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Biocidal Activity of Polyester Fabrics Modified with SiO_2 NPs

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Abstract

This article devoted to modify polyester fabric with silicon dioxide nano particles using IR dyeing machine. Alkali hydrolysis of the fabric was done to facilitate the penetration affinity of SiO_2 NPs into the mentioned fabrics. The morphological structure of the treated fabrics was investigated by field-emission scanning electron microscopy (FE-SEM). Dispersive X-ray spectroscopy (EDX) of the untreated and treated polyester fabrics was measured. Fourier transformed infrared spectroscopy (FTIR) of the untreated and treated fabrics was elucidated. TEM of the used silicon dioxide nano particles was monitored. Thermogravimetric analysis (TGA) and UV protection and of the treated fabrics were studied. Antimicrobial activity of finished polyester fabrics containing SiO_2 NPs was tested against Gram-positive (*Staphylococcus aureus*), Gram-negative (*Escherichia coli*), and pathogenic fungi (*Candida albicans and Aspergillus flavus*). The results indicate that the nano-silica has an excellent effect on antimicrobial activity and good protection of both UVA and UVB even after ten washing cycles, indicating the excellent laundering durability. The performance of treated fibers to be used as medical textile was determined by radar chart area.

Key Words: Polyester fabrics, Nano- silica, antimicrobial activity and UPF protection, Radar Chart.

Introduction

Polyethylene terephthalate (PET) is one of the most commonly synthetic fibers in textile industry, due to its high strength, stretch resistance, washability, wrinkle and abrasion resistant. However, PET has undesirable properties such as pilling, static and lack of dyeability associated with its hydrophobic nature. PET fiber has a low moisture regain about 0.4 % [1, 2]. Therefore, many attempts were done to activate the polyester fibres by using many chemical compounds and techniques. It was reported that polyester fabric was modified with different concentrations of bentonite nano-particles (BNPs) using pad dry curing technique and IR dyeing machine. The effect of the (BNPs) on the colour strength of dyed polyester fabrics with both disperse and cationic dyestuffs as well as the fastness properties were significantly improved [3, 4].

It was reported that the dyeing of the treated knitted single jersey polyester microfiber (150/288) yarn count with disperse dyes and fastness properties were improved due to the treatment with nano-clay and nano-silica. The dyeing of treated polyester fabrics with disperse dyes and the fastness properties were improved [5]. Pretreated polyester fibres with chitosan in presence of binder enhances trapping of chitosan molecules within the surface of polyester fibres which is manifested by the higher colour strength [6].

Polyester fabrics treated with silicon dioxide nanoparticle in presence of binder imparted high level of surface functionalization to the fabrics. The presence of binder accelerates the dyeing properties of nano SiO_2 pretreated polyester fabrics with disperse

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dye by decreasing the dyeing temperature from 130°C to 90°C without the presence of carrier and increasing the colour strength, compared with the untreated fabrics [7, 8].

Utilization of nanoparticles in the textile industry are very interesting because they have special properties such as shape, size, surface characteristics and inner structure. Nanoparticles such as silver, silicon, titanium, and zinc oxides have been used in the functionalization of fibers and fabrics achieving significantly improved products with new macroscopic properties [9].

The incorporating of nanoparticles into synthetic fibers in the textile industry allow to solve some problems for textiles such as microorganism growth onto fibers, flammability, dyeability, resistance to ultraviolet radiation, and others. In addition, the incorporation of such nanoparticles into special ultrathin fibers is also analyzed [10].

It was found that the non-silicon dioxide improves the dyeability of polyamide and wool fabrics under the combined effects of microwave irradiation. The multi-functions of the treated fabrics with SiO_2NPs including coloration as well as fastness properties, UV protection and antibacterial activity were improved [8].

The main objective of this work is the enhancement of the antimicrobial activity and UPF of the polyester fabrics. To achieve this target nano silicon dioxide was applied to the polyester fabrics using IR dyeing machine. Some mechanical properties of treated fibres were studied.

Experimental

Material

Polyester fabrics were supplied from Misr Spinning and Weaving Co., El Mahalla El Kobra, Egypt. Silicon dioxide nano-particles (SiO_2NPs) was purchased from Sigma–Aldrich, Germany. Its average diameter was less than 21 nm, with a surface area of more than 200 m^2/g and purity of more than 99.5%. Sodium hydroxide of laboratory grade was used.

Methods

Scouring of polyester fabrics

Polyester fabrics were scoured with (2 g/L) nonionic detergent solution (Hostapal C V. from Clariant, Egypt) with a liquor ratio 1: 25, at 45 ºC, for 45 min, then the samples were rinsed twice with tap water, and dried at room temperature.

Hydrolysis of polyester fabrics with sodium hydroxide

The alkali treatment of polyester fabrics with NaOH at high temperature is a conventional process for hydrolyzing the fiber surface, increasing the fineness of the fiber, hydrophilicity, softness, and fabric comfort. In this process, the hydroxide ions attack the fabric surface and produce carboxylate ions on its surface. This reaction is presented in scheme 1.

In this method, polyester fabrics were treated with 8% wt/wt, sodium hydroxide with a liquor ratio 1:25, at 60°C for 30 min with occasional shaking. This process was done to improve the adhesion of SiO_2NPs to the smooth surface of polyester fibres. The treated samples were immediately removed and thoroughly washed with running water for complete removal of alkali [11]. The loss in weight percentage of the alkali pretreated samples were assessed according to the following equation:

$$\textbf{Loss in weight}\ (\%) = \frac{W_1 - W_2}{W_1} \times 100 \qquad (1)$$

Where W_1 is the initial weight of the polyester fabric, and W_2 is the weight of polyester fabric after pretreatment with sodium hydroxide.

Treatment of polyester fabrics with silicon dioxide (NPs) using IR dyeing machine

In this method, the pretreated polyester fabrics were treated with different concentrations of SiO_2NPs solutions (0.5%, 1% and 1.5% wt/wt) with liquor ratio 1:100 the temperature was adjusted at 130ºC for an hour, then treated samples were rinsed with running tap water and left to dry at ambient temperature. Table 1 shows the code of samples and its description.

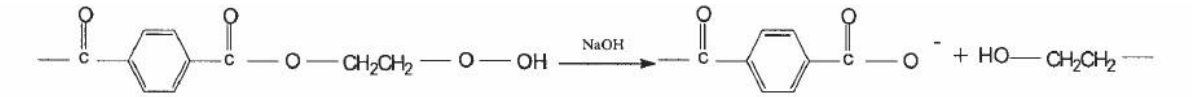
Table 1: description of the treated and untreated polyester fabrics

No. of Sample	Description
U	Untreated PET
1	PET treated with 8% NaOH
2	Alkali treated PET with 0.5% SiO_2NPs
3	Alkali treated PET with 1% SiO_2NPs
4	Alkali treated PET with 1.5% SiO_2NPs

Analyses and Testing

Transmission electron microscopy (TEM)

The morphology of SiO_2NPs was investigated using TEM (JEOL, JEM-1230, and Japan, with an acceleration voltage of 120 kV). The sample for TEM analysis was obtained by placing a drop of the colloid



Scheme 1: Hydrolysis of polyester fibres with sodium hydroxide

dispersion onto a carbon coated copper grid. The samples were dried at room temperature and examined using a TEM without further modification or coating.

Field- Emission Scanning electron microscopy (FE-SEM) and dispersive X-ray spectroscopy (EDX)

ZEISS LEO 1530 Gemini Optics Lens scanning electron microscopy (SEM) with 30 kV scanning voltages was employed to observe the morphological structure of untreated and treated fabrics. Zeiss LEO 438 VP with Oxford Instruments EDX with INCA software system.

Mechanical Properties

Measurements of thickness of the untreated and treated fabrics were performed using the standard method according to (ASTM-D1777). The tensile strength & elongation of untreated as well as treated samples were measured according to ASTM-D5035-11 using universal testing machine

UV-Protection

The transmission of ultraviolet (UV) radiation through fabrics was evaluated using Varian Cary 300 ultraviolet visible spectrophotometer (Mulgrave, Victoria 3170, Australia) at a wavelength range of 320- 400 nm.

Infrared Spectra (FTIR)

Infrared spectra were recorded on FTIR Nicolet 5 DX Spectrophotometer. The samples was examined as 1.5% KBr pellets.

Thermogravimetric analysis (TGA)

From each sample of 4-5 mg were cut and used for thermo-gravimetric analysis experiments. ANETZSCHTGA 209 thermo-balance, airflow of 10.00 cm^3/min and aluminum oxide crucibles were used for thermo-gravimetric measurements. The temperature range was 20 to 700ºC, and the heating rates were 10ºC.

Antimicrobial Activity

Antibacterial activity was assessed quantitatively against Staphylococcus aureus (G +ve) according to AATCC 6538, and ***Escherichia coli*** (G-ve) according to AATCC 25922, and pathogenic fungus (***Candida albican*** and ***Aspergillus flavus***) were assessed quantitatively according to AATCC TM 30. All results were expressed after doing comparison with the control sample and treated samples according the following equation:

$$\textbf{Reductin}\ (\%) = \frac{\mathbf{B-A}}{\mathbf{B}}\ \mathbf{X\ 100}\quad \textbf{(2)}$$

Where B is the number of microorganisms presents on the control sample, and

A is the number of microorganisms present on the treated samples.

Results and Discussion

Durability test

The treated polyester samples were washed using Launder Ometer according to AATCC 36-93. The add-on (%) was calculated according to equation 3 and the obtained results were summarized in Table 2. Data of Table 2 implies that the add-on (%) of the treated sample are comparable. It was found that after 5 washing cycles the add-on (%) decreased significantly compared to that after one washing cycle only. On the other hand, after 10 washing cycles the decrease of add-on (%) are negligible compared to that after 5 cycles. These results indicated that this treatment is durable to wash.

$$\boldsymbol{Addon}\ (\%) = \frac{\boldsymbol{W_2-W_1}}{\boldsymbol{W_1}}\ \boldsymbol{x\ 100}\quad \textbf{(3)}$$

Where: W_2 is the weight of sample after treatment of alkali treated polyester fabric with SiO_2. W_1: is the weight of sample before treatment.

Table 2: Add on % of untreated and alkali treated polyester fibres with SiO_2NPs

Description	Add on %		
	One washing cycle	5 wahing cycles	10 wahing cycles
Alkali treated PET with 0.5% SiO_2NPs	1.51	0.92	0.87
Alkali treated PET with 1% SiO_2NPs	2.45	1.45	1.02
Alkali treated PET with 1.5% SiO_2NPs	3.52	2.33	1.93

Transmission Electron Microscopy

Figure 1 (a, b) show the Transmission Electron Micro Graphs (TEM) of SiO2NPs powder. These figures

clarify that the size of the obtained SiO2NPs powder is within the nano range (20–22 nm).

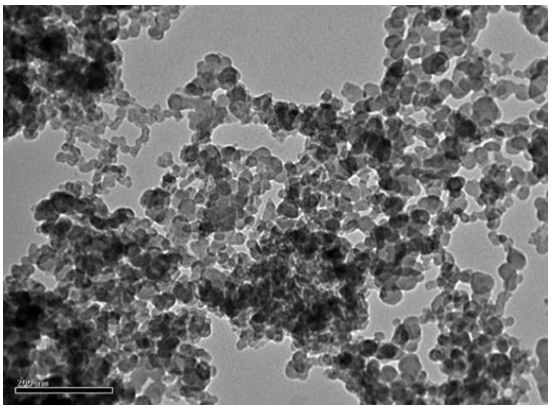


Figure 1 (a)

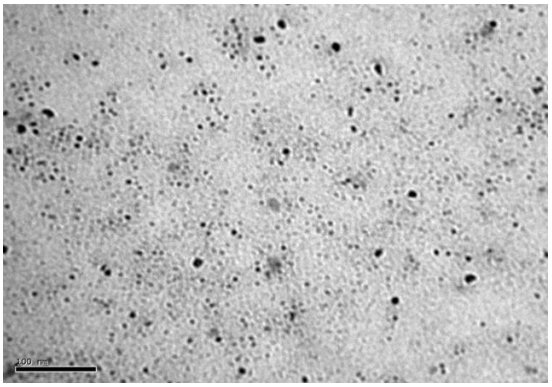


Figure 1 (b)

Figure 1 (a, b): TEM of the SiO_2NPs

Field-emission scanning electron microscopy (FE-SEM) and dispersive X-ray spectroscopy (EDX) of treated polyester fibres with SiO_2NPs

Morphological structure of untreated as well as treated polyester fabrics were investigated using FE-scanning electron microscopy. Figure 2 (a) shows the surface of the untreated which implies the clean and smooth longitudinal fibril surface of the untreated sample. Figure 2 (b & c) show the topographical character of the treated samples with 1% and 1.5% SiO_2NPs respectively. It is observed that a sufficient amount of SiO_2NPs is deposited on the fabric surface. Surface chemical elements of the treated polyester fibres determined by EDX spectroscopy. Figure 3 (a) shows the EDX spectra for untreated sample and Figure 3 (b, c) shows the EDX spectra for treated samples and. It is clear that the spectrum of untreated samples show the original peaks of polyester fabric while Fig. 3 (b) & (c) indicating the presence of silicon element by 1.89%, and 2.82% for fabrics treated with 1% and 1.5% SiO_2NPs respectively.

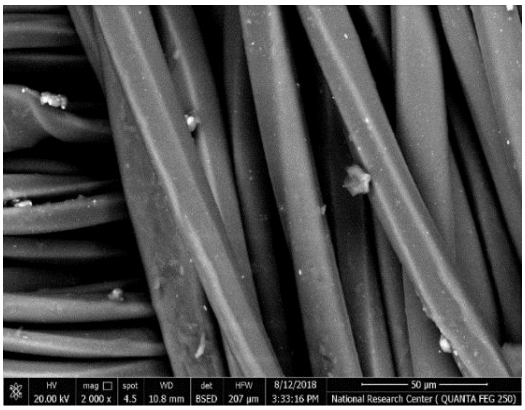


Figure 2 (a)

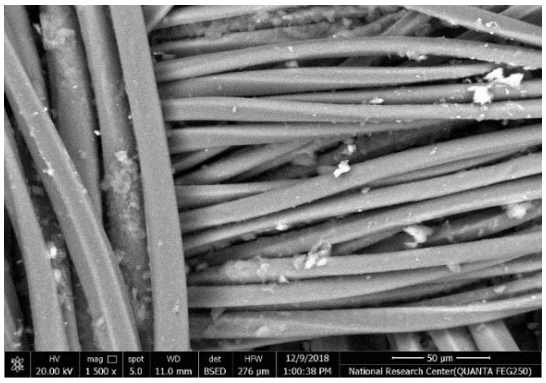


Figure 2 (b)

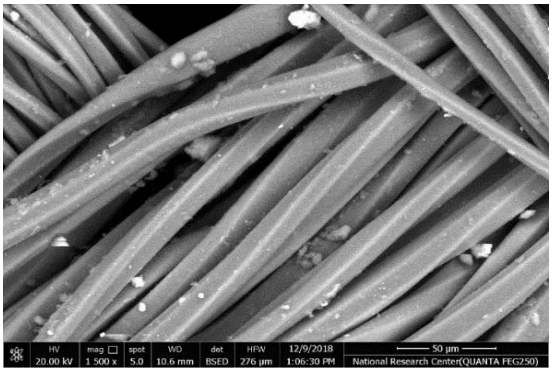


Figure 2 (c)

Figure 2: (FE-SEM) Scanning electron microscopy of untreated and treated polyester fibres. (a) untreated polyester fabrics, (b) Polyester fibres treated with 1% SiO2NPs, (c) polyester fibres treated with 1.5% SiO2NPs

These results prove that the presence of the SiO_2NPs in the treated polyester fibres.

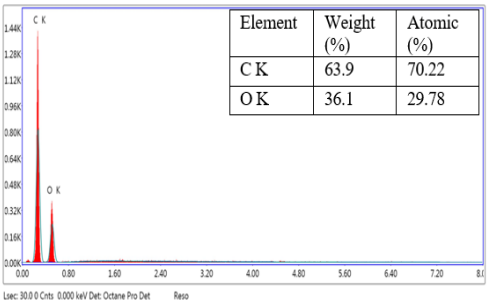


Figure 3 (a)

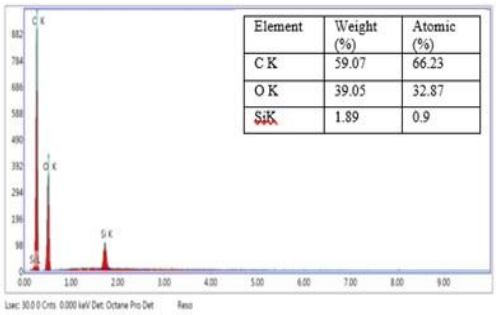


Figure 3 (b)

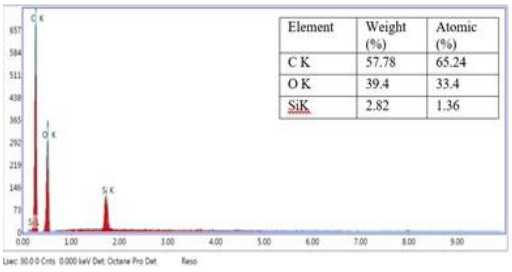


Figure 3 (c)

Figure 3: EDX of treated as well as untreated polyester fibres, (a) EDX of untreated polyester fabrics (b) EDX of sample treated with 1% SiO_2NPs and (c) EDX of sample treated with 1.5% SiO_2NPs

Ultraviolet Protection factor (UPF)

Ultraviolet protection factor (UPF) measures the effectiveness of textile fabrics in protecting the human skin from UV radiation (UVA and UVB radiations). An overdose of UV radiation leads to acute and chronic reactions, skin reddening (erythema) or sunburn, increasing the risk factor of persons susceptible to melanoma and skin cancer [10].

Data in Table 3 illustrate the ultraviolet protection factors of the treated polyester fibres as well as untreated polyester samples. It is found that the treated samples have excellent resistance to ultraviolet radiation compared to the untreated one. Data in Table 3 clarify that the untreated and alkali treated samples have no protection for UV radiations; on contrary the samples treated with 1% SiO_2NPs and 1.5% SiO_2NPs have a very good (30) and excellent (55) protection for UV radiations respectively.

These results may be due to the pores of the polyester fabrics were filled with SiO_2NPs and subsequently minimize the porosity of the fibre vicinity, which acts as a barrier, and thus prevents the UV radiation absorption.

Table 3: Percentage UPF of treated as well as untreated polyester fibres with SiO_2NPs

Sample description	Increase in UPF (%)	Grade
Untreated polyester fibres	8	No protection
Treated with NaOH	10	Poor protection
0.5% SiO_2 NPs	14	Good protection
1% SiO_2 NPs	30	Very Good protection
1.5% SiO_2 NPs	55	excellent protection

Tensile strength & Elongation % and Thickness of untreated and treated polyester fibres with SiO_2NPs

The results of the tensile strength, elongation % and thickness of the untreated, pretreated and treated polyester fibre were tabulated in table 4. The tensile strength increased with treatment with SiO_2NPs than the untreated polyester fibres; it was found that the increase in tensile strength depended on the concentration of SiO_2NPs, this is due to increasing SiO_2 NPs leads to increasing the amount of nano particles in the interfacial space between the polyester filaments. Moreover, it can be seen that the data of elongation % and thickness of the treated polyester fibres with SiO_2NPs were not significant changed compared to the untreated sample.

Table 4: Tensile strength & Elongation % and Thickness of treated and untreated polyester

Description	Tensile Strength (kg / cm^2)	Elongation (%)	Thickness (mm)
Untreated polyester fibres	0.78	26	0.24
Treated with 8% NaOH	0.66	35	0.25
0.5% SiO_2NPs	0.74	31	0.24
1% SiO_2NPs	0.88	29	0.245
1.5% SiO_2NPs	0.92	25	0.245

Fourier transformed infrared spectroscopy (FTIR)

FTIR spectra obtained for untreated and treated PET/8%NaOH polyester fibres are depicted in Fig. 4 (a, b). The FTIR spectra of untreated PET showed broad peak around 3622 cm^{-1} due to absorbed water and around 3427 cm^{-1} due to stretching vibration of carboxylic OH groups respectively. While the peaks around 2961, 2917 and 2852 cm^{-1} are corresponding to CH_2 stretching vibrations, the strong peaks at 1711 cm^{-1} and 1468 cm^{-1} are corresponding to asymmetrical and symmetrical vibrations of the carboxylic ester C=O groups respectively. The peaks at 1578, 1504 and 720 cm^{-1} are related to the C=C of the benzene ring, while the peak at 1339 cm^{-1} is due to bending and wagging vibrational modes of the ethylene glycol segment [14]. The characteristic peaks at 1241, 1094 and 1017 cm^{-1} are due to C-O, C-O-C and C-OH bonds respectively, while the peaks appeared at 871 and 847 cm^{-1} are related to the p-substituted benzene ring. Spectrum of treated PET/ 8%NaOH showed in addition to the mentioned peaks new peaks at 3539 and 1455 cm^{-1} related to the hydrogen bonded OH and $-COO^-$ groups resulted from the alkaline hydrolysis, respectively. On the other hand, the FTIR spectra of PET/1% SiO_2 NPs and PET/ 1.5% SiO_2 NPs (Fig. 4 c, d) showed additional peaks around 3692 cm^{-1} related to Si-OH group, around 470 cm^{-1} attributed to SiO_2 NPs and also showed the three main typical silica bands detectable in the regions near 433, 845 and 970 cm^{-1} related to Si-O-Si, Si-O-Si and Si-O-C bonds [15, 16]. The presence of those peaks (at 433 cm^{-1}, 845 $cm^{-1}$and 970 cm^{-1}) strongly suggests that chemical bonding between PET and SiO_2 Nano-particles was formed during modification process [17].

Thermogravimetric analysis of the untreated and treated polyester fibres with SiO_2NPs

Thermogravimetric (TGA) curves of the untreated and treated PET fibers are presented in Figure 5. Thermal properties of the PET fibers should be investigated to evaluate the extent of thermal stability during their processing at elevated temperature above 200 ° C and to illustrate the nanoparticles–fiber interaction, since dislocations in thermal behavior are expected if novel strong bonding are present [14]. From the TGA, It was found that all PET samples have two degradation steps, the first one is due to dehydration of the polymer as mass loss% varies from 1-2%, (at temperature ranges between 50 and 200℃) , The second step is the main degradation step. The second step is the main degradation step. For untreated PET fibers second degradation state started at 387℃, maximum degradation took place at T max 428℃, and the residual mass at the end of degradation process was 22%.The thermal analysis of the treated PET/nono-SiO_2 composite showed improvement in the thermal stability than untreated PET sample indicated from the higher T onset (401℃), this might due to those SiO_2NPs adsorbed on the PET surface which stabilized it from fast degradation. The residual mass was 19% at the end of degradation step. This observed lower in the residual mass related to the degradation of both PET and SiO_2 particles which confirm the physical interaction between them; maximum degradation took place at T_{max} 440℃. On the other hand, TGA curves of treated PET/NaOH sample showed higher T onset (410 ° C) than the untreated PET sample, this is due to the alkaline hydrolysis with NaOH of the former sample with NaOH,, since alkaline hydrolysis resulted in new free OH and COOH groups which allow the formation of great number of hydrogen bonds, that needs higher energy for degradation, the residual mass was 20% lower than that for untreated sample, might due to degradation of the small fragments resulted from the alkaline hydrolysis. TGA curves of treated PET/Nano SiO_2 composite showed much higher thermal stability than the others samples. This higher thermal stability might due to the formation of strong chemical bonds between The SiO_2NPs and the PET functional groups (OH, COOH), which acts as a barrier for thermal degradation, as observed from the higher residual mass 34%, maximum degradation occurred at T max 429℃. All data was summarized in Table 5.

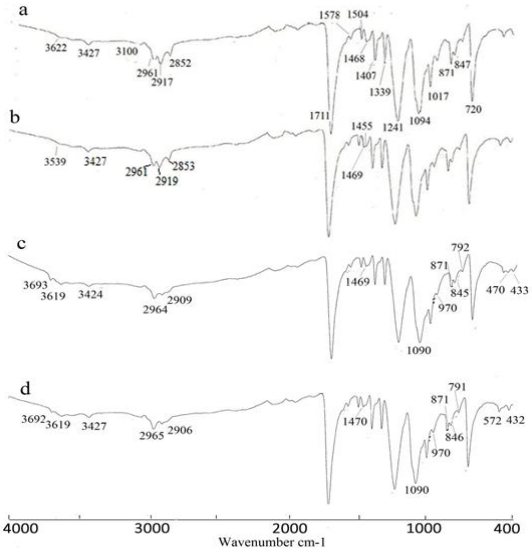


Figure 4: FTIR spectra of (a) untreated PET, (b) treated PET/8%NaOH, (c) PET/ 1% SiO_2NPs and (d) PFT/ 1.5% SiO_2NPs

Table 5: Thermal properties (TGA) of the treated and untreated polyester fabrics

Samples description	T onset (℃)	T max (℃)	T end (℃)	Residual weight (%)
Untreated PET fabric	387	428	450	22
PET/8%NaOH	410	444	470	20
Alkali treated polyester treated with 1% SiO_2NPs	401	440	475	19
Alkali treated polyester treated with NaOH/1.5% SiO_2NPs	385	429	450	34

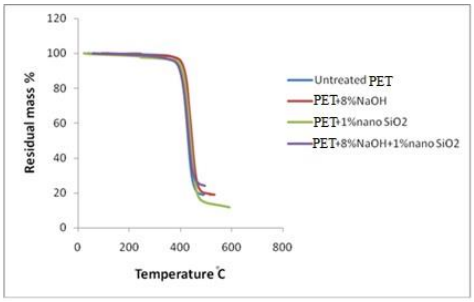


Figure 5: TGA diagram of PET, PET/8% NaOH, PET/1% SiO_2NPs and PET/8%NaOH/ 1% SiO_2NPs

Antimicrobial activity

Table 6: The reduction (%) of antimicrobial species of untreated and treated polyester fabrics

Sample description	Antimicrobial reduction (%)			
	Staphylococcus aureus (G +ve)	Escherichia coli (G -ve)	Candida albicans (fungus)	Aspergillus flavus (fungus)
Untreated PET	0	0	0	0
PET/8%NaOH	45	40	39	38
Alkali treated polyester treated with 1% SiO_2NPs	90	93	90	89
Alkali treated polyester treated with 1.5% SiO_2NPs	95	97	94	91

In this test, the reduction (%) of antimicrobial species of the alkali treated and treated polyester fabrics will be compared. Table 6 points to the reduction in growth of antimicrobial species. It was found that all treated samples either pretreated with NaOH or treated with SiO_2NPs showed higher reduction in *Staphylococcus aureus, Escherichia coli, Candida albicans* and *Aspergillus flavus* compared to the untreated one.

Also, it is obvious that alkali treated sample treated with SiO_2NPs have higher reduction % than that pretreated with NaOH only. This may be due to the strong interaction of their cationic surfaces of nanoparticles with the microorganism cells. Table 6 shows also that the antimicrobial reduction increase as the concentration of SiO_2NPs increase [7, 18].

Radar chart

Fig. 6 shows the radar chart area of the untreated and treated polyester fabrics modified with SiO_2NPs. It was observed that the samples treated with 1.5% SiO_2NPs achieves the best performance followed by the sample treated with 1% SiO_2NPs. From these results it can be concluded that the treated samples are the most recommended fabrics to be used as medical textiles. The QF for both treated with 1% SiO_2NPs and with 1.5% SiO_2NPs are 87% and 89% respectively, compared to the QF 35% for untreated fabrics as shown in table 7.

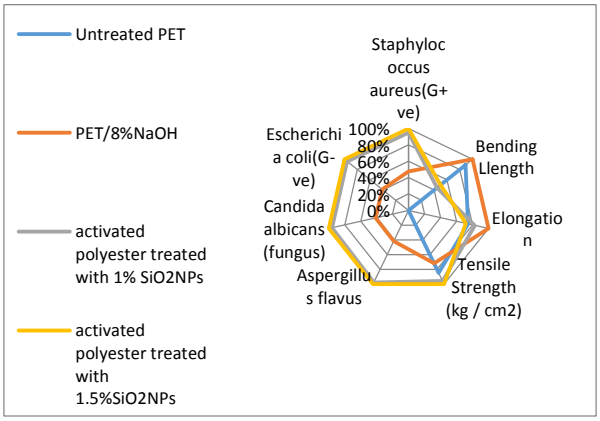


Figure 6: Radar chart of untreated and treated polyester fabrics with SiO_2NPs

Conclusions

The polyester fibers were treated with different concentrations of SiO_2NPs using IR dyeing machine. FE-SEM mentioned the presence of SiO_2NPs on the surface of polyester and this result can be confirmed with EDX and FTIR. EDX was showed the presence of SiO_2NPs between fiber filaments with different ratios depends on the concentration of SiO_2NPs used in the treatment (1 and1.5%).

Table 7: Qualified factor of untreated and treated polyester fabrics with SiO_2NPs

Sample	Quality Factor	Rank
Untreated PET fabric	35%	4
PET/8%NaOH	63%	3
Alkali treated polyester fabric treated with 1% SiO_2NPs	87%	2
Alkali treated polyester treated with 1.5%SiO_2NPs	89%	1

FTIR spectra of treated samples appeared the characteristic peak of Si-O and Si-O-Si compared to the untreated one, which indicates the presence of SiO_2NPs in the polyester samples. Also, the results of this work clarify that the treatment of the alkali treated polyester fabric with SiO_2NPs improved the thermal analysis (TGA) comparing to the untreated one. The ultraviolet protection of the treated fabric was enhanced after the treatment with SiO_2NPs compared to the untreated one.

The antimicrobial activity illustrated that all the treated samples either pretreated with NaOH or treated with SiO_2NPs showed a higher reduction in *Staphylococcus aureus*, *Escherichia coli*, *Candida albicans* and *Aspergillus flavus* compared to the untreated one, and the reduction depends on the concentration SiO_2NPs. This result may be due to the strong interaction of their cationic surfaces of nanoparticles with the microorganism cells. The radar chart area results indicate the possibility of using the treated fabrics as medical textiles.

Conflict of interest

There are no conflicts to declare.

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