Egyptian Journal of Chemistry

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Antibacterial Cellulosic Pigment Prints: Process Establishment and Characterization

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Abstract

Antibacterial textiles, a topic which is scientifically and economically essential, will be the direction for development of fabrics and clothing. In this study antibacterial cotton and viscose fabrics were produced by incorporating inorganic nanoparticles such as AgNPs, ZnONPs, and ZrO2NPs into solvent-free pigment printing pastes, followed by printing and microwave curing for 5 minutes at 386 W. Cotton and viscose fabrics were tested for pigment printability as well as antibacterial activity against Escherichia coli (E. coli) and Staphylococcus Aureus (St. Aureus). The suggested method was shown to be highly dependent not only on the nanoparticle concentration in the printing paste, but also on the kind of cellulosic substrate and the nanoparticles used. The obtained pigment prints were characterized by scanning electron microscope (SEM), and energy dispersive X-rays (EDX) to confirm deposition of specific nanoparticles on printed fabrics.

Keywords: Cellulosic fabric; Pigment prints; antimicrobial activity; inorganic-nano materials

1. Introduction

It is well known that, cellulose is one of the most widely used fiber due to its superior comfort properties and hygroscopic nature. Introduction of flexible cellulose materials such as micro fibrillated cellulose or cellulose nanofibers as functional membrane, supporting component or papers in medicinal, electronic and packaging applications, attracts people's great attention. For the nature of the existing of -OH group on cellulose surfaces, the simplicity of absorbing moisture or directly contacting with water during distribution, storage, and application is of a major challenge to mechanical endurances or antidegradability of these cellulose materials [1-4]. The pigment printing for textile is the most commonly and extensively used technique over the last 50 years, about 50% share, due to obvious advantages, such as ease of application, applicability to almost every kind of substrate, meet the demands of fastness properties, versatility as well as the ability to avoid washing steps after fixation. The main contents of a pigment printing paste are pigment dispersion, thickening agent, binding, fixing agent and some auxiliaries, e.g. softener, emulsifier, defoamer, etc. taking into account the ecological interest [5-12]

Additional functional properties like antibacterial activity were also usually preferred for preparing functional cellulose materials. However, when fabricating antimicrobial cellulose fibers, attention has been paid on the preparation and applications of the antimicrobial agents used, which represents a prominent characteristic and significant advantage of cellulose fibers [13-20].

Because of their undeniably unique properties, inorganic and metallic-based nano-structured materials have created a new interesting field for continuous investigations in all branches of sciences, nanomaterials can also be categorized into metal nanoparticles; metal oxides and nano-composites for textile functionalization. Incorporation of nanomaterials in textiles enables a smart functionalization of the natural fibers providing new features such as antibacterial activity, superhydrophobic, photo-degradation and self-cleaning [21-29].

Accordingly, the present study is aimed at searching for appropriate printing/ antibacterial formulations of cotton and viscose cellulosic fabrics,

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Receive Date: 21 March 2022, **Revise Date:** 14 April 2022, **Accept Date:** 19 April 2022, **First Publish Date:** 19 April 2022
DOI: 10.21608/EJCHEM.2022.127754.5702

using certain nanometal and nanometal oxides namely AgNPs, ZnONPs, and ZrO_2NPs, for attaining high antibacterial functional properties.

2. Experimental

2.1. Materials

Plain wave 100% mill-scoured and bleached cotton (120 g/m2) and viscose (110 g/m2) cellulosic fabrics were used throughout the study.

printofix® Binder MTB-01 liquid (acrylate based copolymer ,anionic clariant), Printofix Binder 86 (acrylate based copolymer dispersion, self-crosslinking of Egcodar, clariant), Alcoprint® PB-55 (aqueous dispersion of self-crosslinking butadiene copolymer,Ciba) and GBinder® FMD (based on polyacrylate,anionic,GB Chem ,BASF, Egypt) printofix® thickener 160 EG liquid(synthetic thickening agent based on ammonium polyacrylate ,clariant); GBresin® CPN (based on hydroxymethylated 4,5dihydroxyethylene urea , GB Chem ,BASF, Egypt); Durex® Silicone -1020 (based on modified polysiloxane microemulsion, Texchem, Egypt), Imperon® Royal Blue SP pigment (Dystar), Unisperse® Blue G pigment (Ciba), pritofix® Blue R2H pigment (clariant).

Zirconium (IV) oxide nanoparticles, (10 wt% dispersion in water, Aldrich), Zinc oxide, dispersion nanoparticles, <100 nm (pls) particle size <35 (50 wt% in water, Aldrich), nm avg. Silver dispersion, nanoparticles, 40 nm (TEM 0.02 mg/ml in aqueous buffer, Sigma, Aldrich).

All other chemicals used during this study were of laboratory reagent grade.

2.2. Methods

2.2.1. Printing paste preparation

The printing paste for solvent-free pigment printing and antibacterial finishing of cotton and viscose cellulosic fabrics was prepared according the following recipe (Table 1).

Table 1 Solvent free pigment printing and antibacterial finishing formulation

Printing paste components	g/Kg paste
Pigment	20
Thickener	20
Binder	100
Crosslinker	20
Softener	10
NH_4-persulfate	2
Nano material	20
Water	808
Total	1000

2.2.2. Printing procedure

Printing was carried out by the conventional flat screen printing technique.

2.2.3. Fixation

samples printed with the prepared printing paste were then simultaneously dried and thermofixed in a commercial microwave oven with oven at power of 386 W/5 mi n.

2.2.4. Washing

Washing process of the prints was carried out through three stages: 1) rinsing thoroughly with warm water, 2) soaping using 2g/l non-ionic detergent at 90-95 °C for 15 min., 3) and rinsing with cold water. The samples were dried and evaluated for color strength and all fastness properties.

2.3. Measurements

Color strength expressed as (K/S) values were determined from the reflectance measurements using the Kubelka Munk equation (Judd & Wyszeck, 1975):

$$K/S = (1-R)\ 2/2R,$$

Where K/S is the ratio of absorption and scattering coefficient, R is the reflectance at the wavelength of maximum absorbance of the used pigments.

Fastness properties to washing, rubbing and light of the obtained pigment prints were determined according to AATCC Test Methods (61-1972), (8-1972), and (16A-1972) respectively.

Antibacterial activity assessment against G+ve bacteria *(S. aureus)* and G−ve bacteria *(E. coli)* was tested qualitatively according to AATCC Test Method (147-1988) and expressed as zone of growth inhibition (mm).

Scanning electron microscope (SEM) images of nanoparticles loaded –fabric samples were obtained with a JEOL, JXL 840A electron probe microanalyser, equipped with energy disperse X-ray spectroscopy (EDX) for the composition analysis.

The metal content of the treated samples was quantitatively determined by using Flame Atomic Absorption Spectrophotometer GBC-Avanta, Australia.

3. Results and discussion

Factors affecting the imparted antibacterial and pigment printing properties of cotton and viscose fabrics brought about by individual inclusion of nanometal and nanometal oxides namely AgNPs, ZnONPs, and ZrO2NPs into the pigment paste formulation, using different pigment colorants, followed by screen printing and microwave curing were thoroughly investigated. Discussion of the obtained results follows.

3.1. Effect of nanoparticles type and concentration:

Different concentrations of the nominated nanoparticles were used in the printing paste, from zero up to 20 g/kg paste. It's clear that increasing the

nanoparticles followed by screen printing and microwave fixation at 386W for 5min showed considerable enhancement in the K/S values of the obtained cotton and viscose pigment prints. As shown in Fig.1: (i) increasing Ag-NPs, ZnO-NPs, and ZrO2-NPs concentration up to (20 g/kg) in the printing paste is clearly accompanied by an enhancement in the K/S values of the printed substrates, as follow: Ag-NPs (from 11.19 to 13.31) for cotton, (from 6.9 to 11.29) for viscose, ZnO-NPs (from 11.19 to 13.4) for cotton, (from 6.9 to 8.98) for viscose, ZrO_2-NPs (from 11.19 to 13.21) for cotton, (from 6.9 to 12.69) for viscose respectively (ii) The extent of pigment fixation is significantly improved during the microwave fixation step, this in agreement with the positive role of some nanometal oxides, i.e. ZnO-NPs, as a co-catalyst and/or via, (iii) the positive role of the formed metal chelates, through picking up and fixing of the metal cations by the -COOH groups of the used binder, in darkening the deepness of the obtained prints without negatively affect the hue [7, 11]

On the other hand, figure 2 shows the antibacterial properties of simultaneously pigment printed and functionally finished cellulosic fabric samples in the absence and presence of Ag-NPs, ZnO-NPs, or ZrO_2-NPs (20g/kg paste). As can be seen from the results, for a given set of printing paste formulation and printing conditions, the antibacterial activity of the printed samples was influenced by the presence of the nominated nano-materials. This can be explained by the fact that: (i) All in all, the inclusion of any of the nominated nano-materials in the printing paste enhanced the imparted antibacterial property for a wide range. (ii) the variation in the antibacterial activity is attributed to differences in chemical structure, size, shape, and size distribution of these nanoparticles. It also results from the differences in concentration, the extent of loading and fixation of nano-particles onto the binder/substrate matrix during the microwave fixation, availability and accessibility, mode of attack on microorganisms as well as compatibility with other agents in the printing paste [30-33].

The enhancement in the antibacterial activity of Ag-NPs loaded pigment prints is a direct consequence of: impair the cell bacterial membrane, causing permeability changes that followed by the leakage of intracellular components causing the death of bacteria [34-38]. While, the antibacterial activity of the loaded nano-metal oxides, i.e. ZnO-NPs and ZrO_2-NPs is a direct consequence of the generation and release of active oxygen species, e.g. hydroxyl radicals, superoxide anions, single oxygen, H_2O_2, that can damage DNA, proteins, and enzymes, leading to destruction of bacteria cell due to the photocatalytic effect [24, 39-42].

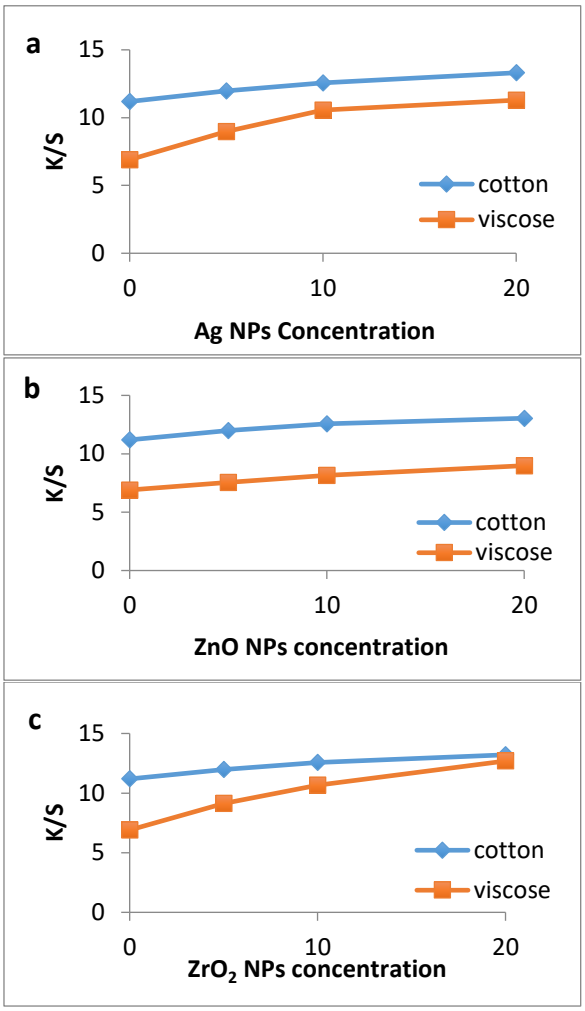

Fig.1: Effect of inclusion of nanoparticles into the pigment printing paste on K/S values of the obtained pigment prints.

Moreover, the antibacterial pigment prints showed better antibacterial activity against G+ve bacteria in comparison with G−ve one. This phenomenon is most probably due to the different cell wall structures of the two types of bacteria. Therefore, the high resistance of Gram-negative *(E. coli)* might be related to the low permeability of the outer cell membrane, thereby acting as a barrier to the antibacterial effect [14, 43].

Additionally, the differences in the imparted antibacterial properties of the cellulosic fabrics is determined by the type of cellulosic substrate, i.e. difference in fabric construction, surface area, pore structure, surface morphology, degree of polymerization, amorphous to crystalline regions, cellulose content and degree of subsequent fixation of the nominated nanoparticles [44].

3.2. *Effect of binding agent*

Table 2 showed the depth of shade, expressed as the K/S values, and fastness properties for the pigment printed fabrics using several types of binder with different composition in the formulation of the printing paste to search for the appropriate binder for pigment prints, where other parameters were fixed and in presence of the nominated nanomaterials.

From the data obtained it is observed that: (i) adding of any of the used binding agents in the printing paste results in a significant enhancement in the imparted printing properties, i.e. K/S and fastness ratings, and (ii) the increase in the above-mentioned properties especially K/S values is governed by the type of binding agent and follows the descending order: Alcoprint® PB-55 > GBinder FMD > Printofix® MTB> Printofix® Binder 86 > none.

The differences in the extent of enhancement in the tested fastness properties by using the aforementioned binders are attributed to the variation among them in chemical structure, e.g. butadiene copolymer or based on polyacrylate, reactive ingredients, film forming properties, compatibility with other agents in the printing paste, binder efficiency and capacity, extent of encapsulation of the pigment particles and entrapment in between the binder and the substrate surface as well as in the holding capacity of nanoparticles and extent of their loading onto the printed fabric surface [11, 45-47]. On the other hand, the fastness properties in case of using the binding agent are much better than in its absence reflects the positive role of using the binder to achieve better film-forming properties, facilitate the formation of a three-dimensional networks onto the substrate, that increase accommodation and entrapment of the pigment particles during the microwave fixation step [47, 48].

3.3. *Effect of Binder/pigment concentration*

The effect of different binder/pigment concentrations on the color strength (K/S), the imparted antibacterial activity (ZI), and the fastness properties of the obtained printed fabrics were studied, and the results of this study were tabulated in (Table 3). Within the range examined, the data of this table showed that increasing binder/pigment ratio from 75/10 up to 125/30g/kg is accompanied by a noticeable improvement in the extent of pigment fixation, expressed as K/S values, and Ag-NPs, ZnO-NPs, or ZrO_2-NPs immobilization thereby upgrading both the antibacterial and coloration properties of the obtained prints without affecting their fastness properties, and irrespective of the used substrate. The outstanding improvement in the aforementioned printing and antibacterial properties might be explained by the fact that using the appropriate binder concentration to achieve better film-forming properties, facilitate the formation of three-dimensional networks onto/with the substrate, to help accommodation and entrapment of the pigment particles, as well as to ensure loading of the proper amount of nanoparticles onto the obtained pigment prints [8, 37].

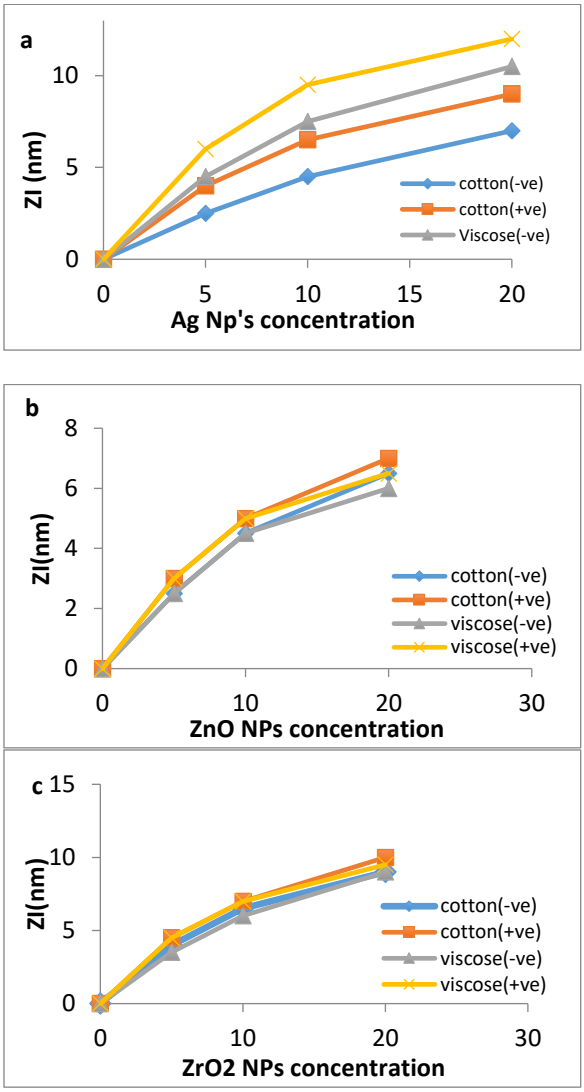

Figure 2: Effect of inclusion of nanoparticles into the pigment printing paste on ZI values of the obtained pigment prints.

Table 2. Effect of using different binding agents in the printing paste formulation on functionality and printability of the obtained cellulose pigment prints.

Binder (100 g/kg)	NPs	Substrate	K/S[a]	WF[c]		RF[d]	
				Staining	Alteration	Dry	Wet
printofix® MTB-01	Ag	Cotton	13.31	4	3	4-3	3-2
		Viscose	11.00	4-3	3	4	3
	ZnO	Cotton	12.13	3-2	3	4-3	3
		Viscose	10.34	3	3-2	3	2
	ZrO_2	Cotton	13.21	4-3	4	4-3	3
		Viscose	12.69	4	3	4-3	2
Printofix® 86	Ag	Cotton	11.56	4	3	4	3-2
		Viscose	10.73	4-3	3	4-3	2
	ZnO	Cotton	11.64	3	4-3	4	3-2
		Viscose	8.98	4-3	3-2	3	2
	ZrO_2	Cotton	11.77	4-3	4	4	3
		Viscose	10.89	4-3	3	4	2
Alcoprint® PB-55	Ag	Cotton	15.19	4	4	5-4	3-2
		Viscose	16.58	4-3	3	4	3-2
	ZnO	Cotton	13.25	4-3	4	5-4	3-2
		Viscose	13.58	3	3-2	4	2
	ZrO_2	Cotton	15.17	4	4	5-4	3
		Viscose	16.03	4-3	3	5-4	2
GBinder® FMD	Ag	Cotton	13.88	4-3	3	5-4	3-2
		Viscose	11.29	4-3	2	5-4	3-2
	ZnO	Cotton	13.04	3	4	4	3-2
		Viscose	9.19	3	3-2	4	2
	ZrO_2	Cotton	14.13	4-3	4	5-4	3
		Viscose	13.65	3	3	5-4	2

Printing paste components: Binder (100 g/kg); Printofix® Thickener 160 EG (20 g/kg); Imperon® Royal Blue SP (20 g/kg); GB Resin® (20 g/kg); silicone softener (10 g/kg); $(NH_4)_2S_2O_8$ (2g/kg); fixation at 386 W for 5min.

[a] Color strength.

[b] Wash fastness.

[c] Rubbing fastness.

Table 3. Effect of binder/pigment ratio on the chemical, physical, and functional properties of the obtained cellulose pigment prints.

Binder/ pigment conc. (g/kg)	NPs	Substrate	K/S[b]	Metal-content (%)	ZI[c] (mm)		WF[d]		RF[e]	
					G (-ve)	G (+ve)	Staining	Alteration	Dry	Wet
75/10	Ag	Cotton	8.80	0.2952	5.0	3.0	4	3	3	2
		Viscose	10.12	0.1936	7.0	9.0	4-3	2	3	2
	ZnO	Cotton	9.05	0.3737	3.5	4.0	3-2	3	3	3-2
		Viscose	7.21	0.3381	6.0	5.5	4-3	2	3-2	2
	ZrO_2	Cotton	7.40	0.3951	7.0	6.5	4	3	4-3	2
		Viscose	7.12	0.2731	8.0	8.5	4-3	2	3	2
100/20	Ag	Cotton	13.31	0.4972	7.0	9.0	3-2	3	4-3	3-2
		Viscose	11.29	0.2211	10.5	12.0	4-3	3	4	3
	ZnO	Cotton	13.04	0.3796	6.5	7.0	3-2	3	4-3	3
		Viscose	8.98	0.4389	6.0	6.5	3	3-2	3	2
	ZrO_2	Cotton	13.21	0.4531	9.0	10	4-3	4	4-3	3
		Viscose	12.69	0.3543	9.0	9.5	4	3	4-3	2
125/30	Ag	Cotton	16.26	0.5988	10.0	11.5	5-4	3	4-3	2
		Viscose	11.64	0.3168	11.0	13.5	4-3	3-2	4-3	2
	ZnO	Cotton	14.81	0.4389	8.5	9.5	3-2	3-2	3	3-2
		Viscose	10.12	0.5036	8.0	9.0	3-2	2	3	2
	ZrO_2	Cotton	15.40	0.5113	13.0	11.0	4-3	4	4-3	3-2
		Viscose	15.89	0.3972	12.0	11.0	3	3-2	3	2

Printing paste components: Binder (100 g/kg); Printofix® Thickener 160 EG (20 g/kg); Imperon® Royal Blue SP (20 g/kg); GB Resin® (20 g/kg); silicone softener (10 g/kg); $(NH4)_2S_2O_8$ (2g/kg); fixation at 386 W for 5min.

[a] Color strength.

[b] Zone of inhibition, G+ve (*S. aureus*); G−ve (*E. coli*).

[c] Wash fastness.

[d] Rubbing fastness.

3.4. Effect of Pigment colorant

The ability of nanoparticles to enhance both the antibacterial and coloration properties of cotton and viscose cellulosic fabrics using various pigment colorants along with other nominated printing paste constituents and microwave- fixation technique is demonstrated in Table 4.

The obtained data demonstrated the differences in the imparted antibacterial and printing properties upon using different pigment colorants, keeping other parameters fixed. This reflects that the chemical structure of the pigment color (average particle size, pigment content, chromophore, hue, extent of agglomeration, dispersion stability), compatibility with other ingredients, location and extent of fixation, deposition, antibacterial activity, UV- absorption capacity, and its covering power seems to play an important role in the printing of cotton and viscose fabrics [8, 49].

The results show the following common features: keeping other parameters constant, (i) addition of nanoparticles (20g/kg) to the printing paste constituents remarkably improve of the color deepness, fastness properties, and the imparted antibacterial properties, (ii) the enhancement of K/S values follows the descending order cotton prints > viscose prints, and (iii) the improvement in antibacterial activity follows the descending order viscose prints > cotton prints.

On the other hand, washing and rubbing fastness properties of the obtained pigment prints were good to very good. Moreover, the wet rubbing fastness were found to be slightly lower than the dry one, most probably due to the presence of unfixed pigment particles and /or entrapped in the printed substrate [5].

3.5. SEM images and EDX spectra

The SEM images demonstrated that pigment printed fabric surfaces were clearly covered with printing paste components, pigment particles along with the nominated nanoparticles. EDX micrographs of simultaneously pigment printed and antibacterial samples (Fig. 3 and 4) confirmed the deposition of the print paste together with the loading of nominated nanoparticles onto the fiber surface of the printed samples, Ag-NPs, ZnO-NPs, or ZrO_2-NPs (Fig. 3 (a, b), (c, d) and (e, f) respectively for cotton), and (Fig. 4 (a, b), (c,d) and (e, f) respectively for viscose). The existence of some elements such as carbon, oxygen and silicone were also approved, as a direct consequence of loading the nominated nanoparticles together with the silicone-based softener onto/within the produced three-dimensional network.

Table 4. Effect of using different pigments on the chemical, functional, and coloration properties of the produced cellulose pigment prints.

		printofix® Blue R2H pigment								*Imperon® Royal Blue SP pigment*							
NPs	Substrate	Metal content (%)	[a]K/S	ZI[b] (mm)		WF[c]		RF[d]		Metal content (%)	[a]K/S	ZI[b] (mm)		WF[c]		RF[d]	
				G (-ve)	G (+ve)	Staining	Alteration	Dry	Wet			G (-ve)	G (+ve)	Staining	Alteration	Dry	Wet
None	Cotton	0	9.54	0	0	3	4-3	3	2	0	11.19	0	0	4	4-3	4-3	3
	Viscose	0	9.24	0	0	2	2	3	2	0	6.90	0	0	4	4	4-3	3-2
Ag	Cotton	0.1663	9.12	6.5	12.0	4	4	4	3-2	0.1906	13.31	7.0	9.0	4	4-3	5-4	3
	Viscose	0.2618	8.36	12.5	17.5	3	2	4	2	0.1990	11.29	10.5	12.0	3	3-2	4-3	3
ZnO	Cotton	0.3849	8.76	8.5	9.0	3	4-3	4	4-3	0.4092	13.04	6.0	7.0	4	4-3	5-4	3
	Viscose	0.3525	6.72	12.0	14.5	3-2	3	4-3	2	0.3929	8.98	6.5	6.5	3	2	4	2
ZrO_2	Cotton	0.3542	10.58	13.0	15.0	4-3	4	5-4	4-3	0.4531	13.21	9.0	10.0	4-3	3	4	3-2
	Viscose	0.4641	9.50	20.0	21.0	3	4-3	4	3	0.3543	12.69	9.5	9.5	4-3	3	4-3	2

Printing paste components: Binder (100 g/kg); Printofix® Thickener 160 EG (20 g/kg); nanoparticles (20 g/kg); pigment (20 g/kg); GB Resin® (20 g/kg); silicone softener (10 g/kg); (NH4)2S2O8 (2g/kg); fixation at 386 W for 5min.

[a] Color strength.

[b] Zone of inhibition, G+ve (S. aureus); G−ve (E. coli).

[c] Wash fastness.

[d] Rubbing fastness

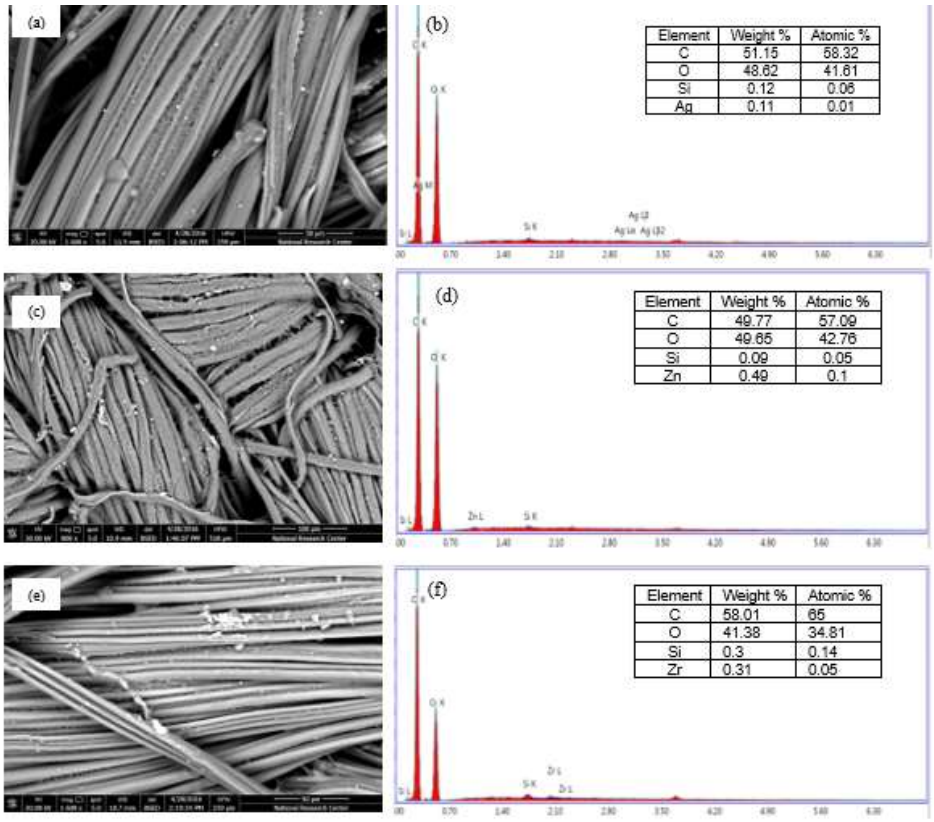

Figure 3: SEM Images and EDX spectrum of Ag-loaded pigment print (a,b); Zn-loaded pigment print (c,d) and Zr-loaded pigment print (e,f) cotton fabrics.

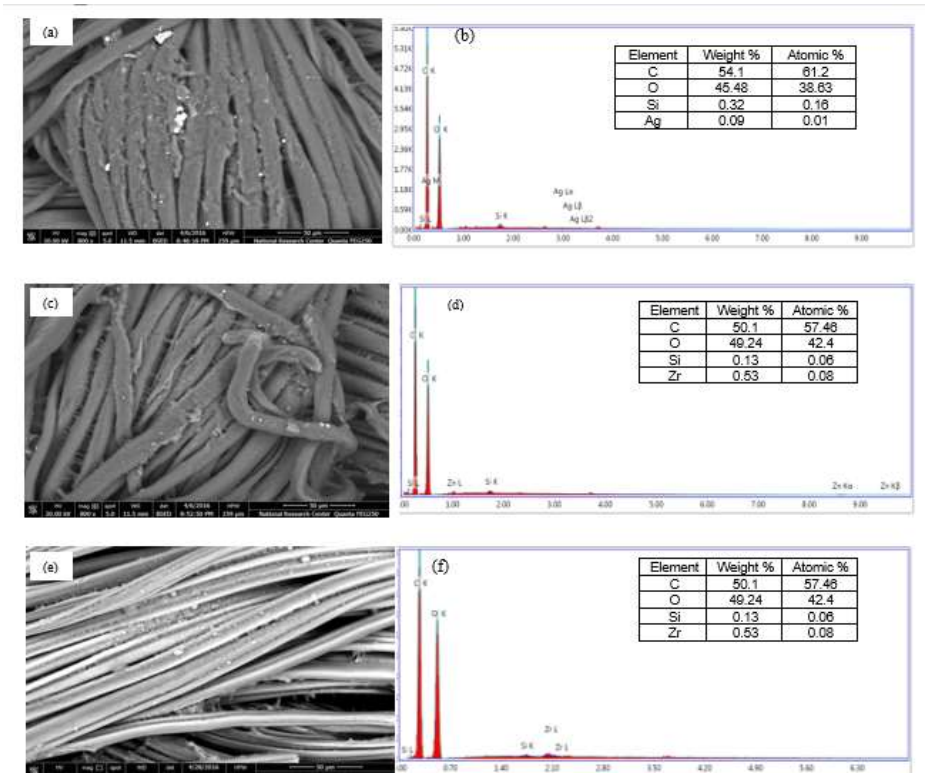

Figure 4: SEM Images and EDX spectrum of Ag-loaded pigment print (a,b); Zn-loaded pigment print (c,d) and Zr-loaded pigment print (e,f) viscose fabrics.

4. Conclusion

Antibacterial cotton and viscose pigment prints were achieved by individual including of nanomaterials namely silver nanometal and zirconium, zinc, nanometal oxide, in the print paste, followed by screen printing and pad-dry microwave fixation. The obtained results revealed that:

• Incorporation of nanomaterials namely silver nanometal and zirconium, zinc, nanometal oxide, (up to 20 g/kg) into the pigment printing paste results in functionalization of the obtained pigment prints.

• The enhancement in the imparted antibacterial properties is governed by the type of the nomainated nanoparticles, type of substrate, type, and concentration of binding agent/ pigment colorant as well as the extent of immobilization and location of nanoparticles along with degree of fixation of pigment particles onto/ within the binder film/fabric matrix.

• EDX spectra confirm the loading of Ag, ZnO, and ZrO_2 nanoparticles (NPs) onto the prints surface.

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