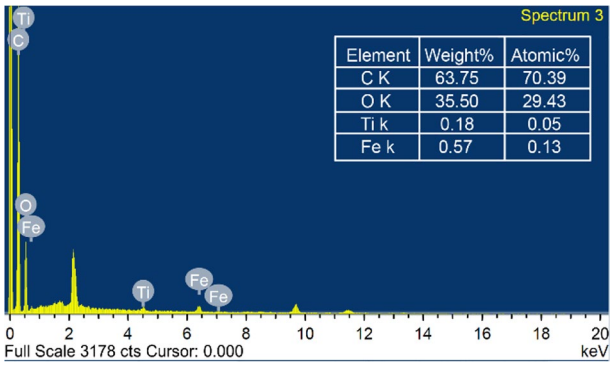

Fig. 11 EDX spectra showing iron-on D/T fabric

XRD results

Figure 8 showed XRD pattern of synthesized IONPs on treated polyester fabrics exhibiting peaks at 2Θ at 30.1, 35.5, 42.6, 53.6, 57.0 and 62.8 which can be assigned to diffraction of the (220), (311), (400), (422), (511), and (440) planes, respectively of spinal structured Fe_3O_4 nanoparticles. Accordingly, we can conclude that the resulted fabric had perfect magnetic responsivity (Xu et al., 2016).

VSM results

Figure 9 shows the relationship between the applied magnetic field (H) and the induced magnetization (M) of the D/T fabric exhibiting a sharp increase in magnetization by increasing the applied field from 0 to 20 KG.

SEM and EDX analysis

The IONPs were uniformly distributed on the surface of the D/T fabric, as seen in Fig. 10. The synthesized IONPs are 20–28 nm and roughly agglomerated. Figure 11 confirmed the successful synthesis of the IONPs on the D/T fabric by using EDX to analyze the chemical compositions. The figure also showed that iron and oxygen were two elements in the D/T fabric regardless of the carbon that was bound to the polyester substrate.

Antibacterial efficiency

To determine the antibacterial activity of the D/T fabrics against *Staphylococcus aureus* and *Bacillus cereus* as a gram-positive bacterium and gram-negative bacterium (*E. coli*); the AATCC T M 100-2004 method was used (Test method for antibacterial finishes on

Table 3 Percentage of bacterial reduction on D/T polyester fabric

Type	*E. coli* count	R (%) *E. coli*	*S. aureus* count	R (%) *S. aureus*	*B. cereus* count	R (%) *B. cereus*
Control	215×10^3	–	1079×10^3	–	975×10^3	–
D/T PET fabric	182×10^3	15	520×10^3	52	284×10^3	71

where *S. aureus* referred to *Staphylococcus aureus* and *B. cereus* referred to *Bacillus cereus*

textile materials, 2004). And according to Eq. (6); the results were reported as percentages of bacteria reduction.

$$R = (A - B)/A * 100 \tag{6}$$

where R is the reduction percentage of bacteria colonies, A and B are the numbers of bacteria colonies recovered from the blank and D/T fabric, respectively after inoculation and incubation. The antibacterial efficiencies of the D/T fabrics against *Staphylococcus aureus*, *Bacillus cereus* and *E. coli* bacteria were 52, 71 and 15%, respectively as shown in Table 3. The reaction between bacteria and IONPs causes the death of bacteria (Shahidi et al., 2018) as a result of changing the metabolic activity of bacteria. IONPs were promising agents for antibacterial applications (Harifi & Montazer, 2014) due to its unique characteristics. Also; by increasing the iron nanoparticles on polyester fabric the magnetic field increases, which is one of the distinctive characteristics of it, which in turn increases the bactericidal action causing bacteria cell death (Gudkov et al., 2021).

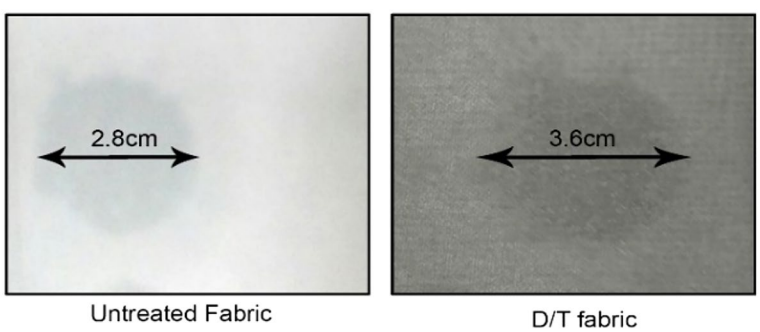

Fig. 12 Length pf diameter of water droplet for untreated fabric and D/T fabric

Table 4 Fastness properties of D/T polyester fabric before and after (5 washing cycles) using overall optimum conditions

Sample	Wash fastness		Rubbing fastness		Light fastness
	St	Alt	Dry	Wet	
Blank	–	–	–	–	–
D/T sample	5	5	4–5	4	4–5
D/T sample after (5 washing cycles)	4–5	4–5	4–5	3–4	3–4

Table 5 Tensile strength (maximum force) of blank and of D/T polyester fabrics

Sample	Tensile strength Force, N
Blank	194.00
D/T sample	287.00

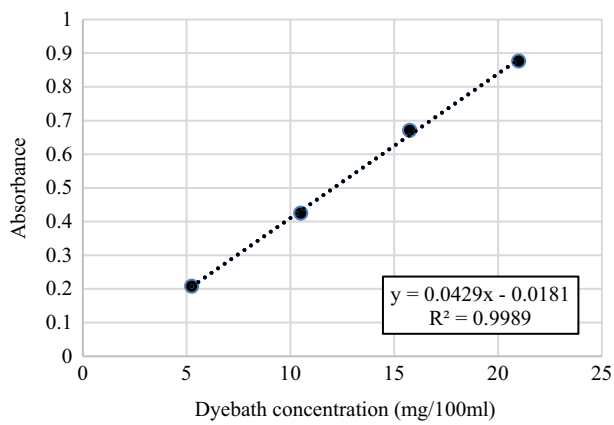

Fig. 13 Calibration curve for dye at known concentrations and corresponding absorbance

Water drop test

The hydrophobicity of untreated and D/T fabrics was measured by the water drop test. A controlled size drop was placed at a constant rate onto the surface of the fabric, and the time required for penetration was calculated. The uptake times have been taken for untreated and D/T polyester fabrics. It has been found that the time spent to absorb one water droplet on the untreated polyester fabric was 6 s, however this time for D/T fabric reduced to 4 s. The surface wettability of the D/T fabrics was enhanced with minor etching effects as a result of surface oxidation of the D/T fabrics.

Also, Fig. 12 reveals the length of water droplet diameter after absorption and spreading in the time frames that were mentioned before for both fabrics.

Fastness properties

By using overall optimum conditions, the durability of D/T polyester fabric was assessed in terms of color fastness mainly washing, rubbing, and light. As seen in Table 4; the wash and rubbing fastness of D/T polyester fabric were excellent and very good, respectively, even after five washing cycles. As for light fastness, moderate values were observed even after five washing cycles. This result may be attributed to the photo fading behaviors of disperse dyes (Suesat & Suwanruji, 2011).

Tensile strength

According to the results of the tensile strength (maximum force), which was listed in Table 5, it can be concluded that the introduction of IONP into the structure of polyester fabric improved the tensile strength of the treated fabric, possibly due to the ionic

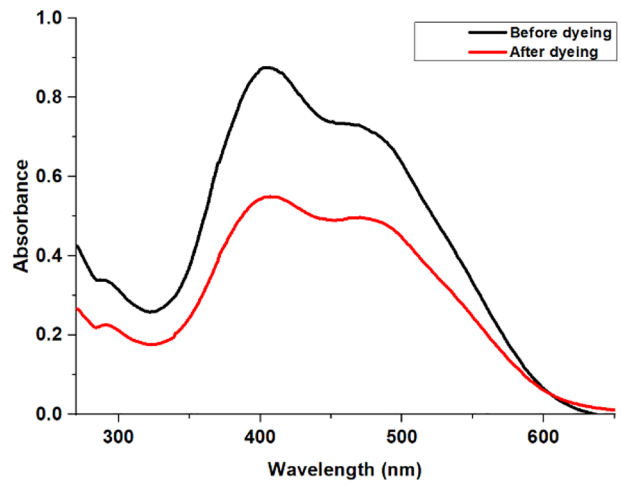

Fig. 14 Absorbance of dye bath before and after dyeing

interactions and hydrogen bonding between IONPs and polyester fabric (Rezaie & Montazer, 2019) and due to the filling of any pits that can result from the hydrolysis of polyester by synthesized IONPs resulting in a uniform distribution of load on the fabric surface (Harifi & Montazer, 2014).

Measurement of dye exhaustion

The dyebath solution was sampled before and after dyeing to measure the dye exhaustion. The solution of the dye was diluted 20-fold with 85% aqueous DMF. Moreover, the absorbance of the dye solution was measured by using UV/VIS spectrophotometer-model: Alpha -1860. A calibration curve with known dilution degrees of the dye concentration in 80% aqueous DMF (Fig. 13) was also used. The dye exhaustion is calculated by using Eq. 7:

$$\%E = \left[1 - \left(\frac{C2}{C1}\right)\right] * 100 \tag{7}$$

where (*%E*) is the percentage of dye exhaustion and C1, C2 are the dye concentrations in the solutions before and after dyeing, respectively. Figure 14 has shown the absorbance of dye bath concentration before and after exhaustion by polyester fabric. The decrease in absorbance is due to the increase of dye exhaustion by polyester fabric, which means that the dye and iron nanoparticles have been absorbed by polyester fabric and the treatment was occurred.

Conclusions

This work discusses a new approach of colouring MIONPs and implementing the fabric with multifunctional properties in one step by using in situ technique. The optimum condition of the treatment has been controlled by adjusting the *pH* at 12, treatment time for 60min., treatment temperature at 100 °C and IONPs concentration of 3% owf. The results of the SEM, EDX and XRD measurements confirmed the successful synthesis of IONPs with size around of 20–28 nm as well as the uniformty of color distribution on

polyester fabrics. Moreover, the colored polyester fabric acquired excellent color fastness properties, their noticeable improve in antibacterial activity against *Bacillus cereus* (with 71% of reduction of bacterial growth), low toxicity of 0.26% against wi-38 cell line and the increase in magnetization pointed to an eco-friendlier approach in textile dyeing and multifunctionalization. Also, the un-attached coloured magnetic IONPs can be collected, re-concentrated and reused again by using a magnet in a simple, cost-effective, and environmental-friendly way for the wastewater treatment process.

Acknowledgements
Not applicable.

Author contributions
TAE conceived the original idea of this study and participated in design and coordination as well as manuscript drafting. SAE, SAA and SH prepared the iron NPs and interpreted the results obtained from VSM, SEM, XRD analyses, antibacterial and water drop tests as well as colorimetric and mechanical properties study. SAA and SH participated in the manuscript writing. SH was responsible for the dyeing experiments and samples preparation. SAA and SH participated in the iron NPs preparation and measured the color strength and fastness of the dyed fabrics.

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Availability of data and materials
The datasets generated and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests
The authors declare that they have no competing interest.

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Publisher's Note

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