An eco-friendly facile approach for imparting multifunctional protection properties to cellulose/wool blends

Nabil A. Ibrahim¹ · Basma M. Eid¹ · El-Amir M. Emam²

Received: 21 August 2021 / Revised: 13 December 2021 / Accepted: 27 December 2021 /

Published online: 13 January 2022

© The Author(s) 2022

Abstract

A green facile nano-finishing route was developed to impart high antibacterial efficacy, UV-protection, self-cleaning and anti-wrinkle functions to cotton/wool and viscose/wool blends using TiO₂, and/or Ag-NPs, as active ingredients, along with citric acid and/or succinic acid/ SHP as ester-crosslinking/fixing systems. The data so obtained demonstrated that outstanding durable functional properties can be achieved using the following formulation: TiO₂/Ag-NPs (20 g/L each), citric acid/ SHP (50 g/L/30 g/L) and the pad- dry microwave fixation at 1300 W for 5 min. SEM and EDX analysis for selected samples as well mode of interactions among the nominated finishing ingredients and the treated substrates were also investigated.

Keywords Multifunctional cellulose · Wool blend · TiO₂NPs · AgNPs · Antibacterial · UV-protective properties

Introduction

Factors affecting multifunctionalization of textile blends include type of blend component, fabric structure, functional finishing formulation constituents, application method, the desired functional properties, environmental concerns and economic aspects [1–4].

Recently, the ever-growing textile consumer demands and expectations for green high value-added, durable, comfortable, and multifunctional protective textile products [5, 6], along with the increasing environmental concerns have received a great

✉ Basma M. Eid
basmaeid@yahoo.com

¹ Textile Research and Technology Institute, National Research Centre, 33 El Bohouth st., Dokki, Giza, Egypt

² Faculty of Applied Arts, Printing, Dyeing and Finishing Department, Helwan University, Cairo, Egypt

attention and created numerous opportunities for replacement of tradition-hazardous finishing chemicals and non-sustainable finishing methods with environmentally benign chemicals and sustainable emerging technologies, taking into consideration product quality, economy, ecology and social aspects [7–11].

Potential applications of nanotechnology and inorganic nanomaterials like metal and/or metal oxides have been efficiently used along with polycarboxylic acids, as an eco-friendly ester-crosslinking and immobilizing agents, for developing green multifunctional high-added value textile products [4, 8, 12–18]. To the best of our knowledge, few studies have been published on multifunctionalization of cellulose/wool blends using noble metals and/or metal oxides, as multifunctional agents, polycarboxylic acids and sodium hypophosphite, as finishing system, and microwave for fixation step [1].

This study is focused on developing a new, facile and environmentally sound approach to surface modification and multifunctionalization of cotton/wool and viscose/wool substrates using TiO_2 -NPs and/or Ag-NPs as multifunctional agents, citric acid and/or succinic acid as ester-crosslinking/immobilizing agents, sodium hypophosphite as the catalyst, and microwave fixation instead of using the traditional thermofixation technique. The imparted functional properties, namely antibacterial, UV-shielding capability and anti-wrinkle property were evaluated, and the possible reaction mechanisms were proposed and discussed as well.

Experimental

Materials

Mill-scoured and bleached cellulose/wool blended fabrics, namely cotton/wool (C/W, 50/50, 230 g/m², thickness 0.76 mm) and viscose/wool (V/W, 50/50, 190 g/m², thickness 0.65 mm) were used in this study.

The titanium dioxide nanoparticles TiO_2 NPs (powder) was an anatase and supplied by Sigma Aldrich. Polycarboxylic acids, namely citric acids (CA) and succinic acid (SA), sodium hypophosphite monohydrate (SHP, $\text{Na H}_2\text{PO}_2 \cdot \text{H}_2\text{O}$) and other chemicals were of reagent grade.

Methods

Preparation of TiO_2 -NPs aqueous dispersion

Aqueous dispersion of TiO_2 NPs was prepared by the mixing of 7.5 g of TiO_2 NPs powder (3 wt%) and appropriate amounts of deionized water in an ultrasonic mixer for 5 h. Then 0.625 g of polyglycol (0.25 wt%, average molecular weight 400 g/mole) was gradually added to the aqueous dispersion for 15 min and then left for 30 min to complete the preparation process [19]. The size of TiO_2 NPs

was determined by Transmission Electron Microscopy (TEM) analysis using JEOL model 1200EX election microscope operated at an accelerating voltage at 120 kV. The image of TiO_2NPs showed agglomerates of the nanoparticles and size was ranged from 8 to 15 nm (Fig. 1a).

Synthesis of Ag-NPs

AgNPs was prepared by using AgNO_3 as a precursor, soluble starch as both reducing and stabilizing agent and water as an appropriate solvent. In this context, 1 g of soluble starch was added to 100 ml of deionized water, heated in microwave oven for complete dissolution, and then 1 ml of 100 mM aq. solution of AgNO_3 was added and mixed well. The obtained mixture was kept in an autoclave, at 15 psi/120 °C for 10 min, to obtain a clear yellow solution of AgNPs [20]. The synthesized AgNPs was spherical in shape observed by TEM. The size of AgNPs was ranged from 3

Fig. 1 TEM images of TiO_2NPs and **b** AgNPs, TEM images **b** and UV–vis spectrum **c** of synthesized AgNPs

to 8 nm and the UV–vis spectrum of the synthesized AgNPs showed an absorption peak at about 435 nm (Fig. 1b, c).

Nano-finishing

Aqueous functional finishing formulations were prepared by mixing CA and/or SA, SHP-catalyst, with the appropriate amount of TiO₂-NPs and/or Ag-NPs, and distilled water in ultrasonic bath at 30 °C for 30 min. Subsequently, the cellulose/wool blended fabrics, i.e. C/W and V/W, were padded twice to reach on average wet pick-up 85% by freshly prepared aqueous finishing formulations. Finally, the incorporation of TiO₂-NPs and/or Ag-NPs in finished fabrics was carried out by the pad-dry/cure microwave fixation process at 1300 W for 5 min.

After that, the finished fabric samples were washed and rinsed thoroughly to remove excess and unfixed ingredients and finally dried. Finishing formulation and conditions used were given in the text.

Testing methods

- Ti and Ag contents were quantitatively determined using a flame atomic absorption spectrophotometer, GBC-Avanta, Australia.
- Dry wrinkle recovery angles of the treated fabrics (DWRA), were determined according to AATCC standard method 66–1995.
- The antibacterial activity against Gram-positive (G+ ve, *S. aureus*) and Gram-negative (G– ve, *E. coli*) pathogenic bacteria, was qualitatively determined according to AATCC test method (147–1988) and expressed as a zone of growth inhibition (ZI, mm).
- UV-protection factor, UPF, was determined according to the Australian/ New Zealand standard (AS/NZS 4366–1996). UPF values of 15–24, 25–39, 40–50⁺ are referred to good, very good and excellent protection, respectively.
- Self-cleaning (SC) property of the untreated and some nanofinished fabric samples were determined by staining with methylene blue (MB-dye) and drying at room temperature. The stained fabric samples were irradiated through exposure to an UV-lamp at wavelength of 400 nm for 12 h and calculated using the following equation [21] according to the following equation:

$$SC\% = (K/S)_b - (K/S)_a / (K/S)_b \times 100$$

where $(K/s)_a$ and $(K/s)_b$: color strength after and before exposure at $\lambda_{\max} = 670$, respectively.

- Durability to wash was determined according to AATCC methods 124.
- The surface morphology of nanofinished fabric samples, SEM was studied using a JEOL, JXA-840A electron probe microanalyzer equipped with disperse X-ray spectrophotometer (EDX) for composition analysis.
- All determinations were done in triplicate and the average was taken as a final result.

Results and discussion

Effect of TiO₂-NPs concentration

Table 1 reports the effect of inclusion of TiO₂-NPs (0–40 g/L) into the ester-crosslinking formulation along with CA (50 g/L), as ester-crosslinking and binding agent and SHP (30 g/L), as an appropriate ester-crosslinking catalyst, on the extent of modification and functionalization of viscose/wool (V/W) and cotton/wool (C/W) substrates. It is clear, regardless of the substrate used and for a given finishing condition, that increasing TiO₂-NPs from zero to 40 g/L brings about an increase in TiO₂-content, an improvement in dry wrinkle recovery angles, DWRA, a noticeable enhancement in anti-bacteria activity against Gram-positive (*S. aureus*) and Gram-negative (*E. coli*) pathogenic bacteria, ZI, and a remarkable improvement in UV-shielding ability, UPF, against the harmful UV-radiation.

The increase in TiO₂-content is a direct consequence of the positive role of ester-crosslinking on enhancing the extent of fixation and immobilization of TiO₂-NPs onto/within the fabric structure via free binding and anchoring sites like free –COOH groups, –NH₂, –OH, etc., onto and/or in the cellulose/wool structure [1, 21–23]. On the other hand, the extent of fixation of TiO₂-NPs is governed by the type of substrate, amorphous/crystalline ratio, number, location and availability of active/binding sites as well as the degree of ester-crosslinking and follows the decreasing order V/W > C/W, keeping other parameter constant [1, 4].

Improvement in fabric resiliency, DWRA, by increasing TiO₂-NPs concentration reflects the positive role of nano-TiO₂, as a co-catalyst, in accelerating the formation of reactive intermediates, i.e. cyclic anhydrides, which, in turn, positively affects the extent of esterification of the nominated substrates [1, 22]. On the other hand, increasing TiO₂-NPs in amorphous regions of the fabric structure would further enhance the imparted wrinkle recovery by linking the active

Table 1 Effect of TiO₂NPs concentration on extent of multifunctionalization of cellulose/wool blended fabrics

TiO2 NPs (g/L)	TiO2-content (%)		DWRA (W + F) ^o		ZI (mm)				UPF	
					V/W		C/W			
	V/W	C/W	V/W	C/W	G + ve	G – ve	G + ve	G – ve	V/W	C/W
0	0.000	0.000	230	210	4.0	3.0	3.0	2.0	15	24
20	3.083	2.716	246	228	15.5	10.5	12.0	10.0	32	55
40	3.320	3.104	268	255	20.0	18.5	17.0	16.0	65	98
Untreated	0.0	0.0	200	185	0.0	0.0	0.0	0.0	10	18

Finishing formulation: CA (50 g/L), SHP (30 g/L), TiO₂ NPs (0–40 g/L), nonionic wetting agent (2 g/L), wet pick-up (85%), drying/microwave fixation at 1300 W for 5 min

V/W Viscose/wool blend, C/W Cotton/wool blend, DWRA (W + F) : dry wrinkle recovery angle (warp + weft), ZI Inhibition zone, G + ve Gram-positive bacteria (*S. aureus*); G – ve Gram-negative bacteria (*E. coli*); UPF UV- protection factor

sites of the fabric components, i.e., cellulose and protein chains and restricting the molecular movement, i.e., better anti-wrinkle [24, 25].

Additionally, even in the absence of TiO_2 -NPs, the ester-crosslinked substrate possesses a certain antibacterial activity against the tested pathogenic bacteria reflecting the antibacterial action of CA and its ability to interact with the bacterial components, which in turn, negatively impact its functions and finally lead to their destruction [26, 27]. The data in Table 1 demonstrate that increasing TiO_2 -NPs in the ester-crosslinking formulation brings about a remarkable increase in the imparted antibacterial activity, regardless of the finished substrate.

This outstanding improvement in antibacterial efficiency could be discussed in terms of the unique photocatalytic activity of the immobilized TiO_2 -NPs and generation of reactive oxygen species like $\cdot\text{OH}$, $\cdot\text{O}_2$, H_2O_2 , etc. along with the accumulation of TiO_2 -NPs in the cell wall and their subsequent negative impacts on cell membrane, cell viability and cell growth thereby causing bacterial death [1, 4, 5, 22, 28, 29]. The imparted antibacterial activity against the tested microorganisms follows the decreasing order: Gram-positive (*S. aureus*) > Gram-negative (*E. coli*), most probably due to their structural and sensitivity difference [30–32].

It is also worth noting, Table 1, that increasing the loaded TiO_2 -NPs concentration up to 40 g/L results in a remarkable improvement in UV-protecting effect irrespective of the treated substrates. The higher the TiO_2 -NPs concentration and content, the better the UV-shielding capability, expressed as UPF value. The noticeable increase in UPF value of nanofinished substrates is a direct consequence of upgrading UV-blocking capability of the immobilized TiO_2 -NPs onto the fabric surface via scattering the harmful UV-radiation along with blocking the open pores of the fabric structure via nanocrosslinking during the microwave fixation step, thereby hindering its transmission intensity [28, 33, 34]. The imparted anti-UV functionality is governed by the type of substrate, e.g. fabric structure, weight, thickness, open pores [5, 35, 36], and extent of crosslinking as well as loading of TiO_2 -NPs onto/within the fabric structure. The results in Table 1 signify that the outstanding improvement in UV-shielding capability follows the decreasing order: C/W > V/W, keeping other parameters constant.

Mechanism of modification and functionalization cellulose/wool structure

The tentative mechanism of possible modification and functionalization reactions among cellulose/wool (Cell.OH/W-XH) blended fabrics, CA, as an eco-friendly trifunctional ester-crosslinking agent, SHP, as ester-crosslinking catalyst, along with TiO_2 -NPs, as a photocatalyst, is shown in Scheme 1.

Consequently, a remarkable improvement in desirable functional properties such as wrinkle recovery, antibacterial efficacy and in UV-shielding capability

i- Formation of cyclic anhydride intermediates and ester-crosslinking [1,37]

ii- Ionic crosslinking [1,38]

iii- Immobilization of TiO₂-NPs [1,39,24]

iv- Generation of reactive oxygen species ROS, e.g. $\cdot\text{OH}$, $\cdot\text{O}_2^-$, H_2O_2 , Single oxygen, etc. [1,25,24]

where h^+ and e^- are a positive hole and an electron, respectively

Scheme 1 The possible ester/ionic-crosslinking and immobilization of TiO₂NPs onto crosslinked cellulose/wool fabric, and the subsequent generation of ROS

of the nominated substrates, C/W and V/W, can be achieved using the developed green-finishing formulation constituents and nanofinishing conditions.

Type of polycarboxylic acid

As far as the charge in TiO₂-content and in the imparted wrinkle recovery property, DWRA, antibacterial activity, ZI, and UV-blocking capability to the treated blends, the data in Table 2 signify that the increase in TiO₂-content, the improve in DWRA, the enhancement in ZI as well as the ascending in UPF values follow the decreasing order: CA>CA/SA>SA, keeping other parameters constant. This can be discussed in terms of the variation in acid reactivity, number of available functional groups, i.e. –COOH groups, which in turn, greatly affect the extent of ester-crosslinking and immobilization and fixation of TiO₂-NPs onto/into the blend structure [1, 40, 41], considering that SA, as a bifunctional polycarboxylic acid, acts as an esterifying not as a crosslinking agent compared with the used trifunctional counterpart, i.e. CA, as follows:

Types of inorganic nanoparticle

The development of high-value added multifunctional cellulose/wool blended fabrics using an eco-friendly nanofinishing formulation is the main task of this research work. The effect of using inorganic nanoparticles, namely TiO₂-NPs (40 g/L), Ag-NPs (40 g/L) individually and in combination TiO₂-NPs/Ag-NPs (20/20 g/L) on

Table 2 Effect of type and concentration of carboxylic acids on extent of multifunctionalization of cellulose/wool blended fabrics

Carboxylic acids (g/L)		TiO ₂ -content (%)		DWRA (W + F) ^o		ZI (mm)				UPF	
CA	SA	V/W	C/W	V/W	C/W	V/W		C/W		V/W	C/W
						G + ve	G – ve	G + ve	G – ve		
50	0	3.320	3.104	268	255	20.0	18.5	17.0	16.0	65	98
25	25	2.716	2.529	250	240	15.5	14.0	13.0	10.5	54	79
0	50	2.495	2.289	240	222	13.0	12.0	10.5	9.0	40	56
Untreated	–	–	200	185	0.0	0.0	0.0	0.0	10	18	

Finishing formulation: finishing agent (50 g/L), SHP (30 g/L), TiO₂ NPs (40 g/L), nonionic wetting agent (2 g/L), wet pick-up (85%), drying/microwave fixation at 1300 W for 5 min

CA Citric acid, SA succinic acid, V/W Viscose/Wool blend, C/W Cotton/wool blend, DWRA (W + F)^o: Dry wrinkle recovery angle (warp + weft), ZI Inhibition zone, G + ve Gram-positive bacteria (*S. aureus*); G – ve Gram-negative bacteria (*E. coli*); UPF UV- protection factor

Table 3 Effect of type and concentration of nanomaterial on extent of multifunctionalization of cellulose/wool blended fabrics

Nanomaterial (g/L)		Nanomaterial -content (%)		DWRA (W + F) ^o		ZI (mm)		C/W		UPF	
TiO ₂ NPs	AgNPs	V/W	C/W	V/W	C/W	G + ve	G –ve	G + ve	G –ve	V/W	C/W
40	0	3.320	3.104	268	255	20.0	18.5	17.0	16.0	65	98
20	20	3.145/0.041	2.922/0.033	280	265	22.5	20.5	21.0	19.5	90	120
0	40	0.068	0.051	241	225	21.0	19.0	18.5	17.0	55	85
None	None	0.00	0.00	230	210	4.0	3.0	3.0	2.0	15	24

Finishing formulation: CA (50 g/L), SHP (30 g/L), Nanomaterial (40 g/L), nonionic wetting agent (2 g/L), wet pick-up (85%), drying/ microwave fixation at 1300 W for 5 min

V/W Viscose/wool blend, C/W Cotton/wool blend, DWRA (W + F)^o: dry wrinkle recovery angle (warp+weft), ZI Inhibition zone, G + ve Gram-positive bacteria (*S. aureus*); G–ve Gram-negative bacteria (*E. coli*); UPF UV-protection factor

upgrading and imparting new functional properties to the used substrates is shown in Table 3. As evident from Table 3, the nanofinished fabric samples displayed a remarkable improvement in DWRA, ZI, UPF values of the multifunctionalized substrates. The extent of improvement in the above-mentioned properties is governed by the type of inorganic nanomaterial and follows the decreasing orders:

Regarding to:

DWRA: $\text{TiO}_2\text{-NPs} > \text{TiO}_2\text{-NPs/Ag-NPs} > \text{Ag-NPs} > \text{None}$,

ZI: $\text{TiO}_2\text{-NPs/Ag-NPs} > \text{Ag-NPs} > \text{TiO}_2\text{-NPs} \geq \text{None}$ (Fig. 2), and.

UPF: $\text{TiO}_2\text{-NPs/Ag-NPs} > \text{TiO}_2\text{-NPs} > \text{Ag-NPs} \geq \text{None}$, keeping other parameters constant.

On the other hand, the variation in nanomaterial content is determined by the extent of fixation and immobilization of used inorganic nanomaterials via chelation and electrostatic interactions, e.g. $-\text{COO}^- \dots \text{Ag}^+$ and/or $-\text{COO}^- \dots \text{TiO}^{4+}$ [1, 39, 41].

The remarkable improvement in antibacterial efficacy of Ag-NPs-loaded substrates could be discussed in terms of their ability to: bind to the bacterial outer membrane, interact with the thiol groups of bacterial proteins and to generate reactive oxygen species in the presence of dissolved oxygen as follows [33, 42]:

Thereby leading to subsequent inactivation and oxidation of the bacterial molecular structure [26, 42].

Moreover, the incorporation of Ag-NPs along with $\text{TiO}_2\text{-NPs}$ in the finishing formulation plays a synergistic effect in imparting unique antibacterial and UV-blocking functional properties, regardless of used substrate. The high antibacterial activity of $\text{TiO}_2\text{NPs/AgNPs}$ substrates could be discussed in terms of the synergistic

Fig. 2 Inhibition zone of viscose/wool blended fabric samples treated with $\text{TiO}_2\text{-NPs}$ 1, $\text{TiO}_2\text{-NPs/Ag-NPs}$ 2, Ag-NPs 3 against *S. aureus* and *E. coli*

activity of the generated reactive oxygen species, ROS, along with the loaded Ag^+ from Ag-NPs against the tested pathogenic bacteria [33, 42–44].

Additionally, the enhanced photocatalytic activity of TiO_2 -NPs/Ag-NPs reflects the positive role of Ag-NPs is acting as electron trappers, thereby minimizing and/or inhibiting the rate recombination of the generated electron–hole pairs [Eq. 5] by light on TiO_2 -loaded substrates [44–46], which, in turn, positively affects the UV-protection capability.

Durability of the imparted functional properties

From the previous experimental results, Table 4 that incorporation AgNPs/ TiO_2 NPs (20/20 g/L) into the finishing formulation along with CA (50 g/L) and SHP (30 g/L) brings about remarkable improvement in the imparted functional properties regardless of the used substrate. So, these optimum nanofinishing conditions are used to evaluate the durability of the imparted functional properties namely wrinkle recovery, UV-protection properties, self-cleaning efficiency and antibacterial capability to wash. The data listed in Table 4 demonstrate that increasing the number of washing cycles from 1 to 10 is accompanied by a reasonable decrease in the imparted functional properties, which reflects the high extent of modification and immobilization of the used nanoparticles onto/within the nanofinished substrates, as discussed earlier. The remarkable improvement in the self-cleaning property (SC%) of the nanofinished cellulose/wool blended fabrics is attributed to the photocatalytic action of loaded nanomaterials, thereby increasing the extent of discoloration of MB-stains. On the other, hand, the extent of variation in the durability of the imparted functional properties is governed by the type of blended substrate, as discussed before [21, 35, 47, 48].

Surface morphology study and EDX analysis

The surface morphologies of the nanofinished viscose/wool and cotton/wool blended fabrics with TiO_2 -NPs and Ag-NPs individually or in combination are illustrated in Figs. 3, 4, 5, 6a, c, d using a different type of carboxylic acid as a crosslinking agent. All the images showed deposition of the nanoparticles onto the fiber surface. The nanofinished fabric samples with combined TiO_2 -NPs/Ag-NPs showed higher deposition of nanoparticles than the nanofinished fabric samples with TiO_2 -NPs or Ag-NPs, irrespective of the used substrate and ester crosslinking agent.

The EDX spectra and elemental analysis of nanofinished viscose/wool and cotton/wool fabric samples using CA/SHP as crosslinking are presented in Figs. 3 and 4b, d, f, respectively, while viscose/wool and cotton/wool fabric samples using SA/SHP as crosslinking are presented in Figs. 5 and 6b, d, f, respectively. All the Figures confirm the existence of the Ag, Ti and Ag/Ti elements in case of nanofinishing with AgNPs, and TiO_2 NPs individually and in combination, along other elements, i.e. carbon, nitrogen and oxygen. The extent of loading of the used nanoparticles is higher in case of using CA/SHP as an ester crosslinking system, regardless of the

Table 4 Durability of the imparted functional properties to wash

Finished substrate		V/W		C/W								
Functional property		DWRA (W + F)°		UPF	SC (%)	ZI (mm)	DWRA (W + F)°		UPF	SC (%)	ZI (mm)	
				G + ve	G – ve			G + ve	G – ve			G + ve
Washing cycles	one	280		90	75.2	22.5	20.5	265	120	69.9	21.0	19.5
	10	270		81	70	20.0	17.5	250	105	65.5	18.0	16.0
Untreated	230	15		10	4.0	3.0	210	24	12.0	3.0	2.0	

Finishing formulation: finishing agent (50 g/L), SHP (30 g/L), Ag/NPs/TiO₂ NPs (20/20 g/L), nonionic wetting agent (2 g/L), wet pick-up (85%), drying/ microwave fixation at 1300 W for 5 min

Fig. 3 SEM and EDX for wool/viscose fabric treated with AgNPs (40 g/L) (a, b), TiO₂NPs (40 g/L) (c, d), and AgNPs/TiO₂NPs (20/20 g/L) (e, f) in presence of citric acid (50 g/L)

used substrates. Moreover, treated viscose/wool Figs. 3 and 5b, d, f samples showed higher loading of NPs than cotton/wool treated samples Figs. 4 and 6b, d, f, regardless of the used carboxylic acid.

Conclusion

- In this study, an eco-friendly facile approach for multifunctionalization of cellulose/wool blended substrates using TiO₂ and/or AgNPs along with polycarboxylic acids was reported.

Fig. 4 SEM and EDX for wool/cotton fabric treated with AgNPs (40 g/L) (**a**, **b**), TiO₂NPs (40 g/L) (**c**, **d**), and AgNPs/TiO₂NPs (20/20 g/L) (**e**, **f**) in presence of citric acid (50 g/L)

- Factors affecting the extent of functionalization, namely type and concentration of nanoinorganic materials, kind and concentration of polycarboxylic acid as well as type of blended substrate were investigated.
- The obtained results demonstrated that the incorporation of TiO₂-Ag binary nanoparticles (20 g/L each) along with CA/SHF (50/30 g/L) in the finishing formulation of V/W and C/W substrates using the pad-dry-microwave fixation

Fig. 5 SEM and EDX for wool/viscose fabric treated with AgNPs (40 g/L) (a, b), TiO₂NPs (40 g/L) (c, d), and AgNPs/ TiO₂NPs (20/20 g/L) (e, f) in presence of succinic acid (50 g/L)

technique developed sustainable functional characteristics including antibacterial activity, UV-blocking efficacy, self-cleaning capability as well as antiwrinkle property before and after washing, regardless of the used substrate.

- The change in the fabric morphology and immobilization of nanoparticles onto some selected samples and durability to wash were confirmed.

Fig. 6 SEM and EDX for wool/cotton fabric treated with AgNPs (40 g/L) (a, b), TiO₂NPs (40 g/L) (c, d), and AgNPs/ TiO₂NPs (20/20 g/L) (e, f) in presence of succinic acid (50 g/L)

Funding Open access funding provided by The Science, Technology & Innovation Funding Authority (STDF) in cooperation with The Egyptian Knowledge Bank (EKB).

Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Open Access This article is licensed under a Creative Commons Attribution 4.0 International License, which permits use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons licence, and indicate if changes were made. The images or other third party material in this article are included in the article's Creative Commons licence, unless indicated otherwise in a credit line to the material. If material is not included in the article's Creative Commons licence and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission

directly from the copyright holder. To view a copy of this licence, visit <http://creativecommons.org/licenses/by/4.0/>.

References

1. Ibrahim NA, Emam E-AM, Eid BM, Tawfik TM (2018) An eco-friendly multifunctional nano-finishing of cellulose/wool blends. *Fibers Polym* 19(4):797–804. <https://doi.org/10.1007/s12221-018-7922-8>
2. Ibrahim NA, Eid BM, El-Batal H (2012) A novel approach for adding smart functionalities to cellulosic fabrics. *Carbohydr Polym* 87(1):744–751. <https://doi.org/10.1016/j.carbpol.2011.08.054>
3. Ibrahim NA, Abd El-Ghany NA, Eid BM, Mabrouk EM (2018) Green options for imparting anti-bacterial functionality to cotton fabrics. *Int J Biol Macromol* 111:526–533. <https://doi.org/10.1016/j.ijbiomac.2018.01.013>
4. Ibrahim NA, Aly AA, Eid BM, Fahmy HM (2018) Green approach for multifunctionalization of cellulose-containing fabrics. *Fibers Polym* 19(11):2298–2306. <https://doi.org/10.1007/s12221-018-8602-4>
5. Ibrahim NA, Eid BM, Sharaf SM (2019) Functional finishes for cotton-based textiles: current situation and future trends. In: Shabbir M (ed) *textiles and clothing*. Wiley and Scrivener Publishing LLC USA, pp. 131–190. <https://doi.org/10.1002/9781119526599.ch7>
6. Roy Choudhury AK (2017) Various ecofriendly finishes. In: Roy Choudhury AK (ed) *Principles of textile finishing*. Woodhead Publishing, pp. 467–525. <https://doi.org/10.1016/B978-0-08-100646-7.00014-X>
7. Albrecht MA, Evans CW, Raston CL (2006) Green chemistry and the health implications of nanoparticles. *Green Chem* 8(5):417–432. <https://doi.org/10.1039/b517131h>
8. Joshi M, Bhattacharyya A (2011) Nanotechnology – A new route to high-performance functional textiles. *Text Prog* 43(3):155–233. <https://doi.org/10.1080/00405167.2011.570027>
9. Riaz S, Ashraf M, Hussain T, Hussain MT, Rehman A, Javid A, Iqbal K, Basit A, Aziz H (2018) Functional finishing and coloration of textiles with nanomaterials. *Color Technol* 134(5):327–346. <https://doi.org/10.1111/cote.12344>
10. Ibrahim NA, Eid BM, Kafafy H (2020) Sustainable colorants for protective textiles. In: ul-Islam S, Butola BS (eds) *Advances in functional and protective textiles*. Woodhead Publishing, pp. 569–629. <https://doi.org/10.1016/B978-0-12-820257-9.00021-7>
11. Gulzar T, Farooq T, Kiran S, Ahmad I, Hameed A (2019) Green chemistry in the wet processing of textiles. In: Shahid ul I, Butola BS (eds) *The impact and prospects of green chemistry for textile technology*. Woodhead Publishing, pp. 1–20. <https://doi.org/10.1016/B978-0-08-102491-1.00001-0>
12. Mishra R, Militky J, Baheti V, Huang J, Kale B, Venkataraman M, Bele V, Arumugam V, Zhu G, Wang Y (2014) The production, characterization and applications of nanoparticles in the textile industry. *Text Prog* 46(2):133–226. <https://doi.org/10.1080/00405167.2014.964474>
13. Agarwal H, Venkat Kumar S, Rajeshkumar S (2017) A review on green synthesis of zinc oxide nanoparticles – An eco-friendly approach. *Resour-Eff Technol* 3(4):406–413. <https://doi.org/10.1016/j.reffit.2017.03.002>
14. Montazer M, Harifi T (2018) Nanofinishes for protective textiles. In: Montazer M, Harifi T (eds) *Nanofinishing of textile materials*. Woodhead Publishing, pp. 265–294. <https://doi.org/10.1016/B978-0-08-101214-7.00018-2>
15. Verma A, Arif R, Jadoun S (2020) Synthesis, characterization, and application of modified textile nanomaterials. In: *Frontiers of textile materials*. pp. 167–187. <https://doi.org/10.1002/9781119620396.ch8>
16. Pant B, Park M, Park S-J (2019) One-step synthesis of silver nanoparticles embedded polyurethane nano-fiber/net structured membrane as an effective antibacterial medium. *Polymers* 11(7):1185
17. Pant B, Saud PS, Park M, Park S-J, Kim H-Y (2016) General one-pot strategy to prepare Ag–TiO₂ decorated reduced graphene oxide nanocomposites for chemical and biological disinfectant. *J Alloy Compd* 671:51–59. <https://doi.org/10.1016/j.jallcom.2016.02.067>

18. Pant B, Pant HR, Pandeya DR, Panthi G, Nam KT, Hong ST, Kim CS, Kim HY (2012) Characterization and antibacterial properties of Ag NPs loaded nylon-6 nanocomposite prepared by one-step electrospinning process. *Colloids Surf, A* 395:94–99. <https://doi.org/10.1016/j.colsurfa.2011.12.011>
19. Dong Y, Bai Z, Zhang L, Liu R, Zhu T (2006) Finishing of cotton fabrics with aqueous nano-titanium dioxide dispersion and the decomposition of gaseous ammonia by ultraviolet irradiation. *J Appl Polym Sci* 99(1):286–291. <https://doi.org/10.1002/app.22476>
20. Vigneshwaran N, Nachane RP, Balasubramanya RH, Varadarajan PV (2006) A novel one-pot 'green' synthesis of stable silver nanoparticles using soluble starch. *Carbohydr Res* 341(12):2012–2018. <https://doi.org/10.1016/j.carres.2006.04.042>
21. Karimi L, Mirjalili M, Yazdanshenas ME, Nazari A (2010) Effect of nano TiO₂ on self-cleaning property of cross-linking cotton fabric with succinic acid under UV irradiation. *Photochem Photobiol* 86(5):1030–1037. <https://doi.org/10.1111/j.1751-1097.2010.00756.x>
22. Hashemikia S, Montazer M (2012) Sodium hypophosphite and nano TiO₂ inorganic catalysts along with citric acid on textile producing multi-functional properties. *Appl Catal A* 417–418:200–208. <https://doi.org/10.1016/j.apcata.2011.12.041>
23. Ibrahim NA, Eid BM, Abdel-Aziz MS (2017) Effect of plasma superficial treatments on antibacterial functionalization and coloration of cellulosic fabrics. *Appl Surf Sci* 392:1126–1133. <https://doi.org/10.1016/j.apsusc.2016.09.141>
24. Nazari A, Montazer M, Rashidi A, Yazdanshenas M, Anary-Abbasinejad M (2009) Nano TiO₂ photo-catalyst and sodium hypophosphite for cross-linking cotton with poly carboxylic acids under UV and high temperature. *Appl Catal A Gen* 371:10–16. <https://doi.org/10.1016/j.apcata.2009.08.029>
25. Yuen CWM, Ku SKA, Li Y, Cheng YF, Kan CW, Choi PSR (2009) Improvement of wrinkle-resistant treatment by nanotechnology. *J Text Inst* 100(2):173–180. <https://doi.org/10.1080/00405000701661028>
26. Wu Y, Yang Y, Zhang Z, Wang Z, Zhao Y, Sun L (2019) Fabrication of cotton fabrics with durable antibacterial activities finishing by Ag nanoparticles. *Text Res J* 89(5):867–880. <https://doi.org/10.1177/0040517518758002>
27. Montazer M, Behzadnia A, Pakdel E, Rahimi MK, Moghadam MB (2011) Photo induced silver on nano titanium dioxide as an enhanced antimicrobial agent for wool. *J Photochem Photobiol, B* 103(3):207–214. <https://doi.org/10.1016/j.jphotobiol.2011.03.009>
28. Ibrahim NA, Eid BM, Khalil HM, Almetwally AA (2018) A new approach for durable multifunctional coating of PET fabric. *Appl Surf Sci* 448:95–103. <https://doi.org/10.1016/j.apsusc.2018.04.022>
29. Rashid MM, Simončič B, Tomšič B (2021) Recent advances in TiO₂-functionalized textile surfaces. *Surf Interfaces* 22:100890. <https://doi.org/10.1016/j.surf.2020.100890>
30. Azam A, Ahmed AS, Oves M, Khan MS, Habib SS, Memic A (2012) Antimicrobial activity of metal oxide nanoparticles against Gram-positive and Gram-negative bacteria: a comparative study. *Int J Nanomed* 7:6003–6009
31. Arik B, Karaman Atmaca OD (2020) The effects of sol–gel coatings doped with zinc salts and zinc oxide nanopowders on multifunctional performance of linen fabric. *Cellulose* 27(14):8385–8403. <https://doi.org/10.1007/s10570-020-03322-3>
32. Ibrahim NA, Eid BM, Khalil HM (2015) Cellulosic/wool pigment prints with remarkable antibacterial functionalities. *Carbohydr Polym* 115:559–567. <https://doi.org/10.1016/j.carbpol.2014.09.013>
33. Li S, Zhu T, Huang J, Guo Q, Chen G, Lai Y (2017) Durable antibacterial and UV-protective Ag/TiO₂@ fabrics for sustainable biomedical application. *Int J Nanomed* 12:2593–2606
34. Li G, Liu H, Zhao H, Gao Y, Wang J, Jiang H, Boughton RI (2011) Chemical assembly of TiO₂ and TiO₂@Ag nanoparticles on silk fiber to produce multifunctional fabrics. *J Colloid Interface Sci* 358(1):307–315. <https://doi.org/10.1016/j.jcis.2011.02.053>
35. Ibrahim NA, El-Zairy EM, Eid BM, Emam E, Barkat SR (2017) A new approach for imparting durable multifunctional properties to linen-containing fabrics. *Carbohydr Polym* 157:1085–1093. <https://doi.org/10.1016/j.carbpol.2016.10.074>
36. Ibrahim NA, Eid BM, Abdel-Aziz MS (2016) Green synthesis of AuNPs for eco-friendly functionalization of cellulosic substrates. *Appl Surf Sci* 389:118–125. <https://doi.org/10.1016/j.apsusc.2016.07.077>
37. Ibrahim NA, Abo-Shosha MH, Elnagdy EI, Gaffar MA (2002) Eco-friendly durable press finishing of cellulose-containing fabrics. *J Appl Polym Sci* 84(12):2243–2253. <https://doi.org/10.1002/app.10467>

38. Hashem M, Ibrahim NA, El-Shafei A, Refaie R, Hauser P (2009) An eco-friendly – novel approach for attaining wrinkle – free/soft-hand cotton fabric. *Carbohydr Polym* 78(4):690–703. <https://doi.org/10.1016/j.carbpol.2009.06.004>
39. Zanolto CD, Lansarin MA (2019) Application of polycarboxylic acids as binders for TiO₂ immobilization on cotton textiles. *Braz J Chem Eng* 36(1):181–190
40. Huang W, Xing Y, Yu Y, Shang S, Dai J (2011) Enhanced washing durability of hydrophobic coating on cellulose fabric using polycarboxylic acids. *Appl Surf Sci* 257(9):4443–4448. <https://doi.org/10.1016/j.apsusc.2010.12.087>
41. Wang C-C, Chen C-C (2005) Physical properties of the crosslinked cellulose catalyzed with nano-titanium dioxide under UV irradiation and electronic field. *Appl Catal A* 293:171–179. <https://doi.org/10.1016/j.apcata.2005.07.007>
42. Ibrahim NA, Eid BM, Abdellatif FHH (2018) Advanced materials and technologies for antimicrobial finishing of cellulosic textiles. In: *Handbook of renewable materials for coloration and finishing*. Pp. 301–356. <https://doi.org/10.1002/9781119407850.ch13>
43. Dastjerdi R, Montazer M, Shahsavani S (2010) A novel technique for producing durable multifunctional textiles using nanocomposite coating. *Colloids Surf, B* 81(1):32–41
44. Ibrahim NA, Nada AA, Eid BM, Al-Moghazy M, Hassabo AG, Abou-Zeid NY (2018) Nano-structured metal oxides: synthesis, characterization and application for multifunctional cotton fabric. *Adv Nat Sci: Nanosci Nanotechnol* 9(3):035014
45. Gaidau C, Petica A, Ignat M, Iordache O, Ditu L-M, Ionescu M (2016) Enhanced photocatalysts based on Ag-TiO₂ and Ag-N-TiO₂ nanoparticles for multifunctional leather surface coating. *Open Chem* 14(1):383–392
46. Viet PV, Phan BT, Mott D, Maenosono S, Sang TT, Thi CM, Hieu LV (2018) Silver nanoparticle loaded TiO₂ nanotubes with high photocatalytic and antibacterial activity synthesized by photoreduction method. *J Photochem Photobiol, A* 352:106–112. <https://doi.org/10.1016/j.jphotochem.2017.10.051>
47. Ibrahim NA, Eid BM, El-Aziz EA, Elmaaty TMA, Ramadan SM (2017) Loading of chitosan – Nano metal oxide hybrids onto cotton/polyester fabrics to impart permanent and effective multifunctions. *Inter J Biol Macromol* 105 (Part 1):769–776. <https://doi.org/10.1016/j.ijbiomac.2017.07.099>
48. Saad SR, Mahmed N, Abdullah MMAB, Sandu AV (2016) Self-cleaning technology in fabric: a review. *IOP Conf Series: Mater Sci Eng* 133:012028. <https://doi.org/10.1088/1757-899x/133/1/012028>

Publisher's Note Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.