

New finishing possibilities for producing durable multifunctional cotton/wool and viscose/wool blended fabrics



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ABSTRACT

This research work focuses on the development of a one-bath functional finishing procedure for imparting durable multifunctional properties such as easy care, soft-hand, antibacterial and/or ultra violet (UV) protection to cotton/wool and viscose/wool blends using diverse finishing combinations and formulations. In this study finishing agents such as reactant resin, silicon softeners, 4-hydroxybenzophenone, triclosan, and pigment colorant were selected using magnesium chloride/citric acid as a mixed catalyst and the pad-dry microwave fixation technique. The results reveal that enhancement in the imparted functional properties are governed by type of the finished substrate as well as nature and concentration of finishing formulation components. The finished fabrics still retained high level of functionalities even after 15 consecutive laundering. Surface morphology and composition of selected samples were investigated using scan electron microscope (SEM) and energy-dispersive X-ray spectroscopy (EDX) analysis. The mode of interactions was also investigated. Practical applications for multifunctionalization of cellulose/wool blended fabrics are possible using these sorts of proper finishing formulations and unique finishing application method.

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1. Introduction

Cellulose is the most abundant natural biopolymer composed of β -D-glucopyranose units linked together by β -1-4-linkage. Each unit bears three accessible hydroxyl groups at C₂, C₃ and C₆ sites. Its degree of polymerization depends on its source as well as processing stages. Textile materials based on cellulosic fibres can either obtained from respective plants such as cotton, flax, jute, ramie, sisal or by dissolution and regeneration of cellulosic materials like viscose rayon and Tencel (Buschle-Diller, 2003).

Recently, there has been a great interest in modifying the surface, mechanical, physical, chemical and/or functional properties of cellulosic materials to correct their defects, to improve their inherent properties, and to add or impart new functional properties for attaining an eco-friendly/biodegradable/low cost/high performance functionalized new materials for various potential applications such as reinforcement materials, bionanocomposites, hydrogels, green composites as well as functionalized textile materials (Holme, 2007; Chang & Zhang, 2011; Siqueria, Bras,

& Dufresne, 2010; Thakur, Thakur, & Gupta, 2014a; Thakur, Thakur, & Gupta, 2014b; Thakur, Thakur, Gupta, & Kumar, 2014c; Thakur, Thakur, & Gupta, 2013; Zimniewska, Wladyka-Przybylak, & Mankowski, 2011).

On the other hand, The increase in global population, in natural and/or synthetic fibres consumption as well as in global competition in apparel, interior, home and technical textiles, taking in consideration the environmental, economic and quality dimensions, has intensified R&D efforts and generated many opportunities for utilization of emerging technologies and application of innovative functional finishes for developing and producing innovative/durable/cost-effective textile products (Holme, 2007; Lam, Kan, & Yuen, 2012; Powel & Cassill, 2006; Sawhney et al., 2008).

Lots of work have been done in functionalization of natural fibres, based on cellulose and/or protein, has received a great attention to impart new properties such as easy care, softness, antibacterial, UV-protection, self-cleaning, and/or water/oil repellency to treated textiles to cope with the changing lifestyles, to provide textile-users with highest levels of comfort, protection, safety and aesthetics and to compete in the tough global market place (Bajaj, 2002; Gouda & Ibrahim, 2008; Hashem & Ibrahim, 2008; Ibrahim, Gouda, & Zairy, 2008a; Ibrahim, Abo-Shosha, Fahmy, El-Sayed, & Hebeish, 2008b; Ibrahim, Amr, Eid, & El-sayed, 2010a;

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Ibrahim, Refai, & Ahmed, 2010b; Ibrahim, Khalifa, El-Hossamy, & Tawfik, 2010c; Ibrahim, Amr, Eid, Mohammed, & Fahmy, 2012a; Ibrahim, Eid, & El-Batal, 2012b; Ibrahim, Gouda, Hussein Sh, El-Gamm, & Mahrous, 2009; Ibrahim et al., 2013a; Liu, Wang, & Fan, 2012; Perumalraj, 2012; Radetic, 2013; Sivakumar, Murugan, & Sundaresan, 2012).

However a very limited number of publications are available on functional finishing, and simultaneous functionalization and coloration of cellulose/wool blended fabrics (Chikkodi, Khan, & Mehta, 1995; Haggag & Ibrahim, 1991; Harper & Mehta, 1985; Ibrahim et al., 2008a; Ibrahim, Khalil, El-Zairy, & Abdalla, 2013b; Ibrahim, El-Zairy, Abdalla, & Khalil, 2013c; Mehta, 1973).

Accordingly, the present research work is directed towards searching for proper finishing formulations for functionalization of cotton or viscose/wool blends to impart multifunctional properties to the finished fabrics and to extend their potential applications.

2. Experimental

2.1. Materials

Mill-scoured and bleached cotton/wool (50/50, 230 g/m²) and viscose/wool (50/50, 190 g/m²) blended fabrics were used.

Arkofix[®] NDF liq. C (low content of free formaldehyde, based on modified N-methyloldihydroxyethylene urea, DMDHEU, Clariant), Tinosan[®] CEI (encapsulated organic antimicrobial agent based on triclosan, Ciba), GB stain[®] Repellent FC (hydrophobic, oleophobic and dirt-repellent fluorocarbon compound, BASF), Solusoft[®] TOW Liquid C (hydrophobic silicone nano emulsion, weakly cationic, Clariant), Silastol[®] NSI (nonionic silicon elastomer, Schill & Seilacher), Precosoft[®] SM 40 (nonionic softener based on amino-polysiloxane micro emulsion, Schill & Seilacher), Zylon[®] NFL (nonionic softener, based on fatty acid glycol, Rossarl), Printofix[®] Binder MTB01 EG (APEO-free binder based on acrylate based copolymer, Egcodar), Pigment Red KBN 11009, Pigment Yellow M2GPR12001, Pigment Navy 8309, as well as Hostopal[®] CVL-ET (nonionic wetting agent based on alkyl aryl polyglycol ether, Clariant) were of commercial grade.

Magnesium chloride hexahydrate (MgCl₂·6H₂O), citric acid and 4-hydroxybenzophenone (4-HBP) were of reagent grade (Aldrich).

2.2. Methods

2.2.1. Easy care/soft-finish general procedure

The blended fabric samples were padded twice to wet pick up of 80% with an aqueous finishing formulation containing DMDHEU (50 g/L) as crosslinker, (MgCl₂·6H₂O)/citric acid (8/2 g/L), as a mixed catalyst, in the absence and presence of the softening agent (0–30 g/L) along with 2 g/L nonionic wetting agent, followed by drying and microwave fixation at output of 1300 W for 4 min.

2.2.2. Easy care/soft/antibacterial finish general procedure

To impart multifunctional properties to the nominated substrates, the blended fabric samples were padded twice in a single bath containing: DMDHEU (50 g/L), (MgCl₂·6H₂O)/citric acid (8/2 g/L), softener (20 g/L), antibacterial agent, Tinosan[®] CEL (0–30 g/L) along with a nonionic wetting agent (2 g/L), to give wet pick up of 80%, followed by microwave fixation as mentioned before.

2.2.3. Easy care/soft/water/oil repellency finish general procedure

Blended fabric samples were padded twice in a multifunctional formulation containing: DMDHEU (50 g/L), (MgCl₂·6H₂O)/citric acid (8/2 g/L), softener (20 g/L), and water/oil repellent agent

(0–150 g/L) to a wet pick up 80%, followed by microwave fixation as mentioned in Section 2.2.1.

2.2.4. Easy care/soft/UV-protection finish general procedure

Cotton/wool and viscose/wool blended fabric samples were padded twice in a single-bath containing: DMDHEU (50 g/L), (MgCl₂·6H₂O)/citric acid (8/2 g/L), softener (20 g/L), UV-absorbing agent (0–10 g/L) along with a nonionic wetting agent (2 g/L) to a wet pick up 80%, followed by microwave fixation as given in Section 2.2.1.

2.2.5. Multifunctional finishing/pigment dyeing in one step general procedure

Blended fabric samples were padded twice, to a wet pick up of 80%, in a finishing/dyeing formulation including: DMDHEU (50 g/L), (MgCl₂·6H₂O)/citric acid (8/2 g/L), softener (20 g/L), binding agent (100 g/L), pigment colorant (20 g/L) and a non-ionic wetting agent (2 g/L), followed by microwave fixation at 1300 W/4 min. The coloured/finished specimens were then washed at 50 °C/15 min, thoroughly rinsed in tap water at 25 °C, and then dried in ambient air.

2.2.6. Measurements

The nitrogen content, expressed as %N, was determined according to Kjeldhal method.

Colour strength of the obtained dyeings, expressed as K/S values, was calculated from reflectance data using Kubelka–Munk equation (Judd & Wyszeck, 1975):

$$\frac{K}{S} = \frac{(1 - R)^2}{2R}$$

where K and S are the absorption and scattering coefficient respectively, and R is the reflectance at wavelength of maximum absorbance of the used pigment colorants.

Fastness properties to washing, rubbing and light were evaluated according to AATCC test methods: (61-1972), (8-1972), and (16A-1972), respectively.

Surface roughness, arithmetic average roughness (Ra), of the treated and unfinished fabric samples was evaluated according to JIS B 0031-1994 Standard, analyzed in an automated roughness tester (Surfcorder SE 1700; Kosaka Laboratory Ltd, Kosaka, Japan). When the roughness curve is represented by $y = f(x)$, Ra is the value in microns (μm) found from the formula:

$$Ra = \frac{1}{l} \int_0^l |f(x)| dx$$

where l: reference length.

Dry wrinkle recovery angle (WRA) was evaluated according to ASTM Method D-1296-98.

Antibacterial efficacy of functionalized fabric samples against G +ve (*S. aureus*) and G –ve (*Escherichia coli*) bacteria was assessed using agar diffusion test according to AATCC test method 147-1988.

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

Water repellency (WRR) and oil repellency rating (ORR) were performed using the spray test method 22-1989 and AATCC test method 118-2007.

The durability to wash was evaluated after 15 laundering cycles according to AATCC test method 135-2000.

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

3. Results and discussion

Since the main task of the present study is to search for a proper single multifunctional finish stage for imparting specific functional properties in addition to easy care and softness to cotton/wool and viscose/wool blended fabrics, a wide range of finishing formulations and combinations have been used.

3.1. Bifunctional easy care/soft hand finish

As far as the changes in the nitrogen content (%N), the softness (SR), as well as the fabric resiliency (WRA) as a function of application of Solusoft® TOW silicone nano emulsion, along with the reactant resin, Arkofix® NDF, and MgCl_2 /citric acid, as a mixed catalyst, in a single bath, Fig. 1(a–c) shows that (i) increasing the silicone softener concentration up to 20 g/L results in an improvement in the %N of the finished substrate (Fig. 1a), an enhancements in the softness, expressed as the decrease in SR values (Fig. 1b), as well as a remarkable increase in the fabric resiliency, expressed as WRA values (Fig. 1c), (ii) the improvement in the above mentioned properties is a direct consequence of enhancing the extent of fixation of the silicone softener into/onto the resin/fabric matrix as well as within the finished fabric structure thereby lowering the friction coefficient of the cotton cellulose surface as well as minimizing the clearance between the wool scaly, and thus guarantees an ideal soft handle, better surface smoothness as well as imparts anti-wrinkle properties to the treated fabric blends (Hashem, Ibrahim, El-Shafei, Refai, & Hauser, 2009; Ibrahim, Amr, Eid, Almetwally, & Mourad, 2013d; Zia et al., 2011), (iii) the extent of improvement in the imparted easy care and soft handle properties is governed by the type of substrate, i.e. its fabric structure, available active sites, location and distribution of both the finishing and softening agent, extent of fixation of the active ingredients onto and/or within the fabric structure, as well as it's the physico-chemical and mechanical properties (Ibrahim et al., 2013b), (iv) the enhancement in %N, softness as well as anti-wrinkle properties of the finished cellulosic/wool blends follows

the descending order viscose/wool < cotton/wool, keeping other parameters constant, (v) further increase in the softener concentration, beyond 20 g/L, is accompanied by a slight improvement in the above mentioned properties, also (vi) it could be noticed that the optimum softener concentration is 20 g/L as demonstrated in Fig. 1(a–c).

On the other hand, Table 1 demonstrates the effect of using different softening agents along with other active ingredients in the finishing formulation on the %N, SR and WRA values of easy care/soft-finished fabric blends. The data show the following features: (i) incorporation of the nominated softening agents individually into the easy care finishing bath brings about an improvement in %N, a decrease in surface roughness along with an enhancement in fabric resiliency, regardless of the treated substrate, (ii) the extents of improvement in these properties is determined by type of substrate, i.e. viscose/wool < cotton/wool, keeping other parameters fixed, (iii) the effectiveness of the softening agent in upgrading the imparted functional properties, easy care/softness, depends highly on: its chemical structure, active ingredients, functional groups, attached chain length, nature of emulsifier, hydrophilic–lipophilic balance of emulsifier, droplet size, film forming ability and morphology, particle sizes of silicone, e.g. macro, semi-micro, micro, nano etc., compatibility with other finishing ingredients, location/extent of distribution and fixation onto and/or within the fabric structure as well as stability under the dry microwave fixation condition, in addition to the mode of interaction (Chattopadhyay and Vyas, 2010; Choudhury, Chatterjee Saha, & Shaw, 2012; Haberer & Bereck, 2002; Zia et al., 2011; Zuber et al., 2011), (iv) the enhancement in the imparted functional properties, easy care/softness, follows the decreasing order silicone-based softeners < fatty acid glycol-based softener, (v) the improvement in softness as well as fabric resiliency reflects the better lubricating action of silicone-based softeners between the fibres in the yarn as well as between the yarns of the fabric which results in imparting better softness and crease recovery angle to the finished substrates compared with the fatty acid glycol-based one, and (vi) loading of proper silicone softener onto

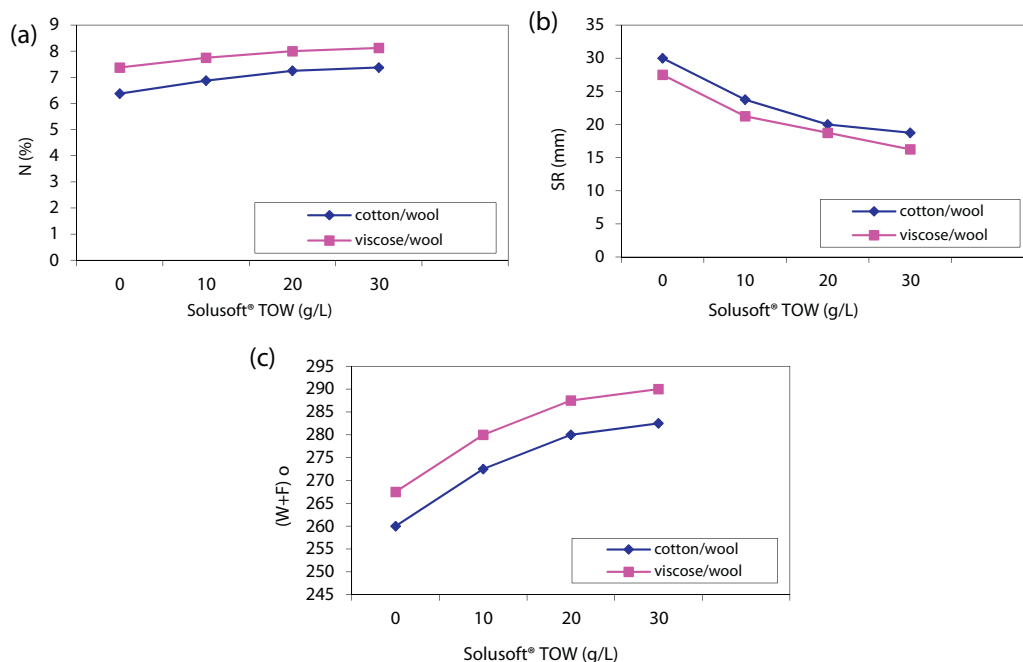


Fig. 1. Effect of Solusoft® TOW concentration on the nitrogen content, %N, (a), surface roughness, SR, (b) and anti crease, WRA, (c) properties of the finished cellulosic/wool blends. Finishing formulation: Arkofix® NDF (50 g/L); $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ (8 g/L); citric acid (2 g/L); nonionic wetting agent (2 g/L); Solusoft® TOW (0–30 g/L). Microwave drying/curing at 1300 W/4 min. N (%): nitrogen content; SR: surface roughness; WRA (W + F)°: Wrinkle recovery angle (warp + weft).

Table 1

Effect of incorporation of different softeners into the easy care finishing formulation on some performance properties of the finished cellulose/wool blends.

Softener (20 g/L)	Substrate	N ^a (%)	SR ^b (mm)	WRA ^c (W + F) ^o
None	C/W	6.41	30.0	260
	V/W	7.35	28.0	268
Silastol® NSI	C/W	6.88	19.6	285
	V/W	7.79	17.2	292
Precosoft® SM	C/W	7.40	18.6	290
	V/W	8.14	17.0	295
Solusoft® TOW	C/W	7.22	20.0	280
	V/W	8.05	17.5	288
Zylon® NFL	C/W	6.55	22.4	270
	V/W	7.42	19.5	276
Untreated	C/W	5.28	28.8	200
	V/W	6.18	25.4	215

Finishing formulation: Arkofix® NDF (50 g/L); softener (20 g/L); MgCl₂·6H₂O (8 g/L). Citric acid (2 g/L); nonionic wetting agent (2 g/L). Microwave drying/curing at 1300 W/4 min. C/W: cotton/wool, V/W: viscose/wool.

^a N (%): nitrogen content.

^b SR: surface roughness.

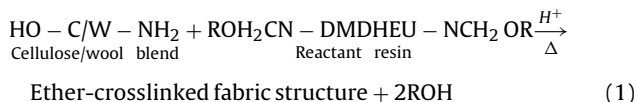
^c WRA: wrinkle recovery angle.

and/or within the crosslinked fabric structure enhances its fabric resiliency as well as minimizes the harsh-feeling of resin finish.

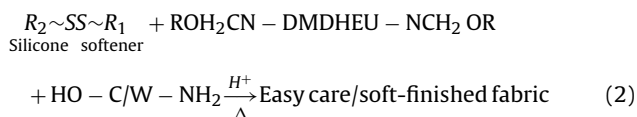
3.2. Tentative mechanism

Treatment of cotton/wool (C/W) and viscose/wool (V/W) blended fabrics with DMDHEU, as a reactant resin, amino-functional softener, as a softening agent, and MgCl₂·6H₂O/citric acid, as a mixed catalyst, followed by microwave fixation would be expected to promote the following interactions.

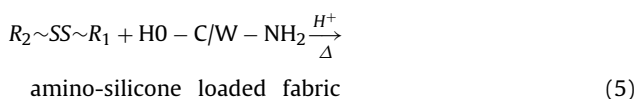
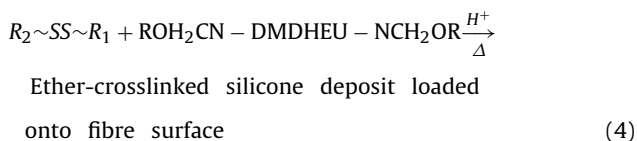
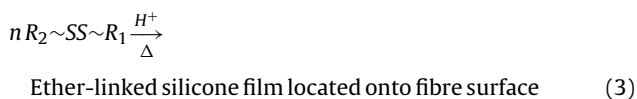
(i) Ether-crosslinking of fabric structure:



(ii) Fixation of silicone softener onto and/or within the fabric:



(iii) Side interactions:

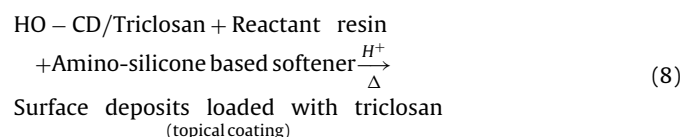
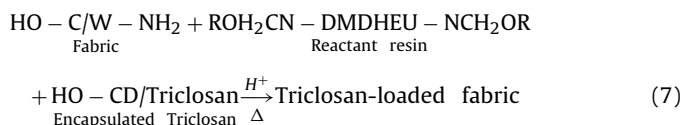
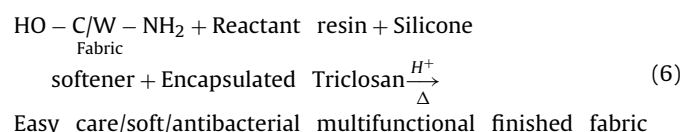


where R₁ = functional group, e.g. amino ethyl, amino propyl, quaternary, etc. R₂ = reactive group = methoxy, ethoxy, OH, etc. (Tung & Daoud, 2009; Zia et al., 2011; Zuber et al., 2011).

Based on the suggested tentative mechanism, we can say that fixation of amino-functional silicone softener onto and/or within the modified fabric structure [Eqs. (2), (3) and (5)] via covalent ether bonds, ionic interaction between the protonated amino group in the softener and the negatively charged active sites in the substrate, hydrogen bond and/or Van der Waals forces results in a formation of semi-inter and/or intra-penetrated network along with the positive role of the finishing agent in formation of crosslinks between adjacent cellulose chains thereby enhancing the extent of crosslinking and fixation of the softening agent and guaranteeing very high easy care and softness functional properties (Hashem & Ibrahim, 2008; Choudhury et al., 2012; Ibrahim et al., 2010a; Zia et al., 2011).

3.3. Easy care/soft hand/antibacterial multifunctional finish

The results in Table 2 show that treatment of the blended substrates with the reactant resin (50 g/L), softening agent (20 g/L) along with Tinosan®CEL (0–30 g/L), using MgCl₂·6H₂O/citric acid (8/2 g/L) as a catalyst followed by drying/microwave fixation (1300 W/4 min) resulted in a slight decrease in %N, and WRA along with a slight increase in the SR values of the finished substrates, as a direct consequence of loading the antibacterial agent onto and/or within the finish/soft/fabric matrix thereby decreasing the extent of crosslinking, expressed as WRA and %N, as well as minimizing the softness of the finished substrates, according to the following reactions (Ibrahim, Hashem, El-Sayed, El-Husseiny, & Elanay, 2010d; Ibrahim et al., 2013b; Jones, Jampani, Newman, & Lee, 2000; Simoncic & Tomsic, 2010).



On the other hand, increasing the antibacterial agent concentration up to 30 g/L in the finishing formulation resulted in a

Table 2

Effect of Tinosan® CEL concentration on %N, easy care, softness, and antibacterial properties of the multi-finished cellulose/wool blends.

Tinosan® CEL (g/L)	Substrate	Silastol® NSI					Precosoft® SM					Solusoft® TOW					Zylon® NFL				
		^a N (%)	^b WRA (W+F) ^o	^c SR (mm)	^d ZI (mm)		^a N (%)	^b WRA (W+F) ^o	^c SR (mm)	^d ZI (mm)		^a N (%)	^b WRA (W+F) ^o	^c SR (mm)	^d ZI (mm)		^a N (%)	^b WRA (W+F) ^o	^c SR (mm)	^d ZI (mm)	
					G +ve	G –ve				G +ve	G –ve				G +ve	G –ve				G +ve	G –ve
0	C/W	6.88	285	19.6	0	0	7.40	290	18.6	0	0	7.22	280	20.0	0	0	6.55	270	22.4	0	0
	V/W	7.79	292	17.2	0	0	8.14	296	17.0	0	0	8.05	288	17.5	0	0	7.42	276	19.5	0	0
15	C/W	6.66	280	20.0	12	10	7.25	285	19.4	17	15	7.04	272	20.8	14.5	13	6.39	264	23.0	8	6
	V/W	7.66	285	18.0	15	13.5	8.03	290	17.5	19	17	7.89	280	18.8	17	15	7.30	270	20.3	12	10
30	C/W	6.49	275	21.0	17.5	16	7.02	280	20.5	21	19	6.85	265	21.6	19	18	6.22	255	24.0	15	12
	V/W	7.48	280	19.0	20	18	7.82	285	18.5	23	20.0	7.60	271	19.9	22	19	7.15	265	21.2	18	16

Finishing formulation: Arkofix® NDF (50 g/L); softener (20 g/L); Tinosan® CEL (0–20 g/L); MgCl₂·6H₂O (8 g/L); citric acid (2 g/L); nonionic wetting agent (2 g/L). Microwave drying/curing at 1300 W/4 min.^a N (%): nitrogen content.^b WRA: wrinkle recovery angle.^c SR: surface roughness.^d ZI: zone of inhibition; G +ve: *S. aureus*; G –ve: *E. coli*.**Table 3**

Effect of 4-hydroxybenzophenone concentration on %N, easy care, softeners, anti-UV and antibacterial properties of the multi-finished cellulose/wool blends.

4-HBP (g/L)	Substrate	Silastol® NSI						Precosoft® SM						Solusoft® TOW						Zylon® NFL					
		^a N (%)	^b WRA (W+F) O	^c SR (mm)	^d UPF	^e ZI (mm)		^a N (%)	^b WRA (W+F) O	^c SR (mm)	^d UPF	^e ZI (mm)		^a N (%)	^b WRA (W+F) O	^c SR (mm)	^d UPF	^e ZI (mm)		^a N (%)	^b WRA (W+F) O	^c SR (mm)	^d UPF	^e ZI (mm)	
						G +ve	G –ve					G +ve	G –ve					G +ve	G –ve					G +ve	G –ve
0	C/W	6.88	285	19.6	28	0	0	7.40	290	18.6	29	0	0	7.22	280	20.0	26	0	0	6.55	270	22.4	19	0	0
	V/W	7.79	292	17.2	20	0	0	8.14	296	17.0	22	0	0	8.05	288	17.5	18	0	0	7.42	276	19.5	14	0	0
5	C/W	6.76	281	19.9	62	10.5	8.5	7.28	286	19.0	72	14.5	13.5	7.10	270	20.5	67	13	10	6.43	263	22.9	63	14.5	13.5
	V/W	7.68	285	17.5	51	12	10	8.03	299	17.3	58	17	15	7.93	280	18.0	53	16	14	7.31	270	20.0	50	17	15
10	C/W	6.69	274	20.2	77	19	17	7.05	280	19.4	80	19	17	7.00	262	21.0	74	18.5	16.5	6.30	255	23.2	70	19	17
	V/W	7.56	280	18.0	66	17	15	7.86	285	17.5	70	22	18	7.81	271	18.6	63	21.0	19	7.18	261	20.4	59	22	18

Finishing formulation: Arkofix® NDF (50 g/L); softener (20 g/L); 4-hydroxybenzophenone (0–10 g/L); MgCl₂·6H₂O (8 g/L); citric acid (2 g/L); nonionic wetting agent (2 g/L). Microwave drying/curing at 1300 W/4 min.

UPF of untreated C/W and V/W fabrics are 19 and 11 respectively

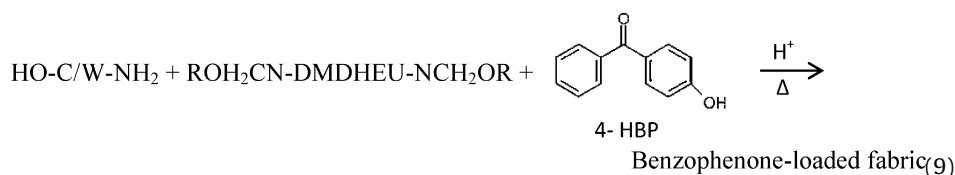
^a N (%): nitrogen content.^b WRA: wrinkle recovery angle.^c SR: surface roughness.^d UPF: UV-protection.^e ZI: zone of inhibition; G +ve: *S. aureus*; G –ve: *E. coli*.

remarkable improvement in the antibacterial activity of the multifunctionalized finished fabrics. The significant improvement in the antibacterial activity of triclosan-loaded fabric samples reflects the ability of the released triclosan to diffuse into the bacterial cell wall, disrupt the cytoplasmic membrane, RNA (ribonucleic acid), as well as inhibit fatty acid biosynthesis, which results in inhibiting or killing the bacteria (Ibrahim et al., 2013b; Jones et al., 2000; Orhan, Kut, & Gunesoglu, 2007). The multifunctionalized fabrics exhibit better antibacterial activity against G +ve than G –ve bacteria, reflecting their differences in their cell wall structure as well as their amenability to disruption (Ibrahim et al., 2013b; Orhan, Kut, & Gunesoglu, 2009). In addition, the extent of variation in the above-mentioned functional properties is governed by type of substrate, kind of softening agent, as well as the extent and mode of interactions among the finishing agent, softening agent, antibacterial agent and the substrate active sites under the given finishing conditions (Ibrahim et al., 2013b).

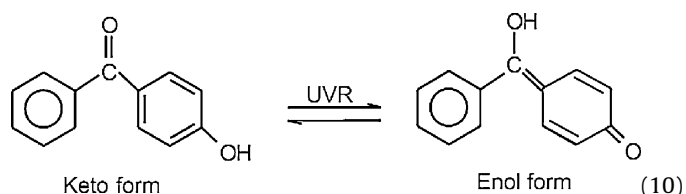
3.4. Easy care/soft hand/anti-UV/antibacterial multifunctional finish

As is evident, Table 3, increasing 4-hydroxybenzophenone (4-HBP) concentration in the multifinishing formulation up to 10 g/L results in (i) a marginal decrease in the %N, WRA as well as in SR values of the finished fabric samples, regardless of the used substrate and, (ii) a remarkable improvement in the UPF values along with a noticeable increase in the antibacterial activity. The slight decrease in fabric %N, resiliency as well as in softness most probably is attributed to side interactions among the reactant resin, softener, and the 4-HBP thereby decreasing the extent of crosslinking, expressed as %N and WRA, as well as formation of surface deposits, i.e. increased SR values.

The remarkable increase in both the UPF and ZI (zone of inhibition) values of the functionalized substrates is certainly attributed to the fixation and immobilization of the UV-absorber, 4-HBP, onto the finish/fabric matrix via ether-crosslinking as follows:



and/or via formation of ionic crosslinking net between the negatively charged 4-HBP and the positively charged amino silicone and/or the wool active sites, $-\text{NH}_2$, under the given acidic/thermal conditions (Hashem et al., 2009; Hong, Liu, & Sun, 2009; Ibrahim et al., 2013c). The upgrading of UV-protection capacity of the finished substrates reflects the higher degree of harmful-UV absorption, UV-B, as well as its ability to dissipate energy via subsequent change from keto to enol form (Hong & Sun, 2008; Ibrahim et al., 2013c) as follows:



On the other hand, the imparted antibacterial activity to the blended fabrics contain benzophenone chromophoric group is a direct consequence of generation of very active radicals capable of killing bacterial under UV-light (Hong & Sun, 2007, 2008).

Additionally, the data in Table 3 signify that: (i) the extent of improvement in the UPF value follow the decreasing order: cotton/wool < viscose/wool, regardless of the used softening agent, reflect their differences in fabric construction, weight, thickness, cover factor, porosity, extent of location and distribution of the loaded UV-absorber along with other finishing ingredients (Ibrahim et al., 2013c), (ii) the improvement in ZI values follows the descend order: viscose/wool < cotton/wool, reflecting the higher extent of loading the antibacterial agent onto viscose/wool blend as well as its better availability, accessibility and releasability compared with the finished cotton/wool blend, and (iii) the antibacterial activity of the multifinished substrates against the abovementioned G +ve and G –ve bacterial follows the decreasing order: G +ve < G –ve, most probably due to their differences in their cell wall structure along with their ability to offer protection against the generated and released active radicals as discussed earlier (Hong & Sun, 2007, 2008; Ibrahim, Aly, & Gouda, 2008c).

3.5. Easy care/soft hand/water & oily repellency multifunctional finish

As far as the changes in the abovementioned functional properties as a function of including the nominated water/oil repellent, GB stain® Repellent FC, into the finishing bath, the data in Table 4 signify that: (i) increasing the water/oil repellent concentration up to 150 g/L brings about a slight decrease in the %N, an improvement in fabric resiliency and softness, along with an outstanding improvement in both the water & oil repellency properties, (ii) the effectiveness of multifunctionalization depends not only on the extent of loading and orientation of the repellent agent but also on the chemical composition of the softening agent, mode of interactions among the crosslinking agent, softening agent, water/oil repellent agent and the substrate, as well as the physico-chemical and morphology structure of the treated blend, and (iii) better film formation and orientation of perfluorinated side chains provide fibre surface with lower surface energies and significantly decrease

their surface tension thereby achieving and imparting a remarkable water and oil repellency properties to multifinished blended fabrics (Bagherzadeh, Montazer, Latifi, Sheikhzadeh, & Sattari, 2007; Ibrahim et al., 2013d; Xu & Shyr, 2001).

Finishing of the nominated blends in the absence of the used water/oil repellent has no positive impact on their water/oil repellency properties.

3.6. Multifunctionalized pigment dyeings

Inspection of the results shown in Table 5 revealed that inclusion of pigment colorants along with the binding agent into dyeing/finishing formulation brings about a slight decrease in the %N, WRA and softness properties, along with an outstanding increase in the UPF values of the simultaneously dyed/multifunctionalized

Table 4
Effect of GB stain® Repellent FC concentration on %N, easy care, softness, oil and water repellent properties of the multi-finished cellulose/wool blends.

GB stain® Repellent (g/L)	Substrate	Silastol® NSI				Precosoft® SM				Solusoft® TOW				Zylon® NFL			
		^a N (%)	^b WRA (W + F) °	^c SR (mm)	^d ORR	^e WRR	^a N (%)	^b WRA (W + F) °	^c SR (mm)	^d ORR	^e WRR	^a N (%)	^b WRA (W + F) °	^c SR (mm)	^d ORR	^e WRR	
0	C/W	6.88	285	19.0	0	0	7.40	280	18.6	0	0	7.22	280	20.0	0	0	0
	V/W	7.79	292	17.2	0	0	8.14	296	17.0	0	0	8.05	288	17.5	0	0	0
50	C/W	6.71	296	18.0	7	80	7.30	290	16.2	7	80	7.06	285	18.5	6	70	5
	V/W	7.69	309	16.0	7	80	8.03	304	15.5	7	80	7.93	294	16.5	5	70	4
100	C/W	6.58	305	16.0	8	80	7.21	300	15.0	8	80	6.92	295	17.8	7	80	7
	V/W	7.52	318	15.0	8	80	7.90	312	14.3	8	80	7.80	300	15.4	7	80	7
150	C/W	6.49	310	15.0	8	80	7.10	306	13.5	8	80	6.80	300	16.5	8	80	8
	V/W	7.40	325	14.2	8	80	7.81	320	13.0	8	80	7.70	310	14.9	8	80	8

Finishing formulation: Arkofix® NDF (50 g/L); softener (20 g/L); water/oil repellent agent (0–15 g/L); MgCl₂ (8 g/L); citric acid (2 g/L); nonionic wetting agent (2 g/L); Microwave drying/curing at 1300 W/4 min.

^a N (%): nitrogen content.

^b WRA: wrinkle recovery angle.

^c SR: surface roughness.

^d ORR: oil repellent rating.

^e WRR: water repellent rating.

cellulosic/wool blended fabrics. The change in the abovementioned properties is governed by type of substrate, chemical composition of the softening agent, pigment colorant as well as extent of and mode of interactions among the different ingredients and the treated substrates. On the other hand, the change in the coloration properties, K/S and fastness properties, of the functionalized dyeings is governed by type of pigment, i.e. particle size, chemical composition, colour and hue, location and extent of accommodation and binding pigment particles, within the binder/crosslinker/softener network, and subsequent fixation onto the fabric surface during the microwave fixation step (Ibrahim et al., 2013b; Ibrahim, Eid, Abd El-Aziz, & Abou-Elmaaty, 2013e).

Table 5 also clearly demonstrates that the remarkable enhancement in the UPF values of the obtained functionalized pigment dyeing is determined by type of substrate, C/W < V/W, as well as ability of the used pigment colorants to block and/or absorb the harmful UV-B radiation (Ibrahim et al., 2013b).

3.7. Durability to wash

Table 6 shows the effect of laundering on the retention of imparted functional properties to selected fabric samples softened with Precosoft® SM 40. It is observed that: (i) even after 15 washing cycles, the imparted functional properties remained at a high level, (ii) laundering has a positive impact on fabric resiliency and softness especially in case of the functionalized pigment dyeings, most probably due to the removal of surface deposits, e.g. unfixed pigment particles, pigment/binder by products, etc., (iii) the differences in retained functional properties are governed by the finishing formulation constituents as well as extent of interaction and fixation of the active ingredients onto and/or within the multifunctionalized substrates, (iv) the durability of the imparted functional properties to 15 washing cycles is also determined by type of the finished substrate and its degree of functionalization, and (v) the remarkable washing durability demonstrated the proper fixation and loading of the nominated active ingredients onto/into the fabric structure.

3.8. SEM and EDX analysis

SEM images of the untreated cotton/wool and viscose/wool, Fig. 2A(a) and B(a) respectively, showed a clean surface for both the substrates. Compact and intact scales covered the wool fibre can be obviously seen while viscose fibre displayed clearly striation along its fibre length. Additionally the EDX spectra were also been given and the only peaks visible are carbon, oxygen, and nitrogen peaks.

Comparing the SEM images and EDX patterns of the control C/W, Fig. 2A(a and b), and V/W, Fig. 2B(a and b) blend fibres with softener treated samples Fig. 2A(c and d) and 2B(c and d) illustrated the presence of silicon element onto the outer surface of the treated substrates.

On the other hand the SEM and EDX spectra of pigment dyeing in presence of softener and/or water oil repellent Fig. 2A and B(e, f, g and h) showed that: (i) the fibres surface were covered by homogeneously distributed thin layer, (ii) the peaks of Si- and/or F- peaks were appeared in EDX patterns of the treated substrates. (iii) The presence and the extent of location of the nominated elements, i.e. silicone and/or fluorine onto the finished fabrics surface is governed by the type of substrate, follow the decreasing order: V/W > C/W, keeping other parameters constant, and confirm the existence of the silicone softener and/or water/oil repellent moieties on the modified fabric structure compared with the untreated ones.

Table 5
Multifunctional pigment dyeings.

Pigment (20 g/L)	Softener (20 g/L)	Substrate	^a N (%)	^b WRA (W + F) ^o	^c SR (mm)	^d K/S	^e WF		^f RF		^g LF	^h UPF
							Alt.	St.	Dry	Wet		
None	Silastol® NSI	C/W	6.88	285	19.6	–	–	–	–	–	–	28
		V/W	7.79	292	17.2	–	–	–	–	–	–	20
	Precosoft® SM	C/W	7.40	290	18.6	–	–	–	–	–	–	29
		V/W	8.14	296	17.0	–	–	–	–	–	–	22
	Solusoft® TOW	C/W	7.22	280	20.0	–	–	–	–	–	–	26
		V/W	8.05	288	17.5	–	–	–	–	–	–	18
Red	Zylon® NFL	C/W	6.55	270	22.4	–	–	–	–	–	–	19
		V/W	7.42	276	19.5	–	–	–	–	–	–	14
	Silastol® NSI	C/W	6.70	275	21.2	3.79	4–5	4–5	4–5	4	4–5	310
		V/W	7.62	280	18.5	2.97	4	4	4–5	4	4–5	120
	Precosoft® SM	C/W	7.22	280	20.8	4.32	4–5	4–5	4	3–4	4–5	328
		V/W	8.00	288	18.0	3.75	4–5	4–5	4	3–4	4–5	144
	Solusoft® TOW	C/W	7.01	270	22.5	3.99	4–5	4–5	4	3–4	4–5	520
		V/W	7.85	276	18.9	3.42	4–5	4	4	3–4	4–5	184
	Zylon® NFL	C/W	6.43	260	23.9	3.55	4	4	4	3–4	4–5	217
		V/W	7.29	269	21.0	2.62	4	4	4	3–4	4–5	110
	Silastol® NSI	C/W	6.37	265	22.9	3.96	4	4	3–4	3	4–5	219
		V/W	7.33	270	21.5	2.96	4	4	3–4	3	4–5	92
Yellow	Precosoft® SM	C/W	6.87	270	22.5	5.62	4–5	4–5	4–5	4	4–5	260
		V/W	7.63	276	21.0	3.99	4–5	4–5	4	3–4	4–5	124
	Solusoft® TOW	C/W	6.60	258	23.0	4.38	4–5	4	4	3–4	4–5	390
		V/W	7.54	262	22.8	3.29	4–5	4	4	3	4–5	135
	Zylon® NFL	C/W	6.03	239	25.0	3.70	4	4	4–5	4	4–5	175
		V/W	7.01	245	23.6	2.64	4	4	5	4	4–5	97
	Silastol® NSI	C/W	6.55	270	22.0	4.00	4	4	4	3–4	4–5	329
		V/W	7.42	275	20.5	3.04	4–5	4–5	4	3–4	4–5	166
	Precosoft® SM	C/W	7.01	275	21.5	5.67	4–5	4–5	4	3–4	4–5	375
		V/W	7.75	280	20.0	4.19	4–5	4–5	4	3–4	4–5	175
	Solusoft® TOW	C/W	6.82	265	24.0	4.15	4–5	4	4–5	4	4–5	604
		V/W	7.70	270	20.5	3.65	4–5	4	4–5	4	4–5	220
Navy	Zylon® NFL	C/W	6.21	248	25.5	3.69	4	4	4	3–4	4–5	260
		V/W	7.22	256	23.0	3.40	4	4	4	3–4	4–5	140

Pigment dyeing/functional finishing formulation: Arkofix® NDF (50 g/L); softener (20 g/L); Printofix® Binder (100 g/L); MgCl₂·6H₂O (8 g/L); citric acid (2 g/L); pigment colorant (20 g/L); nonionic wetting agent (2 g/L). Microwave drying/curing at 1300 W/4 min.

^a N (%): nitrogen content.

^b WRA: wrinkle recovery angle.

^c SR: surface roughness.

^d K/S: colour strength.

^e WF: wash fastness; Alt.: alteration of colour; St.: staining of white cotton.

^f RF: rubbing fastness.

^g LF: light fastness.

^h UPF: UV-protection factor.

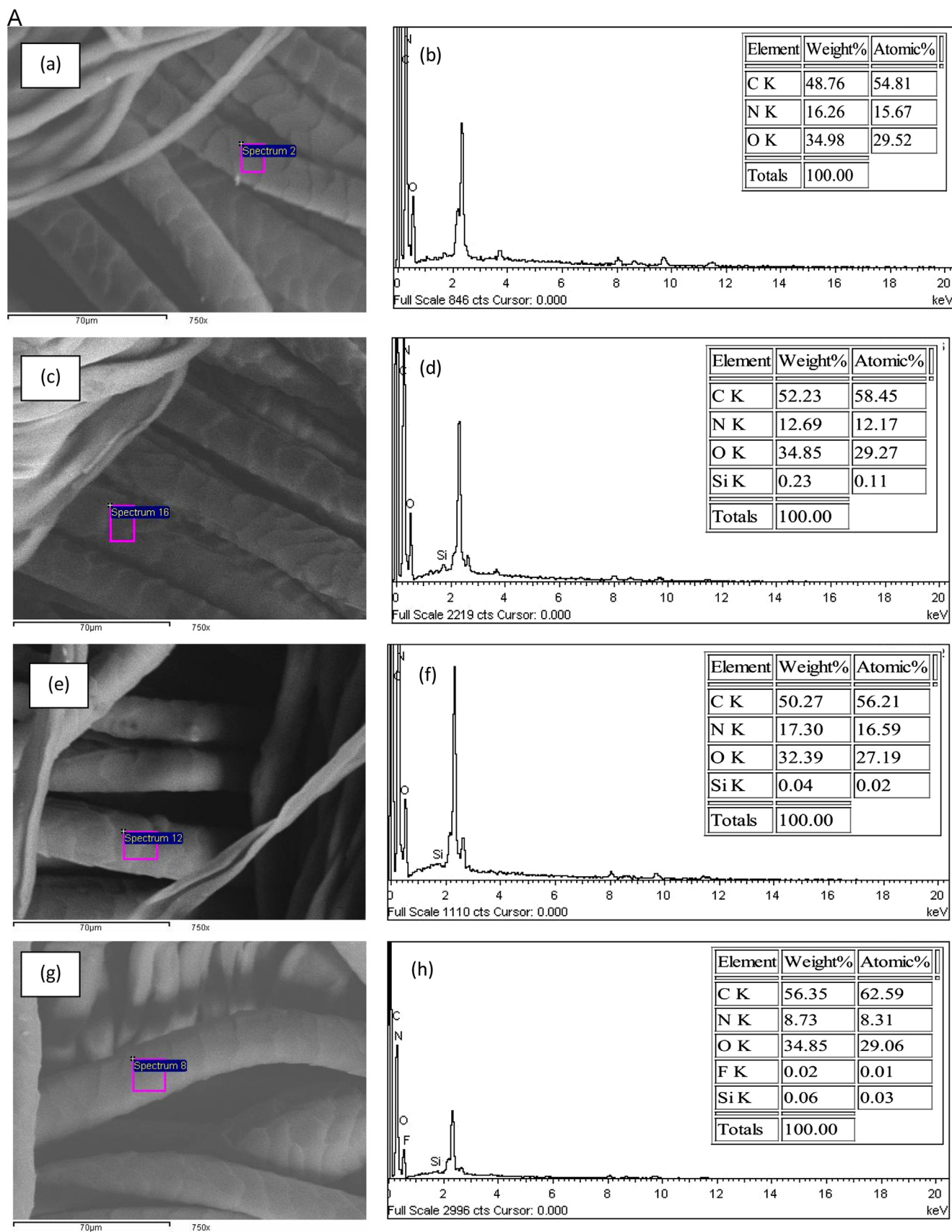


Fig. 2. (A) SEM and EDX spectra of: (a and b) untreated cotton/wool, (c and d) cotton/wool fabric treated with Precosoft®SM softener, (e and f) pigment dyeing cotton/wool fabric with Navy 8309 in presence of Precosoft®SM softener, (g and h) cotton/wool fabric treated with oil/water repellent and Precosoft®SM softener (B) SEM and EDX spectra of: (a and b) untreated viscose/wool (c and d) viscose/wool fabric treated with Precosoft®SM softener (e and f) pigment dyeing of viscose/wool fabric with Navy 8309 in presence of Precosoft®SM softener and (g and h) viscose/wool fabric treated with oil/water repellent and Precosoft®SM softener.

B

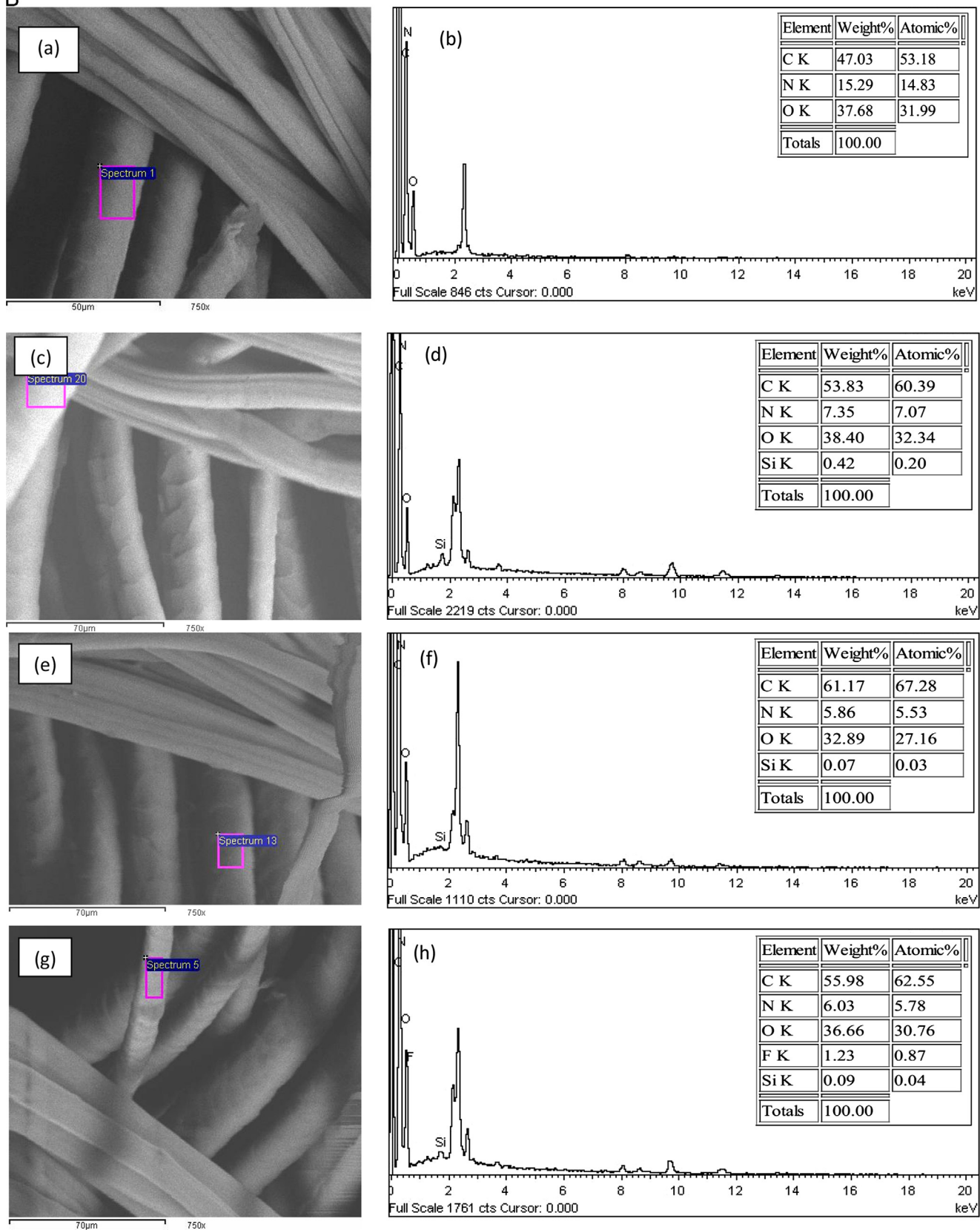


Fig. 2. (Continued)

Table 6
Durability to wash.

Finishing formulation	Substrate	Imparted functional properties							
		^a WRA (W + F) ^c	^b SR (mm)	^c UPF	^d ZI (mm)		^e ORR	^f WRR	^g K/S
					G +ve	G –ve			
[DMDHEU (50 g/L) + Precosoft® SM (20 g/L)] (I)	C/W	290 (278)	18.6 (20)	29 (20)	00 (00)	00 (00)	00 (00)	00 (00)	–
	V/W	296 (280)	17.0 (17.5)	22 (15)	00 (00)	00 (00)	00 (00)	00 (00)	–
(I) + Tinosan® CEL 30 g/L	C/W	280 (270)	20.5 (19)	–	21 (19)	19 (16)	–	–	–
	V/W	285 (272)	18.5 (17.5)	–	23 (20)	20 (17)	–	–	–
(I) + 4-HBP (10 g/L)	C/W	280 (265)	19.4 (18.5)	80 (68)	19 (17)	17 (13)	–	–	–
	V/W	285 (270)	17.5 (16.0)	70 (56)	22 (19)	18 (15)	–	–	–
(I) + GB Stain® Repellent (150 g/L)	C/W	306 (289)	13.5 (14.5)	–	–	–	8 (6)	80 (70)	–
	V/W	320 (305)	13.8 (15.0)	–	–	–	8 (6)	80 (70)	–
(I) + Pigment Red KBN (20 g/L)	C/W	280 (292)	20.8 (19.5)	328 (290)	–	–	–	–	4.33 (4.01)
	V/W	288 (296)	18.0 (17.0)	144 (125)	–	–	–	–	3.75 (3.22)
(I) + Pigment Yellow (20 g/L)	C/W	270 (260)	22.5 (21.0)	260 (230)	–	–	–	–	5.62 (5.25)
	V/W	276 (285)	21.0 (19.0)	124 (101)	–	–	–	–	3.99 (3.50)
(I) + Pigment Navy (20 g/L)	C/W	275 (285)	21.5 (20)	604 (570)	–	–	–	–	5.67 (5.31)
	V/W	280 (290)	20.0 (18)	220 (195)	–	–	–	–	4.19 (3.75)

Finishing formulation: Arkofix® NDF (50 g/L); Precosoft® SM (20 g/L); MgCl₂·6H₂O/citric acid (8/2 g/L); nonionic wetting agent (2 g/L); in the absence and the presence of the selected functional additives, wet pickup (80%), Microwave (fixation 1300 W/4 min).

^a WRA: wrinkle recovery angle.

^b SR: surface roughness.

^c UPF: UV-protection factor.

^d ZI: zone of inhibition; G +ve: *S. aureus*; G –ve: *E. coli*.

^e ORR: oil repellent rating.

^f WRR: water repellent rating.

^g K/S: colour strength.

Values between parentheses correspond to the functional properties after 15 washing cycles.

4. Conclusion

The interest in developing multifunctional textile products to cope with the changing lifestyles, as well as the highly demanded levels of protection, comfort and safety has recently attracted greater attention. In this study proper finishing formulations and combinations such as reactant resin together with silicone softeners, antibacterial agents, water and oil repellent, UV-absorber or pigment colorant were used for multifunctionalization of cotton/wool and viscose/wool blended fabrics using a pad-dry microwave fixation technique. The results obtained demonstrate that the imparted functional properties affected by type and concentration of functional ingredients in finishing formulation, type of substrate as well as extent of interaction and fixation of the active ingredients onto/within the fabric structure. The results of durability to wash confirmed the high retention of the imparted functional properties as a direct consequence of fixation and immobilization of active ingredients. The combination of the used active ingredients and reactant resin, under the given fixation conditions, can be used as a guideline by textile finishers for consistent production of innovative and durable cellulose/wool products taking in consideration materials and energy savings, environmental impacts as well as economical concerns.

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