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Combined antimicrobial finishing and pigment printing of cotton/polyester blends

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ABSTRACT

This study demonstrates the possibility of enhancing the antibacterial functionality and pigment printing properties of cotton/polyester blends (50/50 and 35/65) in one step. Inclusion of chitosan (10 g/kg), choline chloride (15 g/kg), triclosan derivative (20 g/kg), hyperbranched poly amide-amine/silver or zinc oxide nanoparticles (HBPAA/Ag-NP's hybrid or HBPAA/ZnO-NP's hybrid – 20 g/kg) into a pigment print formulation followed by printing and microwave curing at 386 W for 5 min results in an improvement in antibacterial activity and pigment printability. It was further noted that, in all cases, the G+ve (*S. aureus*) bacteria is more susceptible to the action of the immobilized antibacterial agents than the G−ve bacteria (*E. coli*). The functionalized pigment prints exhibited very sufficient antibacterial activity even after 20 washing cycles. Modes of interactions were proposed, and surface modification was also confirmed by SEM and EDX analysis.

1. Introduction

Among the various functional finishes, high attention has given to the textile materials that are bioactive, to cope with the growing awareness of health and quality of hygiene, without adversely affecting their traditional characteristics using different antimicrobial agents. The available antimicrobial agents differ in their chemical structure, antibacterial activity, application method, mode of interaction as well as environmental impact (Gao & Cranston, 2008; Simoncic & Tomsic, 2010).

The most promising antimicrobial agents currently available for textile application include: inorganic compounds such as metal salts, nano-sized metals and metal oxides (Dastjerdi & Montazer, 2010; Gao & Cranston, 2008; Gowri, Almeida, Amorim, Carneiro, Souto, & Esteves, 2010; Ibrahim, Abo-Shosha, Gaffar, Elshafei, & Abdel-Fattah, 2006; Ibrahim, Mahrous, El-Gamal, Gouda, & Husseiny, 2010; Ibrahim, Eid, Hashem, Refai, & El-Hossamy, 2010; Ibrahim, El-Gamal, Gouda, & Mahrous, 2010; Ibrahim, Eid, & El-Batal, 2012; Ibrahim, Eid, Youssef, El-Sayed, & Salah, 2012; Ibrahim, Amr, Eid, Mohamed, & Fahmy, 2012; Ibrahim, Refaie, & Ahmed, 2010; Mahapatra & Karak, 2008) halogenated phenols (Hashem, Ibrahim, El-Sayed, El-Husseiny, & Elanany, 2009; Hashem, Ibrahim, El-Shafei, Refaie, & Hauser, 2009; Ibrahim, Hashem, El-Sayed, El-Husseiny, & Elanany, 2010; Orhan, Kut, & Gunesoglu, 2009), N-halamines (Gouda & Ibrahim, 2008; Ibrahim, Aly, & Gouda, 2008; Liu & Sun, 2006), chitosan (Lim & Hudson, 2003; Shin, Yoo, & Jang, 2001), Neem oil (Ibrahim, Eid, & El-Zariy, 2011; Joshi, Ali, & Rajendran, 2007), antibiotics (Ibrahim, Eid, et al., 2010), quaternary ammonium compounds (Gao & Cranston, 2008), and immobilized enzymes (Ibrahim, Gouda, El-Shafei, & Abdel-Fattah, 2007).

In recent years, the use of nanotechnology in textile field has increased rapidly to develop efficient, non-toxic, durable and cost effective functionalized textiles with multifunctional properties and increased potential applications (Dastjerdi & Montazer, 2010; Gowri, Almeida, Amorim, Carneiro, Souto, & Esteves, 2010; Hebeish & Ibrahim, 2007; Liu & Dong, 2002; Wong, Yuen, Leung, Ku, & Lam, 2006). The high durability of the imparted functional properties reflects the positive impacts of the large surface area along with the high surface energy of nanomaterials thereby ensuring better affinity for treated fabrics as well as higher durability of textile functions compared with the conventional materials (Becheri, Durr, Nostro, & Baglionl, 2008; Lee, Yeo, & Jeong, 2003).

On the other hand, it is well known that pigment printing is the most economical printing process and can be applied to all substrates. To attain an eco-friendly pigment prints, solvent-free thickening agents and low or zero-formaldehyde binding agents have been used along with other proper additives, e.g. crosslinker, softener, active ingredients, catalyst, etc. (Ibrahim, El-Zairy, Zaky, & Borham, 2005).

The main goal of the current research is to develop a new and effective printing method for enhancing the antibacterial activity

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of pigment printed cellulose-containing fabrics via incorporation of certain antibacterial agents such as chitosan, choline chloride, Invasan®, HBPAA/Ag-NP's and HBPAA/ZnO-NP's hybrids in the print paste formulations.

2. Experimental

2.1. Materials

Plain weave mill-scoured and bleached cotton/polyester (50/50, 118 g/m^2) and (35/65, 123 g/m^2) blends were used throughout the study.

Printofix® Binder MTB-01 liquid (acrylate based copolymer, anionic, Clariant); GB Binder®FMD (based on polyacrylate, anionic, BASF/GB Chem, Egypt) Printofix® Thickener 160 EG liquid (synthetic thickening agent based on ammonium polyacrylate, Clariant); GB Resin®CPN (based on hydroxymethylated 4,5-dihydroxyethylene urea, BASF/GB Chem, Egypt); Durex® Silicone-1020 (based on modified polysiloxane microemulsion, Texchem, Egypt); Printofix®Red H3BD pigment, Printofix®Green HXP (Clariant); Imperon® Royal Blue SP pigment (DyStar), Unisperse® Yellow MR pigment (Ciba), and Invasan® (Triclosan derivative, Huntsman, USA) were of commercial grade.

All of other chemicals namely, chitosan (degree of deacetylation of >85%, Sigma), Silver nitrate ($AgNO_3$, Sigma), zinc acetate ($Zn–(CH_3COO)_2{\cdot}2H_2O$, Aldrich); choline chloride (Aldrich), acetic acid, sodium hydroxide and ammonium persulphate [$(NH_4)_2S_2O_8$] were reagent grade.

2.2. Methods

2.2.1. Synthesis of the hyperbranched poly amide-amine (HBPAA)

HBPAA was synthesized using Ibrahim et al. method (Ibrahim, Mahrous, et al., 2010).

2.2.2. Synthesis of HBPAA/Ag-nanoparticles hybrid (HBPAA/Ag-NP's)

Synthesis of HBPAA/Ag-NP's hybrid was similar to that reported in the literature (Ibrahim, Eid, Youssef, et al., 2012).

2.2.3. Synthesis of HBPAA/ZnO-NP's hybrid

The HBPAA (3.1 g) was dissolved in distilled water (80 ml)/ethyl alcohol (20 ml) mixture at ambient temperature. To the solution the pre-dissolved 1.24 g Zn–acetate was added drop wise with continuous stirring. After 30 min of reaction the pre-dissolved 0.45 g NaOH was added drop wise with continuous stirring to the mother liquor and the reaction mixture was kept under stirring for further 30 min.

2.2.4. Pigment printing

Guide formulation for solvent-free pigment printing of cotton cellulose/polyester using flat screen technique with two Storkes follows:

Printing paste components	g/kg
Pigment colorant	20
Synthetic thickener agent	20
Binding agent	100
Crosslinker	20
Silicone softener	10
$(NH_4)_2S_2O_8$ catalyst	2
Bio-active agent:	
Chitosan	10
Choline chloride	20
HBPAA/ZnO-NP's hybrid	20
HBPAA/Ag-NP's hybrid	20
Or Invasan®	20
Acetic acid	1
Water	817 or 807
Total	1000

Printed fabric samples were then simultaneously dried and fixed in a commercial microwave oven at output power of 386 W/5 min.

2.3. Measurements

Nitrogen content (%N) was determined according to the Kjeldahl method.

The color strength (K/S) values of the obtained pigment prints were determined from the reflectance measurements using the Kubelka Munk equation (Judd & Wyszeck, 1975).

$K/S = (1R)^2/2R$, where K/S is the ratio of absorption and scattering coefficient, and R is the reflectance at the wave length of maximum absorbance of the used pigment colorants.

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

Antibacterial activity assessment against G+ve (*S. aureus*) and G−ve bacteria (*E. coli*) was evaluated qualitatively according to AATCC Test Method (147-1988), and expressed as zone of growth inhibition (mm). Durability of the imparted antibacterial activity to washing was evaluated according to ASTM Standard Test Method (D 737-96).

The morphology and particle size of the prepared HBPAA/Ag-NP's and HBPAA/ZnO-NP's hybrids were determined by transmission electron microscope (TEM) using JEOL, JEM 2100F electron microscope at 200 kV.

The surface morphology of HBPAA/Ag-NP's and HBPAA/ZnO-NP's-loaded pigment prints (SEM) were observed with SEM Model Quanta 250 FEG (field emission gun) attached with EDX unit (energy disperse X-ray analysis) with accelerating voltage 30 kV (FEI Co., Netherland).

3. Results and discussion

In this study we focus on improving the antibacterial activity of pigment printed cellulose-containing fabrics via individual inclusion of certain bio-active ingredients namely HBPAA/Ag-NP's hybrid, HBPAA/ZnO-NP's hybrid, chitosan, choline chloride and triclosan derivative in the print paste formulation along with other ingredients. Discussion of the obtained results follows.

3.1. Effect of hybrid type and concentration

The formation of Ag-NP's as well as ZnO-NP's in the HBPAA matrix are confirmed by TEM images of the prepared hybrids (Fig. 1a and b, respectively). Both the TEM images reveal that the prepared hybrids are well dispersed and almost spherical in shape with particles size in the range of 6–18 and 32–35 nm, respectively.

As far as the changes in %N, K/S and ZI values of the obtained pigment prints as a function of the prepared hybrid and its concentration, the data in Table 1 reveal that: (i) inclusion of any of the prepared hybrid in the pigment – printing paste results in an increase in the %N as well as in the K/S values along with an outstanding enhancement in the antibacterial efficacy against G+ve (*S. aureus*) and G−ve (*E. coli*) bacteria, regardless of the used substrate, (ii) the improvement in both the %N, K/S and ZI values follows the decreasing order HBPAA/ZnO-NP's > HBPAA/Ag-NP's > none and (iii) the higher the hybrid concentration, the darker the shade and the better the imparted antibacterial activity.

The enhancement in the aforementioned properties reflects the positive impacts of including the nominated hybrids in the printing

Table 1
Effect of incorporation of the nominated hybrids into the printing paste[a] on printability and functionality of the cotton/polyester blends.

Hybrid	Conc. (g/kg)	Cotton/polyester (50/50)				Cotton/polyester (35/65)			
		N[b] (%)	K/S[c]	ZI (mm)[d]		N (%)	K/S	ZI (mm)	
				G+ve	G −ve			G+ve	G −ve
None	–	0.201	5.35	0.0	0.0	0.120	5.05	0.0	0.0
HBPAA/Ag-NP's	5	0.226	5.60	9.0	8.0	0.182	5.36	7.0	6.0
	10	0.267	6.05	13.5	12.5	0.225	5.71	11.5	10.0
	20	0.335	6.32	16.5	15.0	0.272	6.01	15.5	13.5
HBPAA/ZnO-NP's	5	0.248	5.82	10.5	9.0	0.199	5.48	8.0	7.0
	10	0.289	6.20	15.0	14.0	0.251	5.85	14.0	12.5
	20	0.356	6.48	19.0	17.5	0.302	6.28	17.0	16.0

[a] Printing paste components: Printofix® Binder MTB-01 (100 g/kg); Printofix® Thickener 160 EG (20 g/kg); Printofix® Red H3BD (20 g/kg); GB Resin®(20 g/kg); silicone softener (10 g/kg); $(NH_4)_2S_2O_8$ (2 g/kg); fixation at 386 W for 5 min.
[b] Nitrogen content.
[c] Color strength.
[d] Zone of inhibition, G+ve (*S. aureus*); G−ve (*E. coli*).

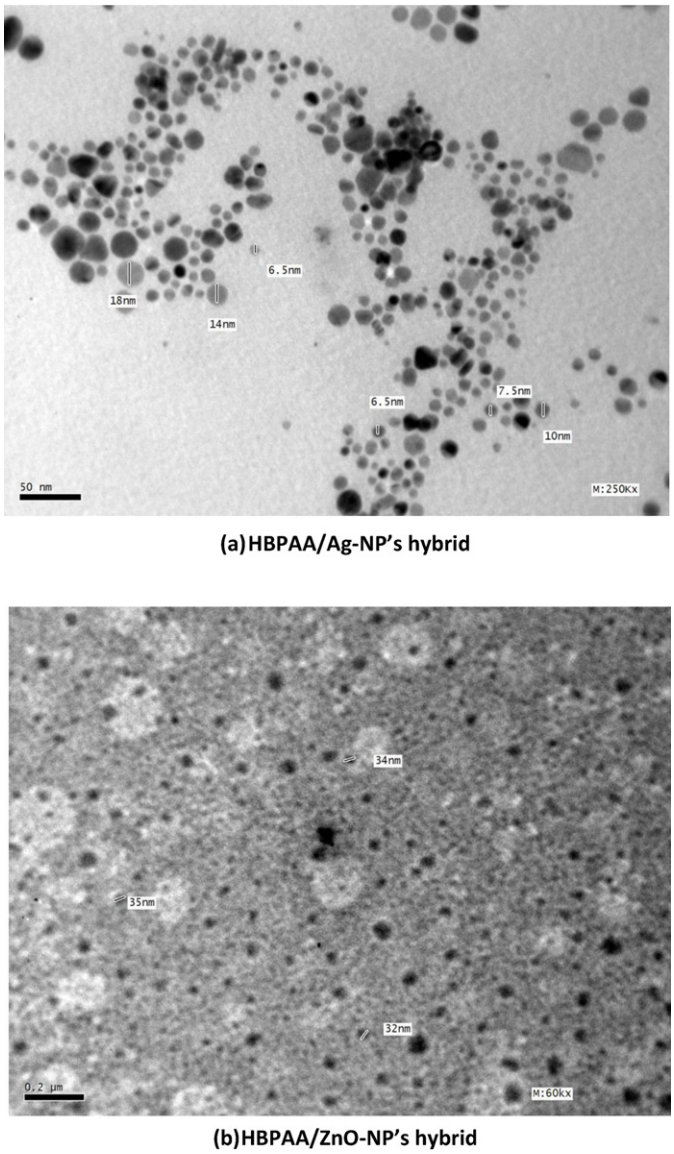


Fig. 1. TEM images of HBPAA/Ag-NP's hybrid (a) and HBPAA/ZnO-NP's hybrid (b).

paste, along with other components, on: (i) enhancing the extent of formation of a three-dimensional linked network, during the curing step, (ii) introducing new active sites into/onto fabric structure, i.e. imino and large number of free terminal —NH_2 groups, thereby enhancing the capacity of the fabric structure to pick-up, interact with and fix the pigment particles and (iii) loading of Ag-NP's or ZnO-NP's, there by exhibiting remarkable antibacterial functionality against G+ve and G−ve strains (Ibrahim, Eid, Youssef, et al., 2012; Ibrahim, Abdel Rehim, & El-Batal, 2010; Zhang, Zhang, Chen, & Lin, 2009; Zhang, Zhang, Lin, & Lu, 2007)

Table 1 demonstrates also that the imparted antibacterial activity is determined by the hybrid type, i.e. HBPAA/ZnO-NP's > HBPAA/Ag-NP's, keeping other parameters constant. This could be discussed in terms of their differences in particle size, specific surface area, antibacterial activity, mechanism of the antibacterial effect, as well as extent of loading and fixation onto/within the crosslinked surface coat. The antibacterial properties of the loaded Ag-NP's could be discussed in terms of their surface interaction with the cell membrane and/or inside interaction, especially smaller particle size, thereby increasing permeability, disturbing respiration as well as causing damage of the cell (Ibrahim, Eid, Youssef, et al., 2012; Jones & Hock, 2010; Mahapatra & Karak, 2008). Oxidation of the molecular structure of bacteria via the generated reactive oxygen species in the presence of dissolved oxygen cannot be ruled out as follows (Jones & Hock, 2010):

$$H_2O + \tfrac{1}{2}O_2 \xrightarrow{\text{Ag-NP's}} H_2O_2 \rightarrow H_2O + [O] \quad (1)$$

Moreover the antibacterial activity of ZnO-NP's is likely attributed to the production of extremely reactive oxygen species such as superoxide anion ($^{\bullet}O_2^-$), H_2O_2, single oxygen and hydroxyl radicals ($^{\bullet}OH$) as follows (Erem, Ozcan, & Skrifvars, 2011; Ibrahim, Refaie et al., 2010):

$$ZnO + h\upsilon \xrightarrow[\text{excitation}]{\text{light}} h^+ + e^- \quad (2)$$

$$H_2O + h^+ \rightarrow {}^{\bullet}OH + H^+ \quad (3)$$

$$O_2 + e^- \rightarrow {}^{-}O_2^{\bullet} + H^+ \rightarrow HO_2^{\bullet} \quad (4)$$

$$2HO_2^{\bullet} \rightarrow O_2 + H_2O_2 \quad (5)$$

which are very powerful oxidizing and toxic species to organic compounds in the bacteria cell. The abrasion effect of ZnO-NP's on the cell of bacteria and its negative impacts including damage to cell membranes, and increasing the diffusion into the cell thereby leading to the death of bacteria (Erem et al., 2011) cannot be ignored.

Table 2
Effect of adding other antibacterial agents to the printing paste[a] on printability and functionality of the cotton/polyester blends.

Additive	Conc. (g/kg)	Cotton/polyester (50/50)				Cotton/polyester (35/65)			
		N[b] (%)	K/S[c]	ZI (mm)[d]		N (%)	K/S	ZI (mm)	
				G+ve	G−ve			G+ve	G−ve
None	–	0.201	5.35	0.0	0.0	0.130	5.05	0.0	0.0
Chitosan	5	0.255	5.70	10.0	9.5	0.223	5.52	7.0	6.0
	10	0.283	6.12	12.0	11.5	0.249	5.85	10.0	9.0
Choline chloride	5	0.225	5.56	12.5	10.5	0.188	5.26	9.5	8.5
	10	0.242	5.78	15.0	14.0	0.210	5.58	13.0	12.5
	15	0.268	5.95	17.5	16.5	0.236	5.73	16.0	15.0
Invasan® (triclosan)	5	0.210	5.46	15.0	14.0	0.153	5.18	10.0	9.5
	10	0.225	5.60	20.0	18.5	0.180	5.50	13.5	13.0
	15	0.250	5.73	24.0	22.5	0.220	6.37	18.0	16.5

[a] Printing paste components: Printofix® Binder MTB-01 (100 g/kg); Printofix® Thickener 160 EG (20 g/kg); Printofix® Red H3BD (20 g/kg); GB Resin®(20 g/kg); silicone softener (10 g/kg); $(NH_4)_2S_2O_8$ (2 g/kg); fixation at 386 W for 5 min.
[b] Nitrogen content.
[c] Color strength.
[d] Zone of inhibition, G+ve (*S. aureus*); G−ve (*E. coli*).

On the other hand, the imparted antibacterial activity against the G+ve and G−ve bacteria follows the descending order: G+ve > G−ve, as well as the enhancement in printability and functionality is governed by type of substrate, i.e. cotton/polyester (50/50) > cotton/polyester (35/65), keeping other parameters constant.

3.2. Type and concentration of other bio-active agents

As far as the changes in the %N, K/S and ZI values of the printed cotton/polyester blends, as a function of type and concentration of the used additives Table 2 demonstrates that: (i) incorporation of any of the nominated additives in the printing paste is accompanied by an increase in the %N and K/S values along with a remarkable improvement in the imparted antibacterial activity of the obtained pigment prints, regardless of the used substrate and type of additive, and (ii) the higher the concentration of the used additive, the better the printability and functionality were. The increase in the %N and K/S values follows the descending order: chitosan > choline chloride > Invasan® > none, reflects the differences among them in chemical composition, molecular weight, functional groups, extent of loading onto/within the binder film/fabric matrix as well as ability to bind pigment particles via their built in cationic active sites, e.g. $-NH_2$, $-^+NR_3$, especially in case of using chitosan and choline chloride respectively, along with terminal —COOH groups of the used binder (Bahmani, East, & Holme, 2000; Cardamone & Turner, 2000; Ibrahim et al., 1996; Ibrahim, Refaie, Youssef, & Ahmed, 2005; Ibrahim, El-Zairy, El-Zairy, & Khalil, 2010; Ravi Kumar, 2000; Zemljic, Strand, Sauperi, & Kleinschek, 2009).

On the other hand, the enhancement in the imparted antibacterial activity of the obtained pigment prints follows the descending order: Invasan® (triclosan derivative) > choline chloride > chitosan > none. This could be attributed to the differences among them in (i) antibacterial effectiveness, (ii) extent of loading and fixation via ether and/or ester crosslinking, with the help of the used crosslinker and/or the binder, as well as via ionic crosslinking, between the binder —COOH groups and the cationic active sites in choline chloride and chitosan, during the fixation step (Chung, Lee, & Kim, 1998; Hashem, Ibrahim, El-Sayed, et al., 2009; Hashem, Ibrahim, El-Shafei, et al., 2009; Ibrahim, Abdel Rehim, et al., 2010; Lim & Hudson, 2004; Orhan et al., 2009), and (iii) mode of action by: damaging of bacterial cells and/or inhibiting of bacterial fatty acid synthesis as in case of using triclosan derivatives (Orhan et al., 2009), by damaging bacterial cell membranes/denaturing of protein along with disrupting of the cell structure as in case of using choline chloride (Gao & Cranston, 2008), or by interfering with bacterial metabolism and/or binding with DNA to inhibit mRNA Synthesis as in case of adding chitosan (Zemljic et al., 2009; Kong, Chen, Xing, & Park, 2010).

Table 2 also shows that the inactivation of G+ve was better than G−ve bacteria, irrespective of the used additive, which reflects their differences in cell wall structure, outer membrane, amenability to inhibition of enzymes and/or extent of inactivation of DNA replication (Gouda & Ibrahim, 2008; Gupta, Bajpai, & Bajpai, 2008).

Moreover, the enhancement in the printability and functionality of the nominated blends is governed by the content of the cellulose component, i.e. cotton/polyester (50/50) > cotton/polyester (35/65), keeping other parameters constant.

3.3. Type of binding agent

For a given set of pigment printing trials, the data in Table 3 demonstrate that inclusion of any of the nominated reactive additives has positive effects on printing and antibacterial performance properties, regardless of the used binding agent. This could be discussed in terms of their ability to modify the formed film properties via introducing an extra positive active sites for accommodation and encapsulation of extra pigment particles, especially in case of using the nominated nitrogenous additives, along with immobilizing antibacterial moieties such as cationic positive groups, triclosan, nano-metal or metal oxide particles, onto/within the crosslinked network during the fixation type, i.e. darker depth of shade along with better antibacterial activity.

Based on the so-obtained data, Table 3 also shows that the improvement in the abovementioned properties is governed by: (i) type of the binding agent, i.e. chemical composition, molecular weight, functional groups, film forming properties, binding capacity as well as ability to form crosslinks and durability to wash and crocking (Hamilton & Chiweshe, 1998; Ibrahim, El-Zairy, et al., 2005), and follows the descending order: Printofix® MTB > G-Binder®FMD, (ii) nature of the loaded-antibacterial agents, and follows the descending order: triclosan derivatives (Invasan®) > HBPAA/ZnO-NP's > choline chloride > HBPAA/Ag-NP's > chitosan ≫ none, (iii) substantivity and accessibility to pigment particles, and follow the descending order: HBPAA/ZnO-NP's ≫ HBPAA/Ag-NP's > chitosan > choline chloride > triclosan > none, and (iv) type of substrate and its cellulose component and follow the descending order: cotton/polyester (50/50) > cotton/polyester (35/65)

Table 3
Effect of using different binders in the print paste formulation[a] on printability and functionality of the cotton/polyester blends.

Binder (100 g/kg)	Additives (g/kg)	Cotton/PET blend	N[b] (%)	K/S[c]	ZI[d] (mm)		WF[e]	LF[f]	RF[g]	
					G+ ve	G− ve			Dry	Wet
Printofix® MTB	Chitosan (10 g/kg)	50/50	0.283	6.12	12.0	11.5	4–5	5	4–5	4
		35/65	0.249	5.85	10.0	9.0	4–5	5	4–5	4
	Choline chloride (15 g/kg)	50/50	0.268	5.95	17.5	16.5	4–5	5	4–5	4
		35/65	0.236	5.73	16.5	15.0	4–5	5	4–5	4
	Invasan® (20 g/kg)	50/50	0.250	5.73	24.0	22.5	4–5	5	4–5	4
		35/65	0.220	5.37	18.0	16.5	4–5	5	4–5	4
	HBPAA/Ag-NP's (20 g/kg)	50/50	0.335	6.32	16.5	15.0	4–5	5	4–5	4
		35/65	0.292	6.01	15.5	13.5	4–5	5	4–5	4
	HBPAA/ZnO-NP's (20 g/kg)	50/50	0.356	6.48	19.0	17.5	4–5	5	4–5	4
		35/65	0.308	6.28	17.0	16.0	4–5	5	4–5	4
	None	50/50	0.201	5.35	0.0	0.0	4	5	3–4	3
		35/65	0.120	5.05	0.0	0.0	4	5	3–4	3
GB-Binder® FMD	Chitosan (10 g/kg)	50/50	0.270	5.75	10.0	8.0	4–5	5	4–5	4
		35/65	0.230	5.40	8.5	6.5	4–5	5	4	3–4
	Choline chloride (15 g/kg)	50/50	0.250	5.46	16.0	13.0	4–5	5	4–5	4
		35/65	0.221	5.30	12.5	10.5	4–5	5	4	3–4
	Invasan® (20 g/kg)	50/50	0.232	5.32	19.0	17.5	4–5	5	4–5	4
		35/65	0.208	5.18	14.5	13.0	4–5	5	4	3–4
	HBPAA/Ag-NP's (20 g/kg)	50/50	0.308	5.96	13.0	11.5	4–5	5	4–5	4
		35/65	0.259	5.60	11.5	9.5	4–5	5	4	3–4
	HBPAA/ZnO-NP's (20 g/kg)	50/50	0.333	6.15	18.0	16.0	4–5	5	4–5	4
		35/65	0.287	5.92	15.0	13.5	4–5	5	4	3–4
	None	50/50	0.201	5.35	0.0	0.0	4	5	3–4	3
		35/65	0.120	5.05	0.0	0.0	4	5	3–4	3

[a] Printing paste components: Binder (100 g/kg); Printofix® Thickener 160 EG (20 g/kg); Printofix® Red H3BD (20 g/kg); GB Resun®(20 g/kg); silicone softener (10 g/kg); $(NH_4)_2S_2O_8$ (2 g/kg); fixation at 386 W for 5 min.
[b] Nitrogen content.
[c] Color strength.
[d] Zone of inhibition, G+ve (*S. aureus*); G−ve (*E. coli*).
[e] Wash fastness.
[f] Light fastness.
[g] Rubbing fastness.

Also, it is clear from the data in Table 3 that (i) the evaluated fastness properties ranged from good to excellent, (ii) the dry rub was found to be slightly better than the wet one, as a direct consequence of the presence of loose and untrapped pigment particles within the produced a three-dimensional network (Ibrahim, El-Zairy, et al., 2005), and (iii) the addition of the nominated antibacterial auxiliaries to the pigment print formulation does not inhibit binder film formation and subsequent pigment entrapment and encapsulation during curing step.

3.4. Pigment colorant

The impact of using different pigment colorants on the printability and functionality of the obtained prints are shown in Table 4. For a given set of pigment printing conditions, the data signify that: (i) the extent of printing as well as enhancement in the imparted antibacterial properties is determined by the type of pigment colorant, keeping other parameters constant, (ii) the changes in printing properties by using different colorants reflect the differences among them in particle size, degree of dispersion, chemical composition, color, hue, compatibility with other ingredients and subsequent entrapment and encapsulation of pigment particles during the microwave irradiation step (Giesen & Eisenlohr, 1994; Ibrahim, El-Zairy, et al., 2005; Uddin & Lomas, 2005), (iii) the change in the antibacterial functionality upon using different pigment colorants could be discussed in terms of differences in the extent of fixation of the nominated antibacterial finish auxiliaries onto/within the crosslinked binder film to produce a three-dimensional network structure along with other ingredients, i.e. binder, crosslinker, softener and pigment particles, during the curing step, (iv) the overall fastness properties of the obtained antibacterial pigment prints ranged from good to excellent, and (v) inclusion of the silicone softening agent along with other ingredients in the print paste formulations enhances the softness of the obtained pigment prints, most probably due to its ability to modify the fabric surface as well as to overcome the surface harshness and hardness of pigment prints (Giesen & Eisenlohr, 1994; Ibrahim, El-Zairy, et al., 2005; Schwindt & Faulhaber, 1984).

Needless to say, the extent of improvement in the imparted antibacterial properties against the G+ve and G−ve bacteria is mainly governed by the type of antibacterial finish auxilary, keeping the other parameters constant.

3.5. SEM images and EDX spectrum

The so-obtained experimental results, especially in case of using the nominated hybrids, were proved and confirmed by observation of surface morphology (SEM) and the corresponding composition analysis (EDX).

SEM images of the pigment printed and HBPAA/Ag-NP's-loaded cotton/polyester blends as well as the pigment printed and HBPAA/ZnO-NP's-loaded blends were demonstrated in Figs. 2(a and c) and 3(a and c), respectively. From the given images, it can be noticed the deposition of the binder film containing different ingredients along with the loaded nano-particles. The location and extent of distribution of the formed coat are governed by the type and morphology of the printed substrate as well as the extent of interactions among the substrate components, the various ingredients and the incorporated hybrids during the microwave irradiation step.

Moreover, the presence of silver and zinc elements on the surfaces of the obtained pigment prints was proved using EDX spectra

Table 4
Effect of using other pigment colorants in the pigment paste formulation[a] on printability and functionality of the cotton/polyester blends.

Pigment (20 g/kg)	Additives (g/kg)	Cotton/PET blend	K/S[b]	ZI[c] (mm)		WF[d]	LF[e]	RF[f]	
				G+ve	G−ve			Dry	Wet
Imperon® Royal Blue SP	Chitosan (10 g/kg)	50/50	9.69	11.0	9.5	4–5	5	4	3–4
		35/65	9.43	9.5	8.0	4–5	5	4	3–4
	Choline chloride (15 g/kg)	50/50	9.39	15.0	12.5	4–5	5	4	3–4
		35/65	9.16	13.0	12.0	4–5	5	4	3–4
	Invasan® (20 g/kg)	50/50	9.20	17.5	15.0	4–5	5	4	3–4
		35/65	8.85	14.5	13.0	4–5	5	4	3–4
	HBPAA/Ag-NP's (20 g/kg)	50/50	10.10	13.5	12.0	4–5	5	4	3–4
		35/65	9.75	11.0	10.0	4–5	5	4	3–4
	HBPAA/ZnO-NP's (20 g/kg)	50/50	11.24	16.0	14.0	4–5	5	4	3–4
		35/65	10.87	14.5	12.0	4–5	5	4	3–4
Unisperse® Yellow MR	Chitosan (10 g/kg)	50/50	7.26	12.0	11.0	5	5	5	4
		35/65	6.80	10.0	8.5	5	5	5	4
	Choline chloride (15 g/kg)	50/50	7.01	18.5	16.0	5	5	5	4
		35/65	6.60	16.5	14.5	5	5	5	4
	Invasan® (20 g/kg)	50/50	6.62	21.0	18.5	5	5	5	4
		35/65	6.23	19.0	17.5	5	5	5	4
	HBPAA/Ag-NP's (20 g/kg)	50/50	7.55	15.0	13.5	5	5	5	4
		35/65	6.95	13.5	12.5	5	5	5	4
	HBPAA/ZnO-NP's (20 g/kg)	50/50	7.98	19.0	17.0	5	5	5	4
		35/65	7.49	17.5	16.0	5	5	5	4
Printofix®Green HXP	Chitosan (10 g/kg)	50/50	9.49	12.5	11.0	5	5	4	3–4
		35/65	9.19	11.5	9.5	5	5	4	3–4
	Choline chloride (15 g/kg)	50/50	9.14	16.0	14.0	5	5	4	3–4
		35/65	8.78	14.5	13.0	5	5	4	3–4
	Invasan® (20 g/kg)	50/50	8.62	19.0	17.5	5	5	4	3–4
		35/65	8.27	17.0	16.0	5	5	4	3–4
	HBPAA/Ag-NP's (20 g/kg)	50/50	10.15	14.5	12.5	5	5	4	3–4
		35/65	9,87	13.0	11.5	5	5	4	3–4
	HBPAA/ZnO-NP's (20 g/kg)	50/50	11.02	18.5	16.0	5	5	4	3–4
		35/65	10.48	16.5	14.5	5	5	4	3–4

[a] Printing paste components: Printofix® Binder MTB-01 (100 g/kg); Printofix® Thickener 160 EG (20 g/kg); Pigment (20 g/kg); GB Resin®(20 g/kg); silicone softener (10 g/kg); $(NH_4)_2S_2O_8$ (2 g/kg); fixation at 386 W for 5 min.
[b] Color strength.
[c] Zone of inhibition, G+ve (*S. aureus*); G−ve (*E. coli*).
[d] Wash fastness.
[e] Light fastness.
[f] Rubbing fastness.

as shown in Figs. 2(b and d) and 3(b and d), respectively. The existence of some elements such as carbon, nitrogen, oxygen and silicone was also confirmed, as a direct consequence of loading of the nominated hybrids as well as fixation of the silicone-based softener onto/within the produced a three-dimensional network coat. On the other hand, higher content of the aforementioned elements loaded-onto cotton/polyester (50/50) blend compared to cotton/polyester (35/65) blend was clearly observed.

3.6. Tentative mechanism

The enhancement in the printability as well as the remarkable improvement in the antibacterial functionality of the obtained pigment prints via inclusion of any of the nominated finish auxiliaries into a single paste formulation could be explained as given in Scheme 1 (Harper & Stone, 1986; Hashem, Ibrahim, El-Shafei, et al., 2009; Ibrahim, El-Zairy, et al., 2005; Ibrahim, Abdel Rehim, et al., 2010; Ibrahim, Eid, Youssef, et al., 2012; Kong et al., 2010):

(i) Reaction between the binder and the cellulose component:

$$\underset{\text{Cotton}}{\text{Cell.OH}} + \underset{\text{self crosslinking polyacrylate binder}}{\text{ROH}_2\text{C—Binder—COOR}} \xrightarrow[\Delta]{\text{H}^+} \underset{\text{(I)}}{\text{Cell.O.H}_2\text{C—Binder—CH}_2\text{COOR}} \quad (6)$$

(ii) Fixation of pigment particles:

$$\text{Cell.OH} + \text{ROH}_2\text{C—Binder—CH}_2\text{COOR} + \text{pigment} \xrightarrow[\Delta]{\text{H}^+} \underset{\text{(II)}}{\text{Pigment-containing binder film-loaded onto fabric surface}} \quad (7)$$

(iii) Enhancing the extent of crosslinking and durability:

$$\text{Cell.OH} + \text{ROH}_2\text{CN—DMDHEU—NCH}_2\text{OR} + \text{ROH}_2\text{C—Binder—COOR} + \text{pigment} \xrightarrow[\Delta]{\text{H}^+} \underset{\text{(III)}}{\text{Crosslinked three-dimensional network-encapsulated pigment particles}} \quad (8)$$

(iv) Enhancing the flexibility of the binder film to give crosslinked/softer film:

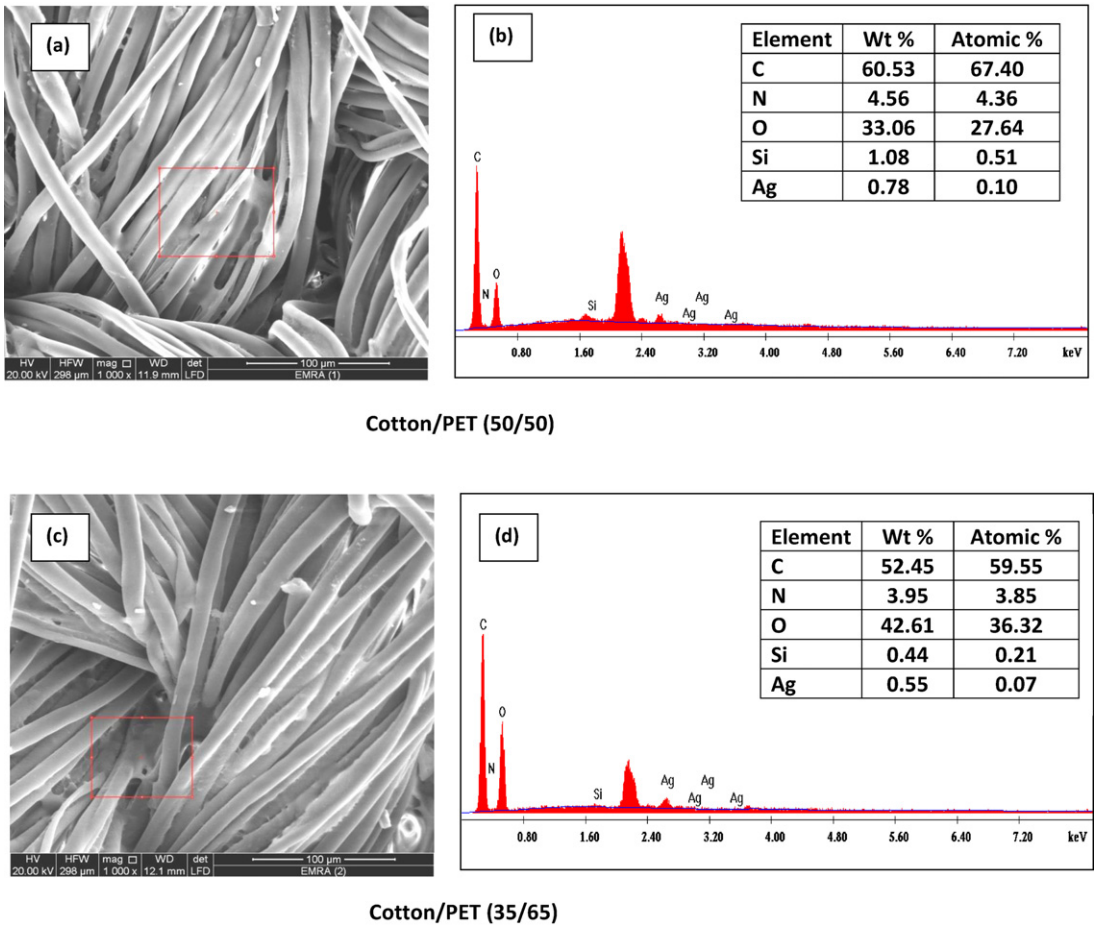


Fig. 2. SEM images and EDX spectrum of: pigment printed and HBPAA/Ag-NP's-loaded cotton/polyester (50/50) (a and b); pigment printed and HBPAA/Ag-NP's-loaded cotton/polyester (35/65) (c and d).

$$\text{Cell.OH} + \text{ROH}_2\text{CN—DMDHEU—NCH}_2\text{OR} + \text{ROH}_2\text{C—Binder—COOR} + \text{H}_2\text{N—silicone softener} + \text{pigment} \xrightarrow[\Delta]{\text{H}^+} \underset{\text{(IV)}}{\text{Crosslinked softer film-containing pigment particles/loaded onto the fabric surface}} \quad (9)$$

(v) Imparting antibacterial activity to the pigment prints via inclusion of the nominated antibacterial auxiliaries into the pigment paste:

$$\text{(I), (II) and/or (III)} + \text{H}_2\text{N—Chitosan} \xrightarrow[\Delta]{\text{H}^+} \text{Antibacterial pigment print contained cationic active sites} \quad (10)$$

$$\text{(I), (II) and/or (III)} + \text{HO—Choline chloride} \xrightarrow[\Delta]{\text{H}^+} \text{Antibacterial pigment print contained quaternary ammonium group} \quad (11)$$

$$\text{(I), (II) and/or (III)} + \text{HO—Triclosan derivative} \xrightarrow[\Delta]{\text{H}^+} \text{Antibacterial pigment print contained triclosan moieties} \quad (12)$$

$$\text{(I), (II) and/or (III)} + \underset{\text{HBPAA/Ag-NP's hyprid}}{\text{[Ag}^\circ\text{(NH}_2\text{)}_6\text{]}} \xrightarrow[\Delta]{\text{H}^+} \text{Antibacterial pigment print contained Ag-NP's} \quad (13)$$

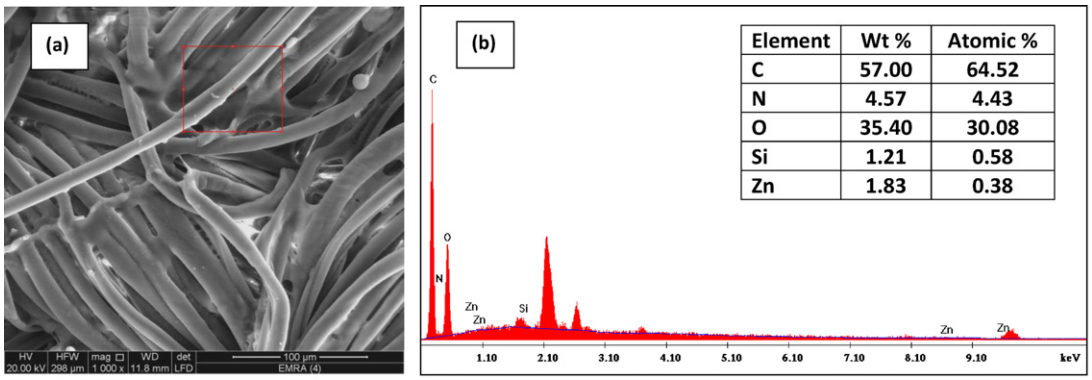
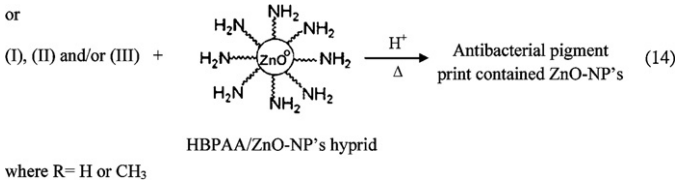


Fig. 3. SEM images and EDX spectrum of: pigment printed and HBPAA/ZnO-NP's-loaded cotton/polyester (50/50) (a and b); pigment printed and HBPAA/ZnO-NP's-loaded cotton/polyester (35/65) (c and d).

Table 5
Print paste formulation[a] vs durability to wash.

Antibacterial auxilary	Cotton/PET blend	One washing cycle			20 washing cycles		
		K/S[b]	ZI (mm)[c]		K/S	ZI (mm)	
			G+ve	G−ve		G+ve	G−ve
Chitosan (10 g/kg)	50/50	6.12	12.0	11.5	5.35	10.0	9.0
	35/65	5.85	10.0	9.0	5.10	8.5	7.0
Choline chloride (15 g/kg)	50/50	5.95	17.5	16.5	5.18	14.5	13.5
	35/65	5.73	16.5	15.0	5.02	13.0	12.5
Invasan® (20 g/kg)	50/50	5.73	24.0	18.0	5.00	20.5	16.0
	35/65	5.37	22.5	16.0	4.75	19.0	18.0
HBPAA/Ag-NP's (20 g/kg)	50/50	6.32	16.5	15.0	5.62	13.5	12.5
	35/65	6.01	15.5	13.5	5.30	12.0	11.5
HBPAA/ZnO-NP's (20 g/kg)	50/50	6.48	19.0	17.5	5.70	16.5	14.5
	35/65	6.28	17.5	16.0	5.48	14.5	13.0

[a] Printing paste components: Printofix® Binder MTB-01 (100 g/kg); Printofix® Thickener 160 EG (20 g/kg); Printofix® Red H3BD (20 g/kg); GB Resin®(20 g/kg); silicone softener (10 g/kg); $(NH_4)_2S_2O_8$ (2 g/kg); fixation at 386 W for 5 min.
[b] Color strength.
[c] Zone of inhibition, G+ve (*S. aureus*); G−ve (*E. coli*).



Scheme 1. Tentative reactions mechanism.

On the other hand, formation of functionalized pigment binder film onto the polyester component during the fixation step cannot be phased out.

3.7. Durability to wash

Based on the obtained results, Table 5, it was observed that increasing the number of washing cycles up to 20 brings about a

reasonable decrease in the depth of the obtained pigment prints, expressed as K/S values, as well as in the imparted antibacterial activity, expressed as ZI values, i.e. very sufficient durability. The extent of color and antibacterial activity retention is governed by the nature of antibacterial agent as well as the cotton/polyester ratio in the blended fabrics, keeping other parameters constant.

4. Conclusions

- In this work inclusion of pigment printing ingredients along with antibacterial finish auxiliaries into a single print paste formulation to get high quality cotton/polyester pigment prints with a remarkable antibacterial functionality were investigated.
- Higher color strength values along with a better antibacterial activities are obtained in the case of using chitosan (10 g/kg), choline chloride (15 g/kg), triclosan derivative (20 g/kg), HBPAA/Ag-NP's hybrid (20 g/kg) or HBPAA/ZnO-NP's hybrid along with other printing paste ingredients followed by microwave irradiation at 386 W for 5 min.
- The extent of improvement in the printing and antibacterial performance properties is governed by type and concentration of antibacterial auxiliary, type of binding and coloring agent as well as the content of cotton cellulose in the blended substrates.
- After 20 wash cycles, the obtained pigment prints still had a remarkable antibacterial function along with a noticeable color retention and soft handle.
- The observed combined process can be easily adapted on industrial scale.

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