Smart options for pigment printing and multifunctionalization of wool and polyester/ wool blended fabrics in single step

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Abstract:
This research work was aimed to produce multifunctionalized wool and polyester/ wool pigment prints in one step process by individually incorporating TiO_2-nanoparticles (TiO_2-NP's, 10g/Kg), silicon micro-emulsion (20g/Kg) or a water/oil-repellent agent (40g/Kg), in pigment printing paste [pigment color (20g/kg); synthetic thickening agent (20g/kg); binder (100 g/kg); crosslinking agent (10g/kg); ammonium persulfate $(NH_4)_2S_2O_8$ (2g/kg)] followed by printing and microwave fixation at 1300W/4 min. The antimicrobial properties, UV-protection, soft-handle or water/oil-repellency along with printing properties were evaluated as well as the depth of the obtained functionalized pigment prints.

Keywords:
Wool; polyester/ wool fabrics; pigment printing; antibacterial finishing; UV-protection; soft-handle; water/ oil-repellency and one-step.

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1. Introduction:

Natural fibers based textiles are good environment for the growth of microorganism (bacteria, fungi, algae, dust mites and yeast). This growth of microorganisms on textiles lead to a lot of negative impacts such as infection by pathogenic microorganisms, unpleasant odor and also on the textile itself such as stains, discoloration and loss in mechanical strength, therefore, it is very important to using antimicrobial agents [1, 2].

The antimicrobial agents inhibit microorganisms in different ways. Generally, attacks the cell wall of the microbe, inhibits the cell wall synthetics, and alters the cytoplasmic membrane permeability [1, 3-5].

Combining polyester and protein fibres improves performance and quality properties, leads to the development of new textile products, lowers the cost of [6, 7]. Functional finishes of textiles include but not limited to antibacterial [8-11], UV protection [12-15], self-cleaning [16, 17], and water and oil repellency [18, 19].

This research aims to improve both the printing properties and functional properties to pigment coloration of wool and polyester/wool blended fabrics such as antimicrobial properties, UV protection, soft-handle, or water/oil-repellency in one-step through inclusion of certain functional additives into pigment paste.

2. Experimental:

2.1. Materials:

Mill-scoured and semi-bleached wool and polyester/wool blended fabrics (220g/m^2 and 50/50, 230 g/m^2 respectively) were used in this study.

Ciba® Oleophobol® Co. (oil, water and stain repellent agent based on dispersion of fluoropolymers containing extender, Ciba), Ultratex® MHT conc. (softening agent based on micro emulsion concentrate of a quaternary polydimethyl siloxane, Huntsman), Knittex® FEL crosslinking agent (reactant crosslinking agent based on a modified dimethylol dihydroxy ethyleneurea, Huntsman), DAICO® Thick. 160 (synthetic thickener based on polyacrylate, Daico, Egypt), Printofix® Binder MTB-01 liquid (binding agent based on acrylate copolymer, Clariant), Pigment Red 146 and Pigment Blue 153 (Daico, Egypt) were of commercial grade.

All other chemicals used in this study such as ammonium persulphate $[(NH4)_2S_2O_8]$ and acetic acid were of laboratory regent grade.

TiO_2-nanoparticles (TiO_2-NP's) were prepared as previously reported using titanium tetraisoproproxide precursor (Sigma) with 2-propanol and nitric acid (10).

2. 2. Methods:

2.2.1. Pigment printing and Functionalization in one-step:

The nominated wool and polyester/wool blend were printed with pigments and functionalized in one-step using a flat screen technology and the following print paste formulas:

Content	g/Kg paste
Pigment color	20g
Printofix® Binder MTB-01	100g
DAICO® Thick. 160	20g
Knittex® FEL crosslinking agent	10g
Ammonium persulphate $(NH_4)_2S_2O_8$	2g
Functional additives:	
Water & oil repellent	0-60g
Silicon-softener	0-30g
TiO_2-NP's	0-20g
H2O	Xg
Total	1000g

Printed fabric samples were then simultaneously dried and fixed in a commercial microwave oven at output of 1300W/4 min.

2.2.2. Testing:

The depth of the obtained prints, expressed as K/S values, was determined from the reflectance measurements using the Kubelka Munk equation:

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

Fastness properties to washing, rubbing, perspiration and light of printed fabric samples were evaluated according to AATCC test methods: (61-1972), (8-1972), (15-1973) and (16A-1972) respectively.

Antibacterial activities of printed samples were evaluated qualitatively, expressed as zone of growth inhibition (ZI, mm) against G+ve bacteria (S. aureus) and G-ve bacteria (E. coli) according to AATCC test method (147-1988).

UV-protection properties expressed as UPF was evaluated according to AS/NZS Standard 4399-1996. According to the AS/NZS Standard, fabrics are rated as good, very good, or excellent protection if their UPF values range from 15 to 24, 25 to 39, or above 40 respectively.

Stiffness of the untreated and treated samples was assessed according to AATCC Test Method (D 1388-96).

The changes in the surface roughness of the untreated and treated fabric samples were evaluated according to JIS 94 Standard, using SE 1700α, Japan (surface roughness measuring instrument)

Water and oil repellency property was assessed according to AATCC Test Method 22-1989.

3. Results and Discussion:

The present work focuses on improving the pigment printing properties and functionalization of wool and polyester/wool fabrics such as antimicrobial properties, UV-protection, soft-handle and water/oil-repellency in one-step through inclusion of functional additives namely TiO_2-nanoparticles, silicon micro-emulsion or a water/ oil-repellent agent into pigment printing paste then printing and microwave fixation.

3.1. Effect of type and concentration of functional additives

Fig 1 (a) Shows that effect of the TiO_2-nanoparticles concentration on the depth of the obtained wool and polyester/wool prints, i) increasing TiO_2-nanoparticles concentration up to 10g/Kg paste leads to a significant increase in the K/S values of obtained wool and polyester/wool pigment prints, ii) This improvement in K/S values may be attributed to TiO_2-NP's impact as a co-catalyst in enhancing the extent of crosslinking of the binder film, which in turn improves the performance of capturing and entrapping pigment particles as well as adhesion and fixation onto the surface of fabrics during microwave thermofixation [11, 20-22] iii) Regardless of the used substrate , further increase in TiO_2-NPs concentration above 10g/Kg has a negative impact on the depth of shade for the printed samples expressed by decrease in the K/S values [7].

Fig 1(b) Shows that effect of the used water/oil repellent agent concentration on the depth of the obtained prints, i) increasing water/oil repellent agent concentration up to 40g/Kg paste leads to enhancement in the K/S of obtained wool and polyester/wool pigment prints, ii) increase in water/oil repellent agent, beyond 40g/Kg, has leads to decrease in the k/s of the obtained pigment prints, iii) This increment in the K/S values is most likely resultant to forming water/oil repellent polymer film during the microwave thermofixation step alone or in combination with other components, such as binder and crosslinker, which in turn lead to increase the extent of fixation of pigment particles onto the coated fabric surface [7, 18, 23].

While, Fig 1 (c) shows that incorporation the silicone softener up to 20g/Kg, into the pigment printing formulation along with other ingredients resulted in significant increase in K/S values of the printed wool and polyester/wool substrates, which could be discussed in terms of better integration and fixation of the pigment particles within the binder and/or the softener film onto the printed samples surface [24, 25].

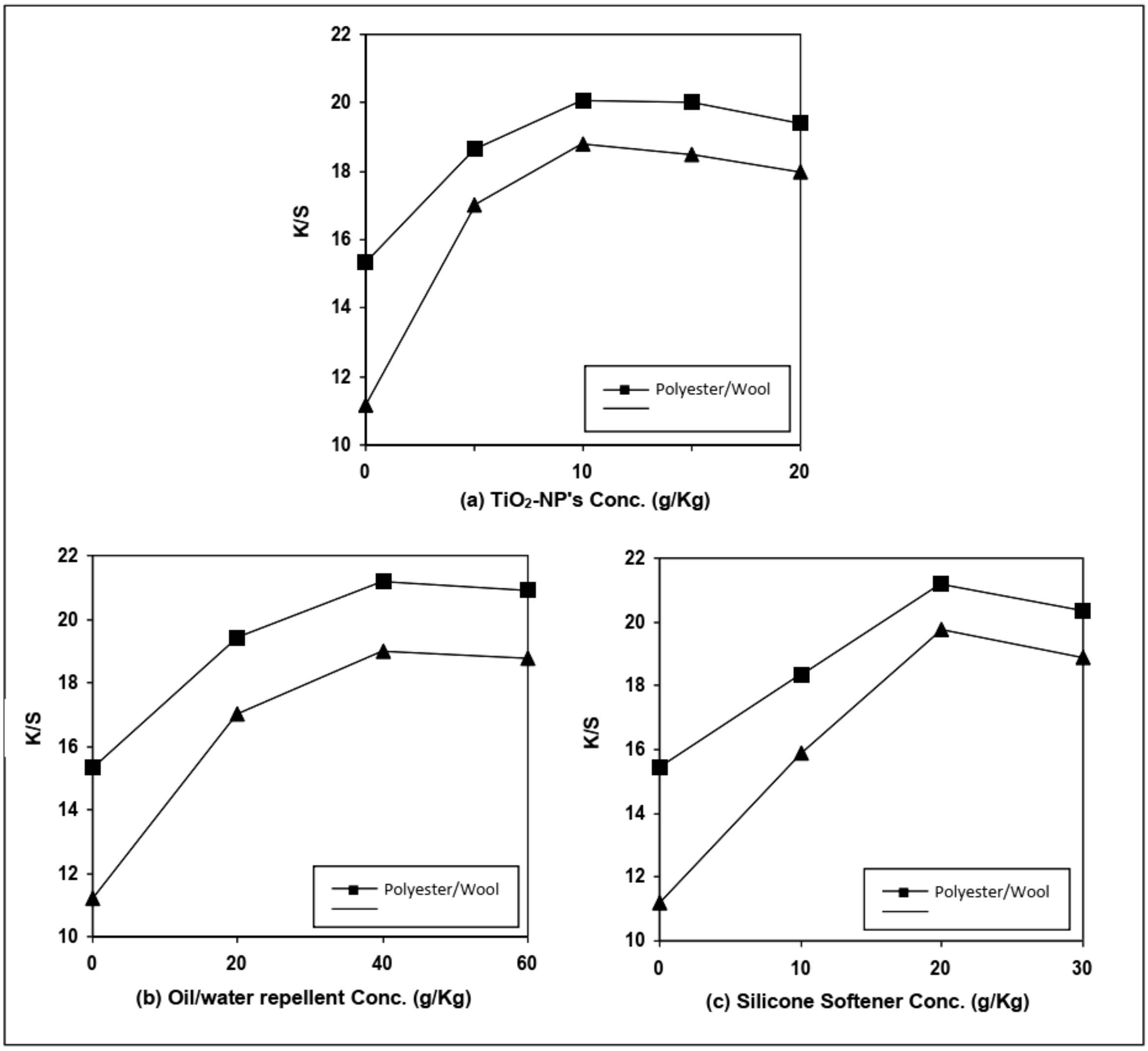

Fig.1. Effect of TiO_2-NP's (a), Oil/water repellent (b) and Silicone Softener (c), concentration on the depth of the obtained prints.

Printing paste components: Pigment Red 146(20g/Kg); synthetic thickener(20g/Kg); Printofix®Binder MTB(100g/Kg); crosslinker(10g/Kg); and $(NH_4)_2S_2O_8$(2g/Kg). Microwave fixation: 1300W/4min.

3.2. Antibacterial/ Anti-uv and pigment printing in one step:

Table 1. demonstrates that inclusion of TiO_2-nanoparticles (10g/Kg) as antibacterial and anti-UV agent into pigment paste on antibacterial and anti-UV functionality /printing properties of printed wool and wool/polyester blends using different pigment colorants, i) an outstanding enhancment in the antibacterial activity, expressed as ZI values (zone of growth inhibition) (ZI, mm), against the tested bacteria, and in the UV-protection capacity, expressed as UPF values, of the obtained pigment prints samples, ii) an enhancment in the K/S values of the obtained pigment prints as well as their fastness properties, regardless of the used pigment colorant, iii) the improvment in the imparted antibacterial property and the printability is governed by the type of substrate and follows the decreasing order: polyester/wool > wool, keeping other parameters constant, while the change in the pigment printability is determind by the type of used pigment [7], iv) the antibacterial activity against the nominated bacteria follows the descending order G+ve > G-ve, indicating differences between them in membrane structure, response for inactivation and ability to resist damage and destruction regardless of the used substrate [26-28], v) the UV-protection functionality of the obtained pigment prints is

governed by the extent of loading TiO_2-NP's onto the substrate, type of substrate (i.e. polyester/wool > wool) as well as the used pigment colorant, vi) The imparted functionalities, i.e. anti-bacterial/ anti-UV properties, are a direct consequence to the positive role of loaded TiO_2-NPs generating highly reactive species, such as superoxide ions, hydroxyl radicals, which in turn enhace the extent of attacking the bacterial cell membranes and destroying them, as well as scattering and absorbing the harmful UV-B radiation [29-31], vii) the improvement in pigment printability properties after the addition of TiO_2-NPs to the printing paste can be attributed to the TiO_2-NPs' role as a co-catalyst in crosslinking and hence enhancing the extent of polymerization and fixation of the binder film onto surface of the printed fabric samples during the subsequent microwave fixation [21, 29], while the slight enhancment in the light fastness properties of the obtained pigment prints is attributed to the TiO_2-NPs' capacity to absorb UV radiation.

Table 1 . Effect of Tio_2NPs treatment on the printing properties and uv-protecting and antimicrobial properties of pigment prints different colors

Pigment Colors	Substrate		K\S	Incr. in K/S (%)	WF		RF		PF				LF	UPF	Antimicrobial Activity ZI (mm)	
									Acidic		Alkaline					
					Alt	C	Dry	Wet	Alt	C	Alt	C			G +ve	G −ve
Pigment Red 146	W	UT	11.18	68.24	3	3-4	4	4	4	3-4	3	3	2-3	46.44	0.0	0.0
		T	18.81		3-4	4	4-5	4-5	4-5	4	4	4	4-5	59.30	12.0	10.5
	W/PET	UT	15.34	30.89	3	3	3	2-3	4	4	2-3	4	4	39.55	0.0	0.0
		T	20.08		3-4	4-5	3-4	4	4-5	5	4	4-5	5	73.08	19.5	13.5
Pigment Blue 15:3	W	UT	10.92	16.94	3	2-3	4-5	4	4-5	2-3	4	3	2-3	44.44	0.0	0.0
		T	12.77		4	4	5	4-5	5	3	4-5	3-4	4-5	53.11	16.0	15.0
	W/PET	UT	13.03	16.11	2-3	4-5	4	3	3	4	4	4-5	2-3	42.46	0.0	0.0
		T	15.13		4	5	4-5	4	4-5	4-5	4	5	4-5	61.56	22.0	20.0

Pigment printing conditions: Pigment color (20 g/kg); DAICOTHICK 1600 (20 g/kg); Printofix Binder MTB-01 (100 g/kg); ammonium persulfate (2 g/kg); KNITTEX® FEL crosslinking agent (10 g/kg); Tio_2NPs (10 g/kg); Curing at 1300/4 min using microwave, followed by after –washing at 40˚C for 15 min. in presence of (2 g/L) nonionic wetting agent, K/S: color depth; WF: wash fastness; RF: rubbing fastness; PF: perspiration fastness; LF: light fastness; Alt: alteration; C: staining on cotton; UPF: ultraviolet protection factor; ZI: zone of inhibition; W: wool; W/ PET: wool/ polyester blend; UT: untreated; T: Pigment printed with Tio_2NPs.

3.3. Combined water/oil repellent/ pigment printing in one step:

Table 2. demonstrates that inclusion of water/oil repellent (40g/Kg) as water/oil repellent agent into pigment paste on water/oil repellency and printing properties of printed wool and wool/polyester blends using different pigment colorants, i) the outstanding increase in water/oil repellent ratings was govened by the type of substrate which follow the decreasing order: wool (70/5) > polyester/wool (50/4), ii) The enhancment in the water/oil repellent properties is attributed to the formation of a hydrophobic/oleophobic polymer film on the printed fabric surface which lead to decrease fabric surface energy and hence repellent properties [32](21, 22), iii) The addition of the water/oil repellent to the printing formulation has a favourable effect on the K/S of the obtained wool and polyester/wool prints as well as a slight improvement in the fastness properties as a direct result of enhancing the extent of entrapment and entanglement of the pigment particles in the crosslinked polymer film onto the fabric surface. Additionally, the data reveals that the incorporation of the used water/oil-repellent brings about an outstanding improvement in the surface smoothness of the printed fabric samples as a result of the deposition of a hydrophobic film onto the fibre and fabric surface [7].

Table 2 . Effect of Ciba® OLEOPHOBOL® CO (Oil& Water Repellent) treatment on the printing properties and oil/water repellent properties of pigment prints different colors

Pigment Colors	Substrate		K\S	Incr. in K/S (%)	WF		RF		PF				LF	Water and oil repellency		Surface roughness. (µm)
									Acidic		Alkaline					
					Alt	C	Dry	Wet	Alt	C	Alt	C		Water	Oil	
Pigment Red 146	W	UT	11.18	70.03	3	3-4	4	4	4	3-4	3	3	2-3	0	0	21.61
		T	19.01		4	4	5	5	4-5	4	4	4	4	70	5	18.34
	W/PET	UT	15.34	38.07	3	3	3	2-3	4	4	2-3	4	4	0	0	18.74
		T	21.98		3-4	3	3	3	4-5	4	3	4-5	4-5	50	4	14.12
Pigment Blue 15:3	W	UT	10.92	23.26	3	2-3	4-5	4	4-5	2-3	4	3	2-3	0	0	20.04
		T	13.46		4-5	4	5	5	5	3	4-5	4	4-5	70	5	16.71
	W/PET	UT	13.03	16.27	2-3	4-5	4	3	3	4	4	4-5	2-3	0	0	19.96
		T	15.15		4	5	5	4	3-4	4-5	5	5	4	50	4	14.28

Pigment printing conditions: Pigment color (20 g/kg); DAICOTHICK 1600 (20 g/kg); Printofix Binder MTB-01 (100 g/kg); ammonium persulfate (2 g/kg); KNITTEX® FEL crosslinking agent (10 g/kg); Ciba® OLEOPHOBOL® CO (40 g/kg); Curing at 1300/4 min using microwave, followed by after –washing at 40˚C for 15 min. in presence of (2 g/L) nonionic wetting agent, K/S: color depth; WF: wash fastness; RF: rubbing fastness; PF: perspiration fastness; LF: light fastness; Alt: alteration; C: staining on cotton; Ra: Roughness; W: wool;W/ PET: wool/ polyester blend; UT: untreated; T: Pigment printed with Ciba® OLEOPHOBOL® CO.

3.4. Combined soft-handle finish/pigment printing in one step:

Table 3 reveals that: i) incroporation of the silicone softener in the pigment printing formulation, (20g/Kg), leads to an outstanding decrease in the stiffness of the obtained pigment prints in addition to a significant improvment in the smoothness of the obtained pigment prints, ii) The improve in surface properties of obtained prints is due the positive role of silicon as a lubricant between the fibres in the yarn and between the yarns of the fabric, which results in improving the soft touch of fabric surface and reduce the stiffness [33, 34], iii) an enhancment in the K/S of the obtained pigment prints along with their fastness properties, irrespective of the kind of employed pigment [24], iv) the improvement in fabric handle properties depend on the type of the used substrate and follows the decreasing order: polyester/wool > wool.

Table 3 . Effect of Softener micro emulsion (ULTRATEX® MHT CONC. Silicone concentrate) treatment on the printing properties and soft handle properties of pigment prints different colors

Pigment Colors	Substrate		K\S	Incr. in K/S (%)	WF		RF		PF				LF	Stiffness	Surface roughness. (µm)
									Acidic		Alkaline				
					Alt	C	Dry	Wet	Alt	C	Alt	C			
Pigment Red 146	W	UT	11.18	76.65	3	3-4	4	4	4	3-4	3	3	2-3	1207.2	21.61
		T	19.75		4-5	4-5	4-5	4-5	5	4	3-4	4-5	4-5	655.4	11.51
	W/PET	UT	15.34	15.84	3	3	3	2-3	4	4	2-3	4	4	1959.2	18.74
		T	21.17		4-5	4	4	4	4	4-5	4	4-5	4-5	685.2	7.05
Pigment Blue 15:3	W	UT	10.92	29.67	3	2-3	4-5	4	4-5	2-3	4	3	2-3	864.7	20.04
		T	14.16		4	4	5	4-5	4-5	4	4-5	3-4	4-5	536.5	10.95
	W/PET	UT	13.03	26.09	2-3	4-5	4	3	3	4	4	4-5	2-3	2543.6	19.96
		T	16.43		4-5	4-5	4-5	4	4-5	4	4	5	4	903.9	8.15

Pigment printing conditions: Pigment color (20 g/kg); DAICOTHICK 1600 (20 g/kg); Printofix Binder MTB-01 (100 g/kg); ammonium persulfate (2 g/kg); KNITTEX® FEL crosslinking agent (10 g/kg); Softener micro emulsion (20 g/kg); Curing at 1300/4 min using microwave, followed by after –washing at 40˚C for 15 min. in presence of (2 g/L) nonionic wetting agent, K/S: color depth; WF: wash fastness; RF: rubbing fastness; PF: perspiration fastness; LF: light fastness; Alt: alteration; C: staining on cotton; W: wool; W/ PET: wool/ polyester blend; UT: untreated; T: Pigment printed with Softener micro emulsion.

4. Conclusion:

- The improvment in the imparted functionalities namely antibacterial, UV-protection, soft-handle or water-repellency as well as the enhancment in the printability i.e. K/S and fastness properties of obtained wool and polyester/wool prints are achieved by inclusion of TiO2-NP's (10g/Kg), silicone softener (20g/Kg) or the water/oil repellent agent (40g/Kg) as functional additives in the pigment printing formulation, followed by screen printing and thermofixation using at 1300W/4 min.
- The improvement in the aforementioned properties is governed by the kind of additive and the type of substrate.
- Conclusivly, The obtained outcomes have significant practical ramifications for the implementation of concurrent functional finishing and pigment printing in an one step process, taking into consideration both the environmental and economical concerns.

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