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An Eco-Friendly Multifunctional Nano-Finishing of Cellulose/Wool Blends

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Abstract: A new approach for an eco-friendly multi-functionalization of cotton/wool (C/W) and viscose/wool (V/W) blended fabrics was investigated. In this study, Ag-nanoparticle (Ag-NP) and/or ZnO-nanoparticle (ZnO-NP) functional agents were incorporated into the finishing bath along with citric acid (CA) or succinic acid (SA) as ester-crosslinking or esterifying agent, and sodium hypophosphite catalyst using the padding technique. The obtained results indicated that the extent of multi-functionalization expressed as antibacterial activity, UV-blocking functionality and wrinkle recovery ability were determined by kind of nanomaterial, nature of carboxylic acid, i.e., bi- or tri-functional and type of substrate. The results also demonstrated that blended fabrics finished with Ag-NP/ZnO-NP/CA/SHP nano-finishing formulation exhibited outstanding durable multi-functional properties even after 10 washing cycles. In addition, the change in surface morphology and the existence of Ag and/or Zn onto the selected V/W fabric surfaces have been confirmed by SEM and EDX analysis respectively.

Keywords: Cellulose/wool blends, Nanosized Ag and ZnO, Eco-friendly nano-finishing, Synergistic effect, Durable multi-functional properties

Introduction

Application of nanotechnology in textile wet processing as a useful emerging tool for upgrading the product quality, improving the performance as well as increasing added value of textiles made from natural and/or synthetic fibers, taking into consideration the environmental concerns, is growing fast [1-4].

Recently, potential applications of metals, e.g. Ag, Au, and metal oxides, e.g. ZnO, TiO_2, Al_2O_3, MgO, SiO_2 nanomaterials in the realm of textile finishing have attracted a great deal of attention to add new capabilities and functionalities, e.g. antibacterial ability, UV-protection, self-cleaning, easy care, water/oil repellency as well as to meet the increasing demand for performance-enhanced/value-added textile materials [5-14].

Very recently, Ibrahim *et al*. have already highlighted several options for multi-functionalization of linen containing fabrics via pre-carboxymethylation to create new active sites followed by loading of different type of ingredients such as chitosan, organosilane quaternary ammonium compound, Ag-NP, and TiO_2-NP alone and in admixtures [8]. Additionally, enhancing the antibacterial properties of pigment prints is carried out in one step by individual incorporation of several bioactive ingredients such as choline chloride, triclosan derivative, PEG-600, and 4-hydroxybenzophenone into the pigment print formulation followed by microwave fixation. The obtained results demonstrate significant improvement in functional and coloration properties of the obtained prints [15]. All the treated substrates showed a high durability to wash even after 15 washing cycles.

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On the other hand, Kiwi and Pulgarin have studied the feasibility of producing self-cleaning and bactericide textiles by loading TiO_2, in the form of colloid and/or powder, onto textile materials followed by curing at proper temperatures. The obtained functionalities have been achieved in the presence of O_2 by RF-plasma and vacuum-UV to improve the adhesion of the TiO_2 on the textiles [10]. Additionally, multi-functional cotton fabric has fabricated by using sol gel and solvothermal techniques for coating cotton fabric with silica and titania sols in the presence of quaternary ammonium salt and silver salts, as antimicrobial doping agent, to attain cotton fabric with antimicrobial, self-cleaning, and UV-protective properties. The obtained results showed that the enhancement in the obtained functional properties were governed by the type of the used additive [9].

Currently, one of the most common and facile techniques to impart multi-functional cellulose-containing fabrics is co-application of an eco-friendly crosslinking and binding agents like polycarboxylic acids along with other active ingredients such as nano-metals using sodium hypophosphite as a proper catalyst in a single-stage process [16-21]. Ibrahim *et al.* have prepared a new water-soluble poly(acrylic acid)/poly(ethylene glycol) adduct and were successfully used as an ester cross linking and binding agent for Ag-NP and TiO_2-NP on cotton fabric in presence of Na-hypophosphite as catalyst to impart multi-functional properties [18]. While Karthik *et al.* have used citric acid as a crosslinking agent and Na-hypophosphite as a catalyst in presence of TiO_2-NP as a co-catalyst to improve the mechanical and physical properties of treated cotton fabric [17]. On the other hand, Sunder and Nalankilli have investigated the use of different polycarboxylic acids namely maleic acid, citric acid, tartaric acid, and itaconic acid as a crosslinking agent alone or together

to improve both functional and performance properties of treated cotton fabric [21].

Herein, we report the technical feasibility of imparting new/durable functional properties to cellulose/wool blended fabrics in a single-stage process via inclusion of Ag-nanoparticles (Ag-NP) alone and in combination with zinc oxide nanoparticles (ZnO-NP), as functional materials into the zero-formaldehyde finishing formulations. Consequently, the best treatment conditions for optimizing the imparted characteristics of the treated substrates were demonstrated.

Experimental

Materials

Mill-scoured and bleached cotton/wool (C/W, 50/50, 230 g/m^2) and viscose/wool (V/W, 50/50, 190 g/m^2) were used in this study.

Citric acids (CA), succinic acid (SA), sodium hypophosphite monohydrate (SHP, $NaPo_2H_2 \cdot H_2O$) as well as other chemicals were of reagent grade.

Ag-nanopartlices (Ag-NP) and zinc oxide nanoparticles (ZnO-NP) were synthesized according to using procedures described by [8] and [22], respectively (Figure 1).

Methods

Functional Finishing

The aqueous finishing dispersion was prepared by mixing the carboxylic acid (CA or SA) and the used catalyst, SHP, with the proper amount of the nominated nano-materials, Ag-NP and/or ZnO-NP, and distilled water in ultrasonic bath for 20 min.

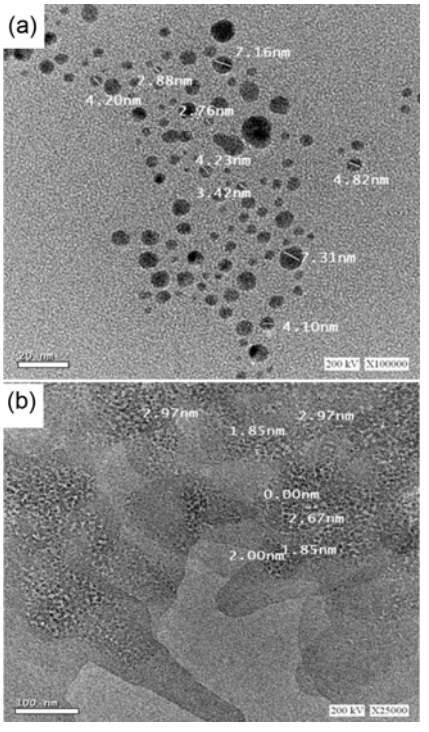


Figure 1. TEM images of (a) Ag-NP (with size ranged from 2 to 8 nm) and (b) ZnO-NP (with size ranged from 1 to 3 nm).

The nominated substrates were padded twice with 80 % wet pick-up by freshly prepared aqueous finishing solutions. Padded fabric samples were microwaved dried/fixed at 1300 W for 4 min. The treated fabric samples were washed at 50 ^oC for 10 min, rinsed thoroughly to remove excess and unfixed reactants and finally dried.

Testing Methods

Silver and zinc elements were quantitatively determined by using a flame atomic absorption spectrophotometer, GBC-Avanta, Australia.

Dry wrinkle recovery angle (WRA) of the fabric samples was 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) expressed as zone of growth inhibition (ZI, mm).

UV-protection factor (UPF) was evaluated according to the Australian/New Zealand standard (AS/NZS 4366-1996).

The durability to wash was assessed after 10 laund ering cycles according to 61(2A)-1996: Colorfastness to Laundering, Home and Commercial: Accelerated (AATCC, 2002). Laundering conditions outlined in Test 2A: for fabric that are expected to withstand repeated low temperature machine washings.

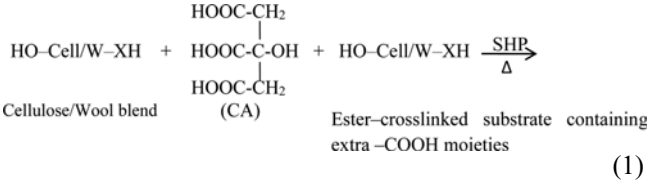
Scanning electronic microscope (SEM) images of selected samples were evaluated using a JEOL, JXA-840A electron probe microanalyzer equipped with disperse X-ray Spectro-photometer (EDX) for the composition analysis.

Results and Discussion

Tentative Mechanism

Functional finishing of C/W and V/W blended fabrics using CA or SA as an eco-friendly esterifying/binding agent, SHP, as an efficient esterification catalyst, along with Ag-NP and/or ZnO-NP, followed by microwave fixation would be expected to promote the following interactions.

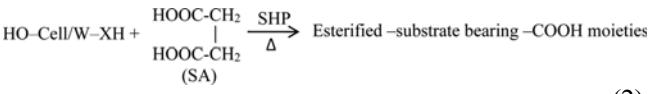
Ester-Crosslinking Using CA



(1)

via formation of two five membered cyclic anhydrides as reactive intermediates and subsequent reaction with the substrate active site, -XH, such as -OH, -NH_2, and -COOH to complete the formation of ester-linkages [16,23].

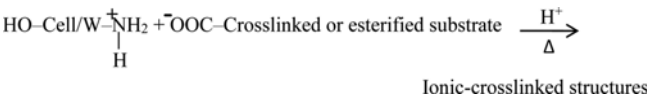
Single-Ended Esterification Reaction Using SA



(2)

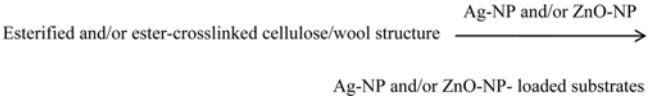
via formation of one anhydride intermediate and subsequent esterification of the nominated substrate active sites [16].

Ionic-crosslinking [24,25]



(3)

Loading of Nanoparticles



(4)

via chelation and electrostatic interactions between the nanomaterials and the -COOH, -NH_2, and -OH potential ligands in the modified cellulose/wool structure [6,8,26,27].

Ag-NP/CA or SA Nano-Finishing System

As far as the changes in Ag-content, antibacterial activity, expressed as ZI, against both the pathogenic Gram-positive (*S. aureus*) and Gram negative (*E. coli*) bacteria as well as in fabric resiliency, expressed as WRA, as a function of Ag-NP concentration, type of carboxylic acid and kind of cellulose/wool blend, Table 1 demonstrates that increasing Ag-NP concentration from 25 up to 50 g/*l* results in an increase in Ag-content, an improvement in antibacterial ability as well as in fabric resiliency irrespective of the used polycarboxylic acid and the treated substrate. The increase in Ag-content is attributed to the holding capacity of Ag-NP and extent of loading onto the untreated, esterified and/or ester-crosslinked substrates via chelation and electrostatic interactions between Ag-NP and the potential reaction sites of unmodified, modified and ester-crosslinked substrates such as -OH, -NH_2, and -COOH (equation (4)). The extent of loading follows the decreasing orders: ester-crosslinked > esterified > blank, and CA > SA ≥ None. It can be observed that the higher the concentration of Ag-NP, the larger the Ag-content of the treated substrates. Clearly it can be seen in Table 1 that the higher the Ag-content, the greater the imparted antibacterial activity to the treated substrates. The imparted antibacterial functionality is determined by type of substrate and kind of carboxylic acid and follows the decreasing order: ester-crosslinked > esterified > blank, keeping other parameters fixed, which is a direct consequence of variation in extent of loading of the nominated nanoparticles onto the treated substrates, most probably due to the differences in number, location, extent of distribution, and availability of binding/anchoring sites onto/within the fabrics structure [28]. The antibacterial efficacy against the nominated pathogens follows the decreasing order: Gram-positive (*S. aureus*) > Gram negative (*E. coli*), reflecting the difference in the nominated bacteria in cell wall structure in addition to their amenability to damage and deactivation by the leached Ag^+ and Ag-NP [15,29].

On the other hand, the imparted antibacterial functionality to the Ag-NP-loaded substrates is attributed to the controlled release of Ag-NP, Ag^+ via:

Table 1. Functional properties of cellulose/wool blended fabrics treated with Ag-NP/carboxylic acid nano-finishing system

Carboxylic acids	Substrate	Ag-NP (g/*l*)	Ag- content (%)	ZI (mm)		WRA (W+F)°
				Gram-positive	Gram-negative	
None	C/W	25	0.013	4.0	3.0	200
		50	0.020	7.5	6.0	210
	V/W	25	0.024	7.0	6.0	215
		50	0.036	10.0	9.0	220
Citric acid	C/W	25	0.056	14.5	13.0	225
		50	0.080	20.0	18.0	230
	V/W	25	0.088	18.0	16.5	240
		50	0.134	24.0	22.0	254
Succinic acid	C/W	25	0.032	10.0	8.0	208
		50	0.048	17.0	15.5	214
	V/W	25	0.052	13.0	11.5	219
		50	0.073	21.0	19.0	229

Finishing bath: carboxylic acid (45 g/*l*); SHP (25 g/*l*); Ag-NP (25 and 50 g/*l*); nonionic wetting agent (2 g/*l*); pH (5); wet pick-up (80 %), microwave fixation at 1300 W for 4 min, ZI: zone of inhibition; WRA: wrinkle recovery angle (warp+weft); Gram-positive bacterial: *S. aureus*; Gram-negative bacterial: *E. coli.*

$$4\ \text{Ag-NP} + 2H_2O + O_2 \longrightarrow 4\ Ag^+ + 4\ OH^- \quad (5)$$

as well as the formation of reactive oxygen species (ROS) via:

$$H_2O + {}^1/_2\ O_2 \xrightarrow{Ag^+ \ \& \ \text{Ag-NP}} H_2O_2 \longrightarrow H_2O + \text{ROS [O]} \quad (6)$$

and their ability to: damage the cell wall, alter the cell membrane permeability, disturb/deactivate physiological functions of bacterial, hinder enzymatic functions of the protein as well as the negative impacts of the generated ROS on lipids, proteins and DNA of bacteria and consequently destruction of the bacterial cells [15,29].

Results of Table 1 signify that using CA, as an eco-friendly crosslinking and binding agent, is accompanied by a remarkable enhancement in the fabric resiliency, expressed as WRA, regardless of the used substrate. The improvement in fabrics resiliency is attributed to the formation of ester-crosslinks (equation (1)) and ionic crosslinks (equation (3)) between adjacent fibers or microfibrills [16,23]. The reasonable improvement in WRA of the SA-finished fabric samples most probably is attributed to the formation of ionic crosslinking (equation (3)). On the other hand, the extent of improvement in the imparted easy-care properties governed by type of carboxylic acid, i.e. bi- or poly-functional, kind of substrate, e.g. fabric weight, components, amorphous/ crystalline ratio, extent of penetration, and diffusion within the fabric structure, type/number/location/accessibility of active site, etc. [26,28], as well as mode of interaction, i.e. esterification (single ended reaction), ester-crosslinking, and/or ionic-crosslinking [16].

ZnO-NP/CA or SA Nano-Finishing System

The results in Table 2 clearly demonstrate that increasing ZnO-NP in the finishing bath results in an increasing in the Zn- content, antibacterial activity, UV-shielding capacity along with a slight improve in treated fabrics resiliency. The extent of improvement in the aforementioned properties is determined by kind of carboxylic acid, i.e. CA > SA >> None, as well as type of substrate V/W > C/W, keeping other parameters constant. The Zn-content depends on chemical structure and morphological structure of cellulose and wool fiber components, mode of binding, adhesion ability, adsorption capacity, along with ability of fibers active sites, and follows the decreasing order V/W > C/W. The remarkable improvement in the imparted antibacterial ability of ZnO-loaded substrates against both Gram-positive (*S. aureus*) and Gram negative (*E. coli*) bacteria reflects their ability to generate extremely reactive oxygen species, i.e. ROS, like super oxide anions ($\bullet O_2^-$), H_2O_2, •OH, single oxygen, etc. as follows [30]:

$$\text{ZnO-NP} + h\nu \longrightarrow \text{ZnO} + h^+ + e^- \quad (7)$$

$$h^+ + H_2O \longrightarrow \dot{O}H + \overset{+}{H} \quad (8)$$

$$e^- + O_2 \longrightarrow \dot{O}_2^- \quad (9)$$

$$\dot{O}_2^- + \overset{+}{H} \longrightarrow H\dot{O}_2 \quad (10)$$

$$2H\dot{O}_2 \longrightarrow O_2 + H_2O_2 \quad (11)$$

which have the ability to destruct the pathogenic bacteria cells [31], along with abrasion and damaging effects of ZnO-NP on the bacteria cell [32]. The imparted antibacterial activity against the nominated pathogenic bacteria follows the descending order: Gram-positive>Gram negative, keeping type of substrate and carboxylic acid fixed. The improvement in UPF values of ZnO-NP loaded substrates reflects their ability to block the harmful UV-B radiation due to their increased surface area and intense absorption in the UV-

Table 2. Functional properties of cellulose/wool blended fabrics treated with ZnO-NP/carboxylic acid nano-finishing system

Carboxylic acids	Substrate	ZnO-NP (g/*l*)	ZnO-content (%)	ZI (mm)		UPF	WRA (W+F)°
				Gram-positive	Gram-negative		
None	C/W	25	0.046	4.0	3.0	24	210
		50	0.070	8.0	6.5	30	217
	V/W	25	0.062	7.5	6.0	18	222
		50	0.091	12.0	11.0	25	230
Citric acid	C/W	25	0.114	12.5	10.0	51	255
		50	0.133	14.5	13.5	74	264
	V/W	25	0.146	16.5	15.0	36	270
		50	0.169	20.5	19.5	66	282
Succinic acid	C/W	25	0.088	11.0	9.0	40	237
		50	0.104	13.5	12.0	56	245
	V/W	25	0.099	14.5	13.0	27	248
		50	0.120	17.5	16.0	45	255

Finishing bath: carboxylic acid (45 g/*l*); SHP (25 g/*l*); Ag-NP (25 and 50 g/*l*); nonionic wetting agent (2 g/*l*); pH (5); wet pick-up (80 %), microwave fixation at 1300 W for 4 min, ZI: zone of inhibition; UPF: UV-protection factor; WRA: wrinkle recovery.

range rather than reflecting and/or scattering [1,2,7]. The extent of improvement in the UV-blocking property is governed by type of substrate, e.g. fabric construction, weigh, thickness, etc. as well as extent of loading and homogeneous distribution of ZnO-NP onto and/or within the fabric structure and follows the decreasing order: C/W > V/W, keeping other parameters constant.

On the other hand, the substantial improvement in the WRA values is determined by type of carboxylic acid, mode of interaction and extent of ester-crosslinking and/or ionic crosslinking and follows the decreasing orders: CA > SA > None. Increasing ZnO-NP concentration has practically a marginal positive impact on enhancing fabric resiliency.

Ag-NP/ZnO-NP Mixture/CA or SA Nanofinishing System

It is quite clear from Table 3 that using Ag-NP/ZnO-NP nano-composite for multi-functionalization of the nominated substrates along with CA, as an ester-crosslinking agent, or

Table 3. Functional properties of cellulose/wool blended fabrics treated with Ag-NP/ZnO-NP/carboxylic acid nanofinishing system

Carboxylic acids	Substrate	Nano-material (50 g/*l*)	Metal content (%)	ZI (mm)		UPF	WRA (W+F)°
				Gram-positive	Gram-negative		
None	C/W	Ag-NP	0.020 (0.013)	7.5 (5.5)	6.0 (4.0)	21 (17)	210 (205)
		Ag-NP/ZnO-NP	0.011/0.031 (0.007)/(0.019)	9.5 (7.0)	8.0 (6.0)	30 (25)	215 (210)
		ZnO-NP	0.070 (0.0462)	8.0 (6.0)	6.5 (5.0)	26 (23)	220 (215)
	V/W	Ag-NP	0.036 (0.025)	10.0 (8.0)	9.0 (7.0)	17 (14)	220 (212)
		Ag-NP/ZnO-NP	0.016/0.050 (0.010)/(0.033)	13.0 (10.0)	12.0 (9.5)	25 (20)	225 (215)
		ZnO-NP	0.091 (0.061)	12.0 (9.5)	11.0 (8.0)	22 (18)	230 (222)
CA	C/W	Ag-NP	0.080 (0.069)	20.0 (17.0)	18.0 (15.5)	32 (28)	230 (223)
		Ag-NP/ZnO-NP	0.035/0.085 (0.028)/(0.073)	21.5 (18.5)	19.0 (16.5)	74 (64)	242 (234)
		ZnO-NP	0.133 (0.112)	14.5 (12.0)	13.5 (11.0)	50 (43)	264 (256)
	V/W	Ag-NP	0.134 (0.114)	24.0 (21.0)	22.0 (18.5)	25 (21)	254 (245)
		Ag-NP/ZnO-NP	0.059/0.094 (0.050)/(0.080)	25.0 (21.5)	24.0 (21.0)	66 (60)	265 (255)
		ZnO-NP	0.169 (0.142)	20.0 (17.5)	19.5 (17.5)	44 (38)	282 (274)
SA	C/W	Ag-NP	0.048 (0.035)	17.0 (13.0)	15.5 (12.5)	28 (23)	214 (206)
		Ag-NP/ZnO-NP	0.027/0.056 (0.021)/(0.041)	19.0 (15.5)	17.0 (13.5)	56 (49)	229 (222)
		ZnO-NP	0.104 (0.076)	13.5 (11.0)	12.0 (9.5)	45 (40)	245 (237)
	V/W	Ag-NP	0.073 (0.053)	21.0 (17.0)	19.0 (15.5)	22 (18)	229 (221)
		Ag-NP/ZnO-NP	0.030/0.0754 (0.021)/(0.055)	22.5 (18.0)	20.0 (16.5)	45 (38)	240 (233)
		ZnO-NP	0.120 (0.094)	17.5 (14.0)	16.0 (13.0)	39 (32)	255 (244)

Finishing bath: CA or SA (45 g/*l*); SHP (25 g/*l*); Ag-NP (50 g/*l*); ZnO-NP (50 g/*l*); Ag-NP/ZnO-NP (25/25 g/*l*); nonionic wetting agent (2 g/*l*); pH (5); wet pick-up (80 %), microwave fixation at 1300 W for 4 min, ZI: zone of inhibition; UPF: UV-protection factor; WRA: wrinkle recovery. (-): values in parentheses indicate the retained functional properties after 10 washing cycles.

SA, as an esterifying agent, and in the presence of SHP, a an esterification catalyst, results in a significant improvement in the imparted antibacterial functionality against the nominated pathogenic bacteria. The variation in antibacterial activity upon using Ag-NP and ZnO-NP alone and in admixture follows the decreasing order: Ag-NP/ZnO-NP > Ag-NP > ZnO-NP, keeping the substrate type constant, which reflects the synergistic effect of applying Ag-NP/ZnO-NP nanocomposite thereby increasing the photocatalytic activity [33]. The high antibacterial performance is attributed to the ability of nanocomposite particles to damage bacterial membrane, to present into bacterial cells and interact with specific target, to suppress the growth of bacteria as well as to generate highly reactive oxygen species (ROS) thereby leading to the loss of membrane integrity, malfunction and finally to bacteria death [34,35]. The variation in metal content values upon using the nominated nanomaterials reflects their differences in size, extent of loading as well as binding to the nominated substrates via their reactive sites. The enhancement in UV-blocking ability, expressed as UPF values, of the nanomaterials-loaded substrates follows the decreasing order: Ag-NP/ZnO-NP > ZnO-NP >> Ag-NP, regardless of the used substrates, most probable is a direct consequence of increasing the UV-B absorption and blocking capability of the Ag-NP/ZnO-NP nanocomposite-loading fabric samples, i.e. higher UV-protection [36]. The increase in fabric resiliency follows the decreasing order: ZnO-NP > Ag-NP/ZnO-NP > Ag-NP which reflects the differences among the nominated nanometerials in enhancing the extent of co-catalyzing the esterification and/or ester-crosslinking interactions, especially ZnO-NP, along with the used SHP used catalyst [37]. The variation in the imparted functional properties is determined by type of the substrate as well as kind of carboxylic acid as discussed before. The developed antibacterial activity against the Gram positive (*S. aureus*) and Gram negative (*E. coli*) pathogenic bacteria

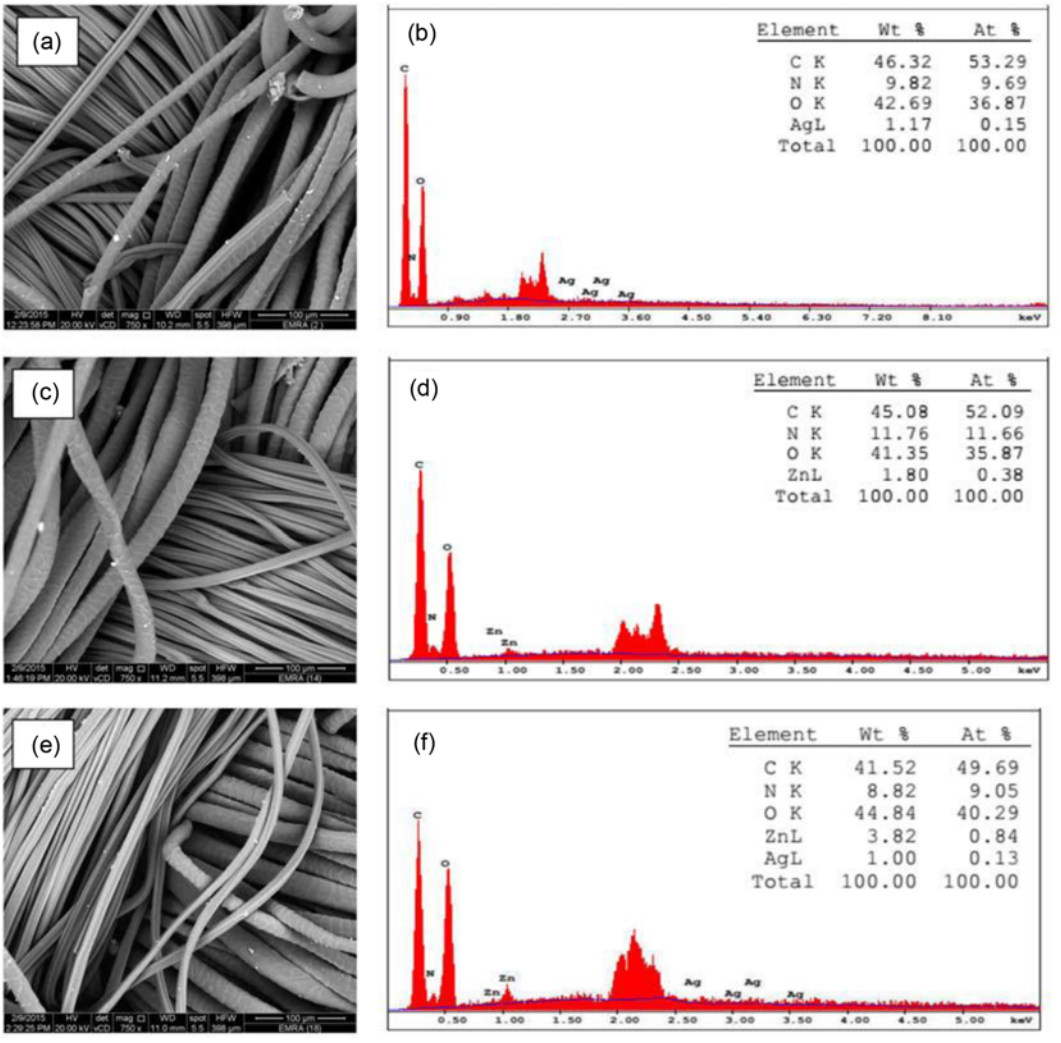


Figure 2. SEM & EDX for Viscose/Wool fabric treated with citric acid (45 g/*l*) and AgNPs (50 g/*l*) (a,b), ZnONPs (50 g/*l*) (c,d), or AgNPs/ZnONPs (25/25 g/*l*) (e,f).

follows the decreasing order: Gram positive > Gram negative due to their differences in cell wall structure [29]. Increasing the number of washing cycles from one to 10 results in a slight decrease in the imparted functional properties reflecting the noticeable efficient of the suggest nanofinishing systems.

SEM & EDX Analysis

The results of SEM-EDX analysis of Ag-NP/ CA, ZnO-NP/CA and Ag-NP/ZnO-NP/CA nano-finished V/W fabric samples are presented in Figure 2(a,b), (c,d), and (e,f) respectively. SEM images demonstrate surface deposits on treated fabric samples. On the other hand, EDX elemental analysis confirms the presence of Ag, Zn, and Ag/Zn elements on the treated fabric samples respectively.

Conclusion

In this research, the effect of nano-finishing of C/W and V/W blended fabrics using nanosized Ag-NP and/or ZnO-NP along with CA or SA/SHP using the pad dry/microwave fixation technique was investigated. Antibacterial activity, UV-blocking ability and fabric resiliency functional properties as well as washing durability of the imparted functional properties were evaluated. An ideal antibacterial, UV-protection and anticrease properties can be achieved by applying Ag-NP/ ZnO-NP nanocomposite (25/25 g/*l*), citric acid (45 g/*l*), and SHP (25 g/*l*), followed by padding (80 % wet pick-up), and drying/ microwave thermofixing (1300 W for 4min). SEM images and EDX spectra of selected fabric samples showed variation in the treated fabric surfaces morphology and confirmed the existence of Ag- and/or Zn elements respectively on the treated fabrics. After 10 washing cycles, the nano-finished fabric samples still retained remarkable functional properties.

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