

Sharif University of Technology Scientia Iranica

> Transactions F: Nanotechnology www.scientiairanica.com



TiO_2 /hematite or magnetite/Ag nanoparticles synthesized on polyester fabric at various temperatures producing different superparamagnetic, self-cleaning and antibacterial textiles

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Received 5 August 2014; received in revised form 20 October 2014; accepted 1 December 2014

KEYWORDS

TiO₂/hematite or magnetite/Ag nanoparticles; Polyester; Super-paramagnetism; Antibacterial; Self-cleaning; Alkaline hydrolysis.

Abstract. An innovative TiO_2 /iron oxide/Ag nanoparticle, with crystal size of about 25 nm, was synthesized on polyester fabric to achieve different multi-features, including super-paramagnetism, antibacterial and enhanced self-cleaning properties. Treatment temperature was varied at boiling and 130° C, creating TiO₂/magnetite/silver and TiO_2 /hematite/silver nanoparticles, respectively. The alkaline hydrolysis of the polyester surface was accompanied by nanoparticle synthesis, which forms a more active surface for nanoparticle adsorption. The co-operation of iron oxide and silver nanoparticles on the TiO₂ surface synergistically improved the self-cleaning efficiency of titanium dioxide nanoparticles by separation of electron-hole pairs, three and four times, for fabrics treated with $TiO_2/Fe_3O_4/silver$ and TiO_2/α -Fe₂O₃/silver nanoparticles, respectively, compared with their corresponding control samples. Moreover, both TiO₂/iron oxide/Ag nanoparticle treated samples indicated an antibacterial efficiency of $\geq 99.99\%$ against Staphylococcus aureus bacteria. Findings suggested that the developed magneto, bio and photo activities could be idealized, depending on the end use of the treated fabrics, through incorporation of different iron oxides in the prepared nanoparticles; magnetite providing the highest saturation magnetization and hematite the best self-cleaning toward degradation of methylene blue under sunlight irradiation.

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1. Introduction

Over the years, imparting multi-functions into textiles without detrimental effects on their inherent properties has been an interesting research field. New and innovative intelligent textiles with potential technological applications for the military, healthcare, space exploration, sports, and consumer fitness fields have been designed. Conductive fibers, energy storage fabrics and wearable electromagnetic wave shielding textiles are some of the recent findings [1-10].

With the growing role of nanotechnology in textiles, research groups all around the world have obtained a variety of textile materials with multifunctional properties through incorporation of nanomaterials. During the last decade, Montazer and his research group have done a vast number of studies on the synthesis of different nanoparticles for imparting multi-functional properties into textiles, including selfcleaning, water repellency, flame retardancy and antibacterial abilities [11-21].

The potential of nano TiO_2 for imparting multi-

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functional properties to different textile materials has been reported in several recent studies. Reducing the photo yellowing of wool, obtaining self-cleaning wool fabrics, nano photo-scouring and nano photobleaching of cotton and wool fabrics, enhancing nano TiO_2 adsorption on a blend of wool/polyester by bio-finishing, polyester fabrics with simultaneous selfcleaning, antimicrobial and UV protection properties, free carrier dyeing of nano TiO_2 treated polyester fabric, mothproofing of wool fabric by treatment with nano TiO_2 , and sono-synthesis of TiO_2 nanoparticles on cotton and wool fabrics producing self-cleaning and UV protection properties are some of the major applications of nano TiO_2 particles in textiles [11-21].

In addition to the broad range of properties imparted into textiles using TiO₂ nanoparticles, $metal/TiO_2$ nanoparticles have been extensively studied as a promising approach to overcoming problems encountered with TiO_2 [22,23]. Reducing the possibility of electron-hole recombination, enhancing the absorption of visible light, increasing the rate of electron transfer to the oxidant and improving the photocatalytic activity of TiO_2 has been confirmed by deposition of Ag nanoparticles on TiO_2 [22,23]. Besides, the addition of silver increases the antibacterial activity of TiO_2 [22]. Ag/TiO₂ nanoparticles have been in-situ or ex-situ synthesized on different textiles providing multi-functional properties [24-27]. Moreover, core/shell iron oxide/ TiO_2 dual-semiconductors have been prepared as magnetically separable nanocatalysts with low photoactivity [28,29]. Also, incorporation of textiles with magnetic nanoparticles, such as maghemite (γ -Fe₂O₃), hematite (α -Fe₂O₃) and magnetite (Fe_3O_4) , will create new properties, including magnetism and electromagnetic wave shielding [30].

In this research, the idea of synthesizing $TiO_2/iron oxide/Ag$ nanoparticles on polyester fabric is considered, aiming to develop a fabric with multifunctional activities. The synergistic effect of iron oxide and silver on a TiO_2 surface is beneficial for enhancing the photocatalytic and antibacterial activities of TiO_2 .

2. Experimental

2.1. Materials and methods

A 100% polyester fabric with weight of 88 g/m² and wrap/weft density of 31/21 yarn/cm from Hejab Co., Shahr-e-Kord, Iran, and ferric trichloride (FeCl₃), ferrous sulfate (FeSO₄.7H₂O), silver nitrate (AgNO₃) and sodium hydroxide (NaOH) from Merck, Germany, were provided. Nanosized TiO₂ (Degussa P25) was employed as a photocatalyst and purchased from Evonik, Germany. The chemicals were of reagent grade used without further purification.

Prior to the treatment, the fabric was washed with 1 g/L nonionic detergent at 60°C for 20 min and was

rinsed with distilled water to remove any impurities. The polyester fabric was treated in a bath containing TiO₂, AgNO₃, FeCl₃, FeSO₄.7H₂O (Fe²⁺/Fe³⁺ molar ratio=2) and NaOH (pH=12), and the volume of the bath was kept at 100 mL with distilled water. The process was carried out at 100°C and 130°C for 1 h, preparing TiO₂/Fe₃O₄/Ag and TiO₂/ α -Fe₂O₃/Ag nanoparticles, respectively. Two other polyester fabric samples were only treated with distilled water under pH=12 at boil and 130°C, and considered as 100-control and 130-control samples. Finally, the treated fabrics were washed with water for 10 min, and dried at room temperature.

2.2. Characterization tests

X-Ray Diffraction analysis (XRD) was performed with a Siemens D5000 X-ray diffractometer using a Cu Ka radiation source (1.5418 °A) operating at 40 kV to recognize the crystalline size and phases of the synthesized nanoparticles on the polyester fabric. Energydispersive spectroscopy (EDX) was used to characterize the elemental composition of the treated fabrics. The surface morphology of the treated samples and particle size of the synthesized nanoparticles were analyzed using a scanning electron microscope (MIRA, Tescan). The quantitative antibacterial efficiency of the treated fabric was also determined against Staphylo*coccus aureus* as a gram-positive bacterium, according to the AATCC-100 test method. Magnetization was measured using a Vibrating Sample Magnetometer (VSM) at room temperature. In order to evaluate the alteration in wettability of the treated samples, the contact angle between water and the fabric surface was calculated, based on the plate method, by Tensiometer Kruss K100-SF (Germany). The fabric was suspended from a balance using a sample holder. Water was moved upwards. As soon as contact was detected, the measurement of the force started, and, using the equation for the plate method (Eq. (1)), the contact angle was calculated from the surface tension of water (σ) , the wetted length (L) and the measured force (F):

$$\cos\theta = \frac{F}{L.\sigma}.\tag{1}$$

3. Results and discussion

3.1. Synthesis of $TiO_2/iron \ oxide/Ag$ nanoparticles

The alkaline condition of the preparation procedure was adjusted by NaOH as a key role in the synthesis of nanoparticles to enhance the surface activity of polyester. Scheme 1 shows the possible reactions involved in the preparation of nanoparticles on polyester fabric.

The hydroxyl ions attack the polyester surface,

$H-[OCH_2CH_2-O-CO(C_6H_4)-CO]_n-OH+OH^-\longrightarrow HOCH_2CH_2OH+H_2O+H-[OCH_2CH_2-O-CO(C_6H_4)-CO]_{n-1}-OH^+O-CO(C_6H_4)-COO-$	(1)
$HOCH_2CH_2OH + Ag^+ \longrightarrow CH_3CHO + Ag^0 + H_2O$	(2)
$\mathrm{Fe}^{2+}+2\mathrm{OH}^{-}\longrightarrow\mathrm{Fe}(\mathrm{OH})_{2}$	(3)
$\mathrm{Fe}^{3+}+3\mathrm{OH}^{-}\longrightarrow\mathrm{Fe}(\mathrm{OH})_{3}\longrightarrow\mathrm{Fe}\mathrm{OOH}+\mathrm{H}_{2}\mathrm{O}$	(4)
$3 \text{Fe}(\text{OH})_2 + 1/2 \text{ O}_2 \longrightarrow \text{Fe}(\text{OH})_2 + 2 \text{FeOOH} + \text{H}_2 \text{O}$	(5)
$Fe(OH)_2 + 2FeOOH \xrightarrow{boil} Fe_3O_4 + 2H_2O$	(6)
$2 FeOOH \xrightarrow{130^{\circ}C} \alpha - Fe_2O_3 + H_2O$	(7)

Scheme 1. Mechanism of TiO₂/iron oxide/Ag nanoparticles synthesis on polyester fabric.

producing hydroxyl (-OH) and carboxyl (-COO⁻) end groups, by removing the low molecular segment of the polymer chains creating pits on the fiber surface [25]. Therefore, the polyester surface provides sites for bonding of the nanoparticles with the polymer, and some of the synthesized nanoparticles could be entrapped into the generated pits. Through the alkaline hydrolysis of the polyester fabric, ethylene glycol and terephthalate anions were formed [25], which could be further reduced by silver ions into silver nanoparticles. Moreover, iron oxide nanoparticles were formed through mixing iron salts with hydroxyl ions [31]. The applied temperature was effective in producing the black precipitates of magnetite or aqueous phase transformation into redbrown precipitates of hematite. The phase transformation from FeOOH to α -Fe₂O₃ occurs through the dissolution precipitation mechanism in aqueous medium [31].

Under pH=12 and considering the isoelectric point of TiO₂ (6.2) [32], the electrostatic attraction between oppositely charged surfaces, promotes synthesis of silver nanoparticles and iron oxides on the TiO₂ surface [25].

3.2. XRD

Figure 1(a) and (b) show the XRD spectra of the polyester fabrics treated at boil and 130°C. The successful synthesis of TiO₂/Fe₃O₄/Ag nanoparticles on the polyester fabric can be confirmed by characterization peaks at $2\theta = 25.4^{\circ}$ (TiO₂ anatase), 35° (magnetite) and 38° (silver), in addition to the main peak of the original polyester fabric at $2\theta = 17^{\circ} - 20^{\circ}$ (Figure 1(a)) [25,31]. Moreover, the peak at $2\theta = 33.1^{\circ}$ indicated the presence of hematite in the TiO₂/ α -Fe₂O₃/Ag treated sample (Figure 1(b)) [31]. Using Scherrer's equation, and from the width of the peak at 2θ angle 25.4°, the crystallite size of the prepared nanoparticles was about 25 nm.

3.3. SEM and EDX analysis

In comparison to the smooth surface of the untreated sample (Figure 2(a)), a number of pits were observed on the surface of the samples treated with sodium hydroxide at boil and 130°C (Figure 2(b) and (c)). Deeper pits were detected on the sample 130-control, as



Figure 1. XRD spectra of a) $TiO_2/Fe_3O_4/Ag$, and b) TiO_2/α -Fe₂O₃/Ag treated fabrics.

a result of alkaline hydrolysis under vigorous condition at $130\,^{\circ}$ C.

The synthesized $\text{TiO}_2/\text{Fe}_3\text{O}_4/\text{Ag}$ nanoparticles with an average size of 50 nm are uniformly distributed on the surface of the treated fiber at boil, and are aggregated (Figure 3(a)). A higher temperature used in the synthesis of TiO_2/α -Fe₂O₃/Ag led to more growth of nanoparticles, and created nanoparticles with an average size of 80 nm (Figure 3(b)).

The successful synthesis of nanoparticles on the treated samples was further confirmed by the chemical compositions analyzed by EDX, as shown in Figure 4. Titanium, silver, iron and oxygen are the main elements on the treated fabrics, apart from the carbon relating to the polyester substrate.

3.4. Self-cleaning

In the textile field and in the area of the selfcleaning properties of textiles, the color difference of the stained sample, before and after light irradiation, is usually considered the criteria for evaluation of the photodegradability of the treated fabric, called a self-



Figure 2. SEM images of a) untreated, b) 100-control, and c) 130-control fabrics. (Images are at 25000× magnification.)



Figure 3. SEM images of a) $TiO_2/Fe_3O_4/Ag$, and b) TiO_2/α -Fe₂O₃/Ag treated fabrics. (Images are at $6000\times$, $25000\times$ and $50000\times$ magnifications from top to bottom.)



Figure 4. EDX spectra of a) $TiO_2/Fe_3O_4/Ag$, and b) TiO_2/α -Fe₂O₃/Ag treated fabrics.

cleaning activity. The color difference can be calculated in $L^*a^*b^*$ or RGB color space.

Here, the self-cleaning property was determined by staining the fabrics with 0.05% methylene blue solution. The stained samples were exposed to sunlight irradiation for 24 h and color differences were evaluated by image processing in RGB color space, according to Eq. (2):

$$\Delta RGB = \left[(R_2 - R_1)^2 + (B_2 - B_1)^2 + (G_2 - G_1)^2 \right]^{1/2}, \qquad (2)$$

where $R_2G_2B_2$ and $R_1G_1B_1$ are color coordinates of samples after and before irradiation, respectively.

Photocatalytic activity of TiO₂ treated textiles has been attributed to the electron-hole pairs generated by light irradiation [10-21]. Silver and iron oxide nanoparticles on the surface of TiO₂ retard the electron-hole recombination and boost their separation rate [25]. Silver nanoparticles trap the excited electrons, and act as electron donors to O₂. Moreover, the interfacial charge transfer to the reducing or oxidizing species completed the electron-



Figure 5. a) ΔRGB of control and treated fabrics. b) Self-cleaning activity of control and treated fabrics stained with methylene blue solution under sunlight irradiation.

hole recombination reaction in the presence of iron oxide on the TiO_2 surface. In comparison to the control samples, the ΔRGB of the treated fabrics was higher, corresponding to the enhanced self-cleaning properties (Figure 5). In this regard, fabric treated with TiO_2/α -Fe₂O₃/Ag was more effective in degrading the Methylene Blue stain (greater ΔRGB), due to the higher temperature of the treatment $(130^{\circ}C)$, possibly, resulting in more nanoparticles adsorbed on the polyester surface. Also, the Fe^{2+}/TiO_2 and Ag/TiO_2 ratio in the prepared nanoparticles had a prominent impact on the photocatalytic activity of the treated fabrics. At high Fe or Ag concentration, the surface of the TiO_2 particles was fully covered by silver or iron oxide nanoparticles, resulting in an optical screening of the titanium dioxide phase and an increased reflection of incident light, consequently, a lower catalytic activity of prepared nanoparticles.

In order to study the self-cleaning activity of textiles, samples are usually stained with dyes or coffee stains [15,33,34]. In this study, methylene blue dye has been used as a model compound for evaluating the photodegradability of the TiO₂/iron oxide/Ag treated fabrics. Considering the photocatalytic activity of the prepared nanoparticles, which has been confirmed against methylene blue stain, it is predictable that the treated samples are capable of degrading other stains, including other dyes and coffee, however, with different Δ RGB values and under different time irradiations.



Figure 6. Room-temperature magnetization curves of a) $TiO_2/Fe_3O_4/Ag$, and b) TiO_2/α -Fe₂O₃/Ag treated fabrics. X axis: magnetic field (H:Oe), Y axis: magnetization (M:emu/g).

For more clarity, Figure 5(b) shows the visual indication of the self-cleaning activity.

3.5. Magnetization

The magnetization curves of $TiO_2/Fe_3O_4/Ag$ and TiO_2/α -Fe₂O₃/Ag treated fabrics were measured using a Vibrating-Sample Magnetometer (VSM) at room temperature and the graphical representation of the relationship between the applied magnetic field (H)and the induced magnetization (M) is shown in Figure 6. Increasing the applied field led to a sharp increase in magnetization, saturating at 6.16 (emu/g)and 0.1 (emu/g) for $TiO_2/Fe_3O_4/Ag$ and TiO_2/α - Fe_2O_3/Ag treated fabrics, respectively. A narrow magnetic hysteresis loop with extremely small coercivity and remanence values was observed, indicating the super-paramagnetic property of the treated samples. The TiO_2/α -Fe₂O₃/Ag treated fabric was less magneto-active due to the presence of hematite as a weaker magnetic nanoparticle, in comparison to magnetite [31].

3.6. Antibacterial efficiency

The quantitative antibacterial efficiency of the treated fabrics was determined against *Staphylococcus aureus*



Figure 7. Antibacterial activity of treated fabrics against Staphylococcus aurous.

using Eq. (3):

$$R\% = \left[\frac{A-B}{A}\right] \times 100,\tag{3}$$

where R is the reduction percentage, and A and B are the number of bacterial colonies from control and treated samples.

In order to evaluate the effect of silver and iron oxide nanoparticles on enhancing the antibacterial efficiency of TiO₂ nanoparticles, two samples were only treated with nano TiO₂ particles at boil and 130°C and inoculated with *Staphylococcus aureus*. While samples treated with titania particles were not successful in completely killing the bacteria (bacteria colonies can be detected in the plates), fabrics treated with TiO₂/iron oxide/Ag nanoparticles showed no growth of bacteria (Figure 7). Hence, according to the dilution of 1 : 100, the fabrics reduced the bacterial count to more than 4 log units and their antibacterial activity is $\geq 99.99\%$.

In this study, nano TiO_2 particles were used and silver and iron oxides were synthesized on their surface, enhancing photocatalytic and antibacterial efficiency. Therefore, the potentiating effect of iron oxide and silver caused the complete killing of the bacteria. Iron oxide nanoparticles possess unique characteristics that make them promising agents for antibacterial applications. This has been attributed to their binding to bacterial cell walls causing membrane disruption. Besides, the antibacterial efficiency of silver nanoparticles is due to the metal ions, and their small particle size and high specific surface area. Damage to the lipids, proteins and DNA of microorganisms are the main antibacterial mechanisms [31,35].

${\bf Sample}$	Contact angle $(^{\circ}\theta)$	
	Before	After
	$\mathbf{sunlight}$	$\operatorname{sunlight}$
$\operatorname{Control}$	59	59
$100 ext{-control}$	34	34
$130 ext{-control}$	0	0
${ m TiO_2/Fe_3O_4/Ag}\ { m treated}$	113	42
${ m TiO_2/\alpha}$ -Fe ₂ O ₃ / Ag treated	103	35

 Table 1. Contact angle of the control and treated fabrics

 before and after sunlight irradiation.

3.7. Wettability

Considering the photo-induced wettability of textiles treated with photocatalysts, the contact angle between the water and fabric surface was quantified under two conditions, before and after sunlight irradiation, for 24 h, and the results are summarized in Table 1. The contact angle of the untreated sample (0-control) reduced from 59° to 34° and 0° for 100-control and 130-control samples, respectively, due to the alkaline hydrolysis of the polyester fabric. This was more prominent in the fabric hydrolyzed at 130°C, as a result of more pits created on the surface at higher temperature, as evidenced by SEM images (Figure 2).

Before the sunlight irradiation, the in-situ synthesis of nanoparticles on the treated fabrics led to more hydrophobic surfaces, and the water contact angle reached 113° and 103° for $TiO_2/Fe_3O_4/Ag$ and TiO_2/α -Fe₂O₃/Ag treated fabrics, respectively. The hydrophobicity of iron oxide nanoparticles as a coating on surfaces has been previously discussed in the literature [36]. The air bubbles trapped in the rough surface of such coatings were reported to be the main reason for their non-wetting property [36]. In addition, due to the photo-activity of the TiO_2 nanoparticles, electrons and holes play a prominent role in changing the water absorption properties of the treated fabrics after sunlight irradiation. In this regard, the wettability of the treated fabrics increased, reaching 42° and 35° for $TiO_2/Fe_3O_4/Ag$ and TiO_2/α -Fe₂O₃/Ag treated fabrics, respectively. The hydrophilic nature of the treated fabrics, due to light irradiation, was reversible to the hydrophobic property to some extent, after storage in the dark, as similarly reported by others [37].

4. Conclusions

Ideal antibacterial activity, super-paramagnetism and self-cleaning properties were achieved by in-situ synthesis of TiO_2/iron oxide/Ag nanoparticles on polyester fabric. Remarkable synergistic effects and improved self-cleaning were observed by the addition of iron oxide and silver onto the TiO_2 surface. Saturation magnetization of 6.16 emu/g and 0.1 emu/g was obtained for $\text{TiO}_2/\text{Fe}_3\text{O}_4/\text{Ag}$ and TiO_2/α -Fe₂O₃/Ag treated samples, respectively. The obtained results revealed that desirable magneto-activity or photocatalytic ability can be accomplished by controlling the treatment temperature at boil or 130°C, resulting in a magnetite or hematite phase in the prepared nanoparticles, respectively.

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Biographies

Tina Harifi was born in 1984 and received her BS, MS and PhD degrees in Textile Engineering from Amirkabir University, Tehran, Iran, in 2007, 2009 and 2014, respectively. Her PhD thesis, under the supervision of Dr. Majid Montazer, concerned the synthesis and stabilization of TiO_2 :Ag:Fe for multi-functional finishing of polyester fabric. Her current research interests mainly include synthesis and deposition of nanoparticles on textile substrates.

Majid Montazer was born in 1965 in Tehran, Iran. He obtained BS and MS degrees in Textile Chemistry and Fiber Science from the Textile Engineering Department of Amirkabir University of Technology, Tehran, Iran, respectively and received his PhD degree, in 1996, from the Textile Institute at Leeds University, England. He is currently Associate Professor at Amirkabir University of Technology, Tehran, Iran. He has coauthored over 100 scientific articles during the last 15 years. He received the title of one of the best nano researchers of years 2011, 2012 and 2013 from the nano organization of Iran, and the best researcher of the year, in 2010, from Amirkabir University of Technology. He also received a Scopous award in 2014, as an Iranian young scientist, for meritorious contributions in the field of material science. He is currently working on the in situ synthesis of nano particles and nanocomposites on textiles in order to obtain multi-functional properties. He is also interested in medical and industrial textiles and natural products.