



## Impact of Various Treatments on Printing Wool Techniques

Ahmed G. Hassabo<sup>a\*</sup>, Nadeen Khaleed<sup>b\*</sup>, Sohaila Shaker<sup>b</sup>, Neama A. Abd El-Salam<sup>b</sup>, Nourhan A. Mohamed<sup>b</sup>, Nehad Z. Gouda<sup>b</sup>, Abdullah Yahya Abdullah<sup>c</sup> and Hanan A. Othman<sup>b</sup>

<sup>a</sup> National Research Centre (Scopus affiliation ID 60014618), Textile Research and Technology Institute, Pretreatment and Finishing of Cellulose-based Textiles Department, 33 El-Behouth St. (former El-Tahrir str.), Dokki, P.O. 12622, Giza, Egypt

<sup>b</sup> Benha University, Faculty of Applied Arts, Printing, Dyeing and Finishing Department, Benha, Egypt

<sup>c</sup> Department of Chemistry, Faculty of Science and Arts, King Khalid University, Mohail Assir, Saudi Arabia

### Abstract

The aims of this research study multifunctional printing techniques on wool fabrics by using textile printing processes which are categorized as direct printing and indirect printing. Direct printing includes screen printing by using natural, synthetic dyes and pigment printing by using functional additives such as TiO<sub>2</sub>- nanoparticles which lead to multifunctional finishing wool. It also includes digital inkjet printing by utilizing air plasma as a pretreatment, Eco-Friendly pretreatment by using H<sub>2</sub>O<sub>2</sub> and papain, and by using different thickeners. Indirect printing includes discharge and resists printing. All these techniques lead to enhance printability, the functionality of wool, and the fastness properties of all wool prints.

**Keywords:** wool fabrics, direct printing, indirect printing, air plasma, TiO<sub>2</sub>- nanoparticles, eco-friendly pretreatment.

### Introduction

Wool is a complex multi-cell system composed of inanimate cells which vary in composition, shape, and properties[1]. Wool fibers are widely utilized in the textile industry because of their outstanding qualities, which include luster, softness, warmth, and biodegradability. Keratin, a fibrous protein, is the chemical substance of wool fibers[2]. Wool and other keratin fibers are protein-based fibers made up primarily of carbon, hydrogen, oxygen, nitrogen, and sulfur[3]. Proteins are the main component of wool fiber. Wool fibers contain a variety of amino acids as well as sulfur-containing groups, lipids, mineral salts, nucleic acid residues, and carbohydrates. Sulfur forms a disulfide connection between two of its atoms, which serves as the main cross-linking agent between the components of wool. The high concentrations of lipids and carbohydrates influence fiber function and properties[4]. Wool fibers are made up of around 20 different types of amino acids that are distinguished by their side chains. Wool's polypeptide chain is made up of 20 amino acids that contain amino (-NH<sub>2</sub>), imino (=NH), hydroxyl (-OH), carboxylic (-COOH), and thiol groups (-SH). These functional groups in the fiber, as well as the amphoteric character of wool, have an impact on its

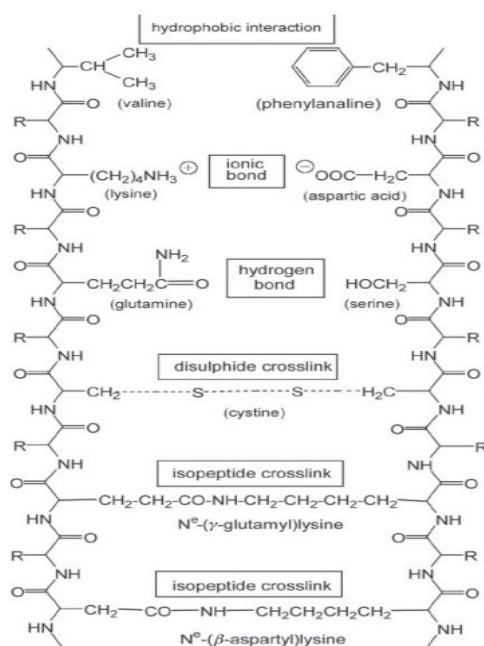
processing, dyeing, and printing. Raw wool fibers have low absorptivity and a tendency to feel (particularly fine wool fibers) due to their chemical and structural complexity. The amino acids are distinguished by their acidic and basic structures (e.g., aspartic acid and histidine), the presence of hydroxyl groups as the side chain (e.g. Serine), the presence of sulfur (e.g. cysteine), and non-existence of reactive groups as the side chain (e.g. glycine)[3]. They impart different features such as hydrophobic, hydrophilic, acidic, or basic. The sequence of amino acids defines the possibility of intermolecular links, the molecular cohesion of keratin, and access of amino acids to chemical reactions [2]. The fiber elements having oppositely charged side chains are linked through salt bridges which can be stated as the second kind of cross-linking[3]. There is also one more type of linking, the isopeptide bond, that is recognized as a third of cross-linking and formed between a glutamic or an aspartic acid and a lysine residue[1, 3]. Wool fibers also include hydrogen bonds which are formed between amide and hydrogen-related groups as shown in **scheme (1)**. [5, 6]

\*Corresponding author Ahmed G. Hassabo, E-mail: aga.hassabo@hotmail.com, Tel. 01102255513

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**Scheme 1. The chemical structure of wool**

Textile printing is defined as a controlled method of painting cloth in specified patterns or motifs using specialized printing techniques and machinery[7]. It is also the process of drawing or generating a color pattern on textile materials[8, 9]. The color is linked to the fiber in properly printed fibers to protect against washing and crocking. Localized dyeing is a term used to describe textile printing[10, 11]. There are two types of traditional textile printing processes: direct printing and indirect printing. Screen printing, block printing, burn-out printing, digital printing, digital inkjet printing, and transfer printing are examples of direct printing, while resist printing and discharge printing are examples of indirect printing[7, 12-14]. Here in this review, we will focus on the printing of wool fabrics in these different techniques[15].

### Direct printing

Direct printing is the most popular method of adding a color pattern. It is often known as 'print-on', and it is the most common and easy printing technique on fabrics in the modern industry[16]. Originally, the term "direct" meant that there had been no preliminary mordanting or dyeing procedure. Printing pastes come into direct touch with the cloth surface without any additional processing adjustments.[17, 18], and the dyes used in dyeing a fiber can theoretically be utilized in printing on the same fiber[7]. But Pigment is the most commonly used colorant in textile direct printing and covers approximately 75-80 % of the total of all printing processes as it is a simple and low-cost approach that does not require a lot of equipment, do not require washing, and produce just a minor quantity of waste[10, 15, 19-22].

### Screen-printing

In this study, the suitability of natural dye (*Juglans regia*) from walnut husk in printing Alpaca wool hand-knitted samples using a hand screen-printing technique was investigated. The biological pigment was extracted by boiling 150 g of herbal source in 5 L of distilled water (pH 6.83) at 100°C for 1 hour, with the addition of 10 g/L sodium chloride. After leaving the solution at an ambient temperature for 2 days, this was filtered and the residual water was evaporated by drying at 100°C in a laboratory dryer for 12 hours, obtaining biological pigment crystals to be used for the printing paste preparation[23].

For the printing paste, concerning the requirements of the substrate and the color characteristics, two different basic printing paste compositions Bath (1) is a printing paste without a mordanting agent while in Bath (2) iron sulfate (FeSO<sub>4</sub>) was used as the mordant. were defined, as shown in **Table 1**. Dextrin-starch derivate-based thickener (British Gum) was examined in dry matter contents of 4% and 8%, and was tested at three different pH values, 4 (acidic, adjusted with Acetic Acid), 7 (neutral) and 9 (alkaline, adjusted with Sodium Carbonate). The rheological properties of the thickeners were defined by their viscosity, fluidity, and elasticity. The printing process was conducted by manual screen printing. After that, the steam-fixing procedure at atmospheric conditions (105°C) was applied for 30, 45, and 60 minutes. After steaming, the samples were rinsed in warm and cold water[23].

**Table 1. the printing paste bath**

Chemical	Paste recipe (1)	Paste recipe (2)
Dye (g)	0.50	0.50
Urea (g)	0.08	0.08
Glycerol (g)	0.08	0.08
Thickener (g)	5	5
Mordant (FeSO <sub>4</sub> ) (g)		0.20

As for the printing pastes based on 8% and 4% dry matter content thickener, the uniform color yield on knitted sample surfaces, as well as a higher content of dye penetration into a yarn structure, were obtained for samples printed with pastes containing 4% dry matter content, so the thickener with 4% of dry matter content was confirmed as being optimal for the given characteristics of fiber and textile substrate. A certain influence of pH is observed for samples printed with a paste containing a 4% dry matter content thickener. For samples printed without metal ions (iron sulfate), the change of pH, and the range of shades from yellowish to reddish brown is obtained. The results also confirmed that the British Gum thickener with 4% dry matter content and a pH of 4 gives the optimal results when printing on

specific knitwear due to its rheology properties. Maximum fluidity was achieved in acidic pH, while the viscosity and the elasticity decrease in acidic pH[23]. The fixing duration was optimized at 45 minutes. With a duration of 60 minutes, a certain instability of the printing paste occurred. It was confirmed that the optimal fastness properties are achieved at pH 4 with a fixation time of 45 minutes. The highest lightness fastness and optimal rubbing fastness were obtained for the sample printed with a paste containing 4% dry matter-based thickener that was fixed for 45 minutes. The highest fastness was obtained for printing paste containing 4% dry matter thickener at pH 4 and mordanting agent with an optimal fixing time of 45 minutes. Based on these conclusions, natural herbal dyes extracted from walnut green shells can be used for wool knitwear printing, obtaining an ecologically acceptable product with satisfactory fastness properties[23].

Another study of Printing Wool Fabric Using Selenium Nanoparticles was applied by screen printing technique. Selenium Nanoparticles (Se-NPs) were prepared through redox reaction according to the method described by Malhotra et al. with an improved modification. Sodium hydrogen selenite was utilized as a precursor for Se-NPs with different concentrations (10- 100 mM). It was added to vitamin C at various concentrations (10-100 mM) in the ratio of 1:1 and at the same concentration under magnetic stirring. Se-NPs stability was maintained using PVP (polyvinylpyrrolidone) dissolved in vitamin C solution (0.3-3 g/100 ml). The color converted from colorless to dark orange[24, 25].

The Se-NPs printing paste was prepared according to the formulation as shown in **Table (2)**. The homogenized Se-NPs-based printing paste was applied to wool fabrics using a screen printing technique, then the printed samples were fixed at 98 °C for 6 min, finally, samples were washed off with non-ionic detergent at 60 °C for 10 min, and material-to-liquor ratio at 1:50[24].

**Table 2. Se-NPs-based printing formulation**

Printing paste components	g/kg paste
Sodium alginate (15 %)	500
Urea	70
Acetic acid (30 %)	50
Se nanomaterial	20
Water	360
Total	1000

The obtained results revealed that the printed wool fabrics have a stable bright color ranging from light orange gradually to dark orange with the concentration of synthesized Se-NPs with additional functionalities. The Se-NPs printed wool exhibited outstanding fastness properties and antimicrobial activity with excellent durability towards washing after 10 washing cycles. Moreover, the obtained

prints effectively blocked the UV radiation providing excellent UV protection. The highest results were listed in **Table (3)**. For all the printed samples, the alteration of color during washing showed very little color change (rating 4/5), and no staining was noticed on wool (rating 5). The rubbing fastness generally showed a very good rating. Dry rubbing fastness was observed to be a little better than the wet one, and the light fastness test showed a very slight color fading rating of 4/5. This revealed the efficiency of the proposed procedure to figure out an excellent coloration process without using any other chemicals (e.g. crosslinkers, binder, or coating materials). The fastness properties and the color strength (K/S) of the obtained Se-NPs wool prints were still high even after 10 laundering cycles. It means that SeNPs were still loaded and fixed onto simultaneously functional printed fabric surfaces [24].

**Table 3. K/S values of Se-NPs printed fabric and Fastness properties**

Se-NPs conc.	K/S	Washing	Rubbing	Light
50mM	7.5	4-5	4-5	4-5

Another study is directed towards pre-modification of the wool component of wool/polyester blend fabric using monochlorotriazinyl  $\beta$ -cyclodextrin (MCT- $\beta$ CD) for attaining high-performance union disperse prints with outstanding UV-protection functions using the screen-printing technique[26].

Pretreatment with MC- $\beta$ CD as Fabric samples were impregnated by the aqueous solution containing the MCT-b-CD (0–80 g/L), modified dimethylol dihydroxy ethylene urea (DMDHEU) (20 g/L), PEG-600 (10 g/L) and catalyst (0–7 g/L), roll squeezed, and thermofixed at 100–130°C for 5 min, thoroughly washed to remove unreacted and any soluble byproducts and finally dried before being printed. Then Portions of untreated and MCT-b-CD loaded fabric samples were dispersed and printed using the flat screen technique and the print paste formulation noted in **Table (4)**. Printed fabric samples were then dried at 85 °C for 5 min and steam fixed at 140 °C for 30 min then rinsed thoroughly, soaped for 15 min at 50 °C in the presence of 2 g/L (nonionic wetting agent and detergent-BASF), then rinsed well and finally dried at 85 °C for 5 min[26].

**Table 4. Disperse printing paste**

Content	G/kg paste
Disperse dye	30
Thickener (5%)	700
Acetic acid (30%)	20
Water	250
Total	1000g

It's concluded that the inclusion of the MCT- $\beta$ -CD, with its hydrophobic cavities, onto the wool/polyester blend fabric was found to exhibit an outstanding increase in the printability with disperse dyes reached K/S (20.59) along with a remarkable improvement in the fastness properties of the obtained solid-shade prints[26]. This enhancement in the printing properties is a direct consequence of modifying the ability of wool components to form solid inclusion complexes (host-guest complexes) with the vapors of the sublimable disperse dyes during the steam fixation step. The investigation reveals that printing of wool/polyester blend, deeper depth of shades along with better fastness properties are achieved if MCT- $\beta$ -CD (60 g/ L), DMDHEU (20 g/L), citric acid (5 g/L), and PEG-600 (10 g/L) are used in the pre-modification step followed by thermofixation at 120 °C 15 min and subsequent disperse printing according to the selected printing conditions. As a result of the inclusions, along with the subsequent improvement in the depth of shades, regardless of the used disperse dye, the UV-blocking functions of the obtained disperse prints were significantly increased[26].

### Pigment printing

The present study focuses on improving the pigment printing properties and functionalization of wool and polyester/wool (50/50) fabrics such as antimicrobial properties, UV-protection, soft handle, and water/oil-repellency in one step through the inclusion of functional additives namely TiO<sub>2</sub>-nanoparticles, silicon micro-emulsion or a water/ oil-repellent agent into pigment printing paste then printing and microwave fixation[27]. The 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 as present in (Table 5):

**Table 5. print paste**

print paste content	g/Kg paste
Pigment color	20g
Binder	100g
Thickening agent	20g
crosslinking agent	10g
Ammonium persulphate(NH <sub>4</sub> ) <sub>2</sub> S <sub>2</sub> O <sub>8</sub>	2g
Functional additives:	
Water & oil repellent	0-60g
Silicon-softener	0-30g
TiO <sub>2</sub> -NP's	0-20g
H <sub>2</sub> O	Xg
Total	1000g

### Effect of TiO<sub>2</sub>- nanoparticles, silicone softener, and water/oil repellent concentrations on the depth of the obtained wool and polyester/wool prints

Increasing TiO<sub>2</sub>-nanoparticles concentration up to 10 g/Kg paste, water/oil repellent agent concentration up to 40g/Kg, and silicone softener up to 20g/Kg leads to a significant increase in the K/S values of obtained wool and polyester/wool pigment prints[28].

This improvement in K/S values may be attributed to TiO<sub>2</sub>-NP's impact as a cocatalyst 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 and by using water/oil repellent and silicone softener polymer film formed 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[28].

further increase above the concentration of functional additives which have been mentioned, leads to a negative impact on the depth of shade for the printed samples expressed by a decrease in the K/S values[28].

### Effect of TiO<sub>2</sub>-NPs treatment on the printing properties

Pigment color	Substrate	K/S	K/S (%)	WF	RF	PF	LF	Water/oil repellent	Surface roughness (μm)
Pigment red 146	W/PET (T)	21.98	38.07	3-4	4-5	4-5	4-5	50(water) 4 (oil)	14.12

### Effect of water/oil repellent treatment on the printing properties

Pigment color	Substrate	K/S	K/S (%)	WF	RF	PF	LF	stiffness	Surface roughness (μm)
Pigment red 146	W/PET (T)	21.17	15.84	4-5	4	4-5	4-5	685.2	7.05

### Effect of silicone softener treatment on the printing properties

Pigment color	Substrate	K/S	K/S (%)	WF	RF	PF	LF	UPF	Antimicrobial Activity ZI (mm)
Pigment red 146	W/PET (T)	20.08	30.89	4-5	3-4	4-5	5	73.08	19.5

Conclusively, the water/oil repellent treatment gives higher K/S regardless of the other properties. The obtained outcomes have significant practical ramifications for the implementation of concurrent functional finishing and pigment printing in a one-step process, taking into consideration both environmental concerns[27].

### **Digital inkjet printing**

Digital inkjet printing as a new environmental technology has been initially applied in the textile industry. It has unique superiorities such as low cost, high precision, and celerity compared with traditional printing which was limited to producing high-quality and personalized products[29]. Among all kinds of dye-based inks, reactive dye ink accounts for an increasing proportion. It has the advantages of bright color, comprehensive color range, good level dyeing, and high fastness, and it can form covalent bonds with both cotton and wool fabrics[30]. The scales on wool fibers caused frictional effects to be responsible for wool felting and prevented the penetration and wicking of dye, resulting in poor printing imaging and low color yield[31]. Therefore, there is a huge potential for printing patterns on wool fabrics through digital inkjet printing technology. Digital inkjet printing on high-class wool textiles is in line with the trend, but the challenge such as reducing the impact of wool scales on print quality is still worth of attention.

### **Digital Inkjet Printing on Wool Fabrics by utilizing Eco-Friendly Pretreatment**

In this study, a novel environment-friendly method was applied to wool inkjet printing pretreatment without any generation of hazardous substances. H<sub>2</sub>O<sub>2</sub> and papain enzymes were adopted to pretreat wool fabric before the inkjet printing process. This work provided a promising direction for decreasing the discharge of harmful substances and improving the utilization rate of dye solutions to achieve cleaner production[32].

Pretreatment of wool fabrics: The wool fabric (2 g) was incubated in a solution containing H<sub>2</sub>O<sub>2</sub> (50 ml/l) and Surfactants aeo-9 (1 g/l) in a shaking bath at 30 °c for 2 h. Finally, the pretreated wool was washed with deionized water and dried at 60 °c. papain-treated wool fabric. The wool fabric (2 g) was treated for 1 h in a 60 ml solution of papain (0.12 g) and aeo-3 (1 g/l) at 50 °c. Then, the fabric was dipped in boiling water for 2 min to deactivate the enzyme and finally washed with deionized water and dried at 60 °c. H<sub>2</sub>O<sub>2</sub> and papain synergetically

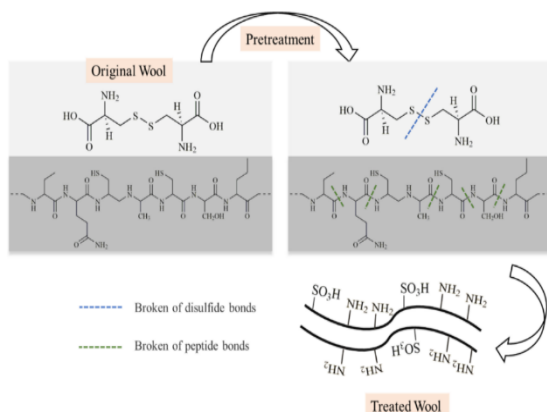
treated wool fabric. The wool fabric (2 g) was first treated with H<sub>2</sub>O<sub>2</sub> and then with papain, then both of them. During the combined treatment process, H<sub>2</sub>O<sub>2</sub> first attacked the disulfide bonds of surface keratin and then provided more possibility for papain to break the amide bonds. As a result, the synergistic effect of H<sub>2</sub>O<sub>2</sub> and papain could effectively decrease the scales, which would improve the inkjet printing quality[32].

Digital inkjet printing ink was prepared using Reactive Red 218 dyes and ultrapure water. The concentration of the dye solution was 50 mmol/L and was filtered by a 0.22 μm filter film before use. The inkjet printing processes and droplet formation were achieved using an inkjet printer with a nozzle diameter of 30 μm. A stable droplet formation was obtained by adjusting the waveform. The droplet diameter was 30 μm, which was the same as the nozzle diameter. Wool fabrics were attached to a platform, and they can move with the platform in front-back and right-left directions. The final printed pattern was a square with 2 cm side lengths[32]. the K/S values of four printed wool samples were measured and are given in **Table (6)**.

**Table 6. Color Parameters and K/S Values of the Inkjet Printed Wool Fabrics Treated with Different Methods**

<b>Inkjet printing wool fabrics</b>	<b>K/S</b>
Untreated	20.54
H <sub>2</sub> O <sub>2</sub> treated	21.63
Papain treated	22.16
H <sub>2</sub> O <sub>2</sub> -papain treated	25.30

It is concluded that H<sub>2</sub>O<sub>2</sub>-papain-treated wool fabrics showed a more vivid color. It is clear that the K/S value of the treated fabric greatly increased and the combined treated wool fabric had the higher K/S value in comparison with other printed wool samples. For the original wool samples, hydrophobic lipids and the disulfide bonds of surface keratin formed dense structures which made it difficult for dye molecules wick into fibers through diffusion. After pretreatment, dye molecules jetted from inks diffused into wool fibers without the existence of scales and bonded with wool as noted in **scheme (2)**. In consequence, high-quality and color strength images were obtained. This is owing to the increase of zeta potential and better wettability on the surface of wool fabric. As a result, in the steaming process, that treated wool fabrics displayed much darker color compared with other wool samples. It is the synergy between H<sub>2</sub>O<sub>2</sub> and papain that removed most of the scales resulting in better wettability[32].



**Scheme 2. Proposed mechanism for the combined pretreatment of wool fabric**

### Digital inkjet printing by utilizing air plasma pretreatment

Another study of treating the wool fabric with air plasma pretreatment, and performed the ink-jet printing process using reactive dye inks[33].

Atmospheric air plasma is a kind of dry ecological processing technology with the advantages of low cost, functionalization, and being environmentally friendly. It has been widely used in many production fields, especially textile pretreatment and finishing in recent years[34]. Textile modification by plasma technology was mainly achieved by surface etching and grafting chemical functional groups[35].

Untreated raw wool was used in the ink-jet printing of this experiment. Reactive red 218 dye and reactive orange 13 dye, Urea (CH<sub>4</sub>N<sub>2</sub>O), sodium alginate (C<sub>6</sub>H<sub>9</sub>NaO<sub>7</sub>), and Ammonium sulfate were used as sizing agents while ethylene glycol (C<sub>2</sub>H<sub>6</sub>O<sub>2</sub>) as a solvent. Then the wool fabric was treated by a plasma generator for 1 min, 2 min, 3 min, and 4 min (dielectric barrier discharge, the electrodes distance: 2 cm, discharge voltage: 260 V). To make the treatment effect more uniform, compressed air was used in the experiment. The treated samples were washed in cold water for 1 min and dried at 70 °C in the oven. Ink-jet printing process occurred which include the dye solution of reactive dye (50 mmol/L) and solvent (the ratio of ethylene glycol to ultrapure water was 3:7). The wool fabrics were treated by ink-jet printing equipment with R-218 dyes and O-13 dyes (printing frequency was 600 Hz; print nozzle diameter: 30 μm). The samples were steamed for 30 min, washed in soap and cold water to remove unfixed dyes and impurities, and then dried at 70°C. [33].

The color parameter values of all printing samples were calculated, and the color difference values were between 0.3 and 0.6, within the requirements of practical application. After plasma treatment, the K/S values of printed wool fabrics with two reactive dyes showed similar variations. In the case of O-13 dye, the best color intensity could reach to K/S value (22.34) after plasma treatment for

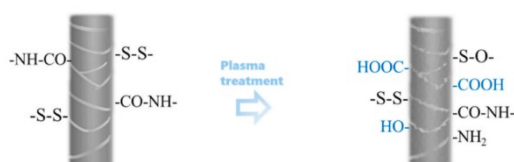
3 min. Compared with untreated wool fabric (14.64), the K/S value of treated samples increased by 52.59%, which indicated that printed images can be attained with higher color intensity. With the extension of the treatment time, the wool surface scale layer was etched and scales became soft making the fibers wet and swell easily during ink-jet printing. The breaking of C–N in the amide bond increased the content of –NH<sub>2</sub>, and the binding sites between reactive dye and wool fabric enhanced. It was easy for the dyes to diffuse into the fiber, and form covalent bonds, increasing the apparent color yield. The essential reason was that the amide bond of cystine in the scale layer was destroyed, which led to the destruction of the dyeing barrier of wool[33].

To explore the influence of plasma treatment on wool fabric physical properties, the tensile strength of untreated wool treated for 4 min was tested. The tensile strength of untreated wool was 57.51 N. After plasma treatment, strength was reduced to 48.55 N, elastic modulus decreased from 16 MPa to 13.72 MPa, which suggested that the plasma pretreatment did not significantly affect the strength of the wool fabric and that the wool fiber matrix was not seriously damaged. The plasma treatment is a feasible pretreatment method for ink-jet printing of wool fabric[33].

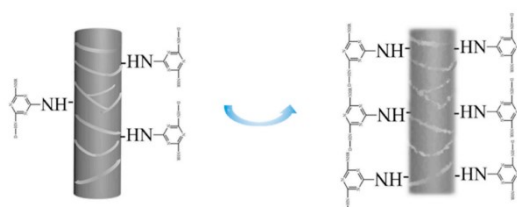
Colorfastness is very important for ink-jet printing products. the dry and wet rubbing fastness of untreated wool fabric was grade 4 and grade 3, respectively, both of which were improved after plasma treatment. The treated wool fabric became more hydrophilic and had more binding sites with reactive dye molecules. Under the same friction conditions, fewer dye molecules were removed, resulting in improved rubbing fastness. In the case of reactive R-218, the staining and discoloration fastness of untreated wool fabric were grade 4–5 and grade 3, after plasma treatment, the staining fastness does not change, indicating that the treated fabric can still maintain its original washing fastness, and the fastness to discoloration was also improved. Plasma treatment endowed the wool fabric with better practicability[33].

After plasma treatment, the hydrophilicity of wool fabric was significantly improved, and the permeability of dye in wool fabric became higher. The K/S value of wool fabric back reached the maximum by treating for 4 min, so the permeability achieved the highest (22.40%), which was responsible for the reduction in K/S of wool fabrics with further treatment. The mechanism of plasma treatment to improve wool fabric reactive ink-jet printing performance was shown in **scheme (3)** The surface of untreated wool fabric was covered by a tight scale layer that was rich in disulfide and amide bonds, resulting in poor hydrophilicity, which was not conducive to ink-jet printing. The surface scale layer was etched and C–C, C–H, and C–N bonds were broken by plasma treatment, the content of –

NH<sub>2</sub> improved, the reaction sites of wool fabric with reactive dyes increased by air plasma as shown in **scheme (4)**. Due to the bombardment of high-energy particle groups, oxygen-containing functional groups such as –COOH and –OH were introduced to the fabric surface, and the wettability of the fabric was significantly enhanced. In conclusion, plasma treatment endowed the wool fabric with excellent hydrophilicity, enhanced ink-jet printing performance, and achieved a higher K/S in a low time[33].



**Scheme 3. Improving mechanism of plasma treatment on wool fabric ink-jet printing performance.**

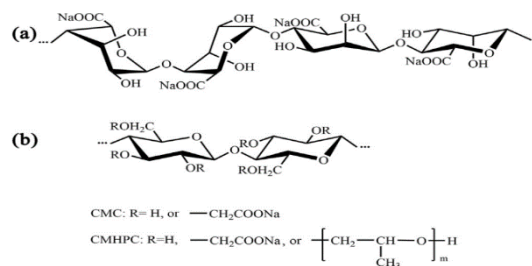


**Scheme 4. Change the reaction sites of wool fabric with reactive dyes.**

Another study is to further enhance the image quality of inkjet-printed wool fabrics by cellulose derivatives and to reveal the relationship between the rheological properties of the pretreatment solution and inkjet printing performance. Given that the rheological properties of the thickeners are closely related to their molecular structure SA, CMC, and CMHPC with different molecular structures as shown in **scheme (5)** were used to treat wool fabrics, and their performance was compared[36].

SA is the most commonly used polysaccharide for fabric pretreatment as a thickening agent which exhibits high viscosity and good film-forming ability due to its good solubility and poor reactivity with dyes[37]. However, because of a limited supply of SA and the demand for high-quality inkjet printing products, many studies are focused on alternatives to SA. Water-soluble cellulose derivatives containing carboxymethyl groups or hydroxypropyl groups have attracted significant interest owing to their widely available sources and poor affinity with dyes[38]. Wool treated with 3% CMC (sodium carboxymethyl cellulose) exhibited good color yield and clarity after inkjet printing with reactive dye inks. Furthermore, excellent color fastness was obtained by CMC-treated wool fabrics when printed with modified Drimarene K dye-based inks. High-substituted hydroxypropyl cellulose (H-HPC) was mixed with other thickeners, such as SA and CMC, to treat fabric for desired digital transfer printing performance with

reactive dyes. CMHPC (carboxymethyl hydroxypropyl cellulose) is a cellulose derivative containing both carboxymethyl groups (–CH<sub>2</sub>COONa) and hydroxypropyl groups (–CH<sub>2</sub>CH(OH)CH<sub>3</sub>), which has been widely used. It combines the characteristics of two types of cellulose derivatives and thus shows good thickening, thixotropy, and stability. Therefore, it has good application prospects in inkjet printing[36].



**Scheme 5. Chain conformation of SA (a), CMC, and CMHPC (b).**

First the pretreatment, wool fabric was treated with protease enzyme (2% polysaccharide) to facilitate the deposition of the treatment solution. then 15 g of urea and 2 g of ammonium sulfate were dissolved in 81 g of deionized water. Then, the mixture was divided into three equal parts. Subsequently, 2 g of SA, CMC, and CMHPC were added to the three parts of the mixture, respectively. The mixtures were stirred evenly and allowed to stay overnight. Finally, a two-dip, two-nip process was used to pad the pretreatment solution on the protease-treated wool fabrics for (80 ± 1) % fabric pickup. The fabrics treated with SA, CMC, and CMHPC were dried at 60 °C. Additionally, the effects of polysaccharide concentration (0.5%–2.5%), urea dosage (2%–25%), and pH of the treatment solution (4–11) on inkjet printing. Inkjet printing process A high-speed digital printing machine was applied to jet cyan ink droplets on the wool surface at a resolution of 600 × 600 dpi. Then, the printed samples were dried (60 °C) and steamed in a steamer (102 °C, 10–115 min). Subsequently, the specimens were thoroughly washed and soaped to remove the auxiliaries and unreacted dyes[36].

The results indicate that CMHPC with pronounced shear-thinning properties and elasticity is more suitable for reactive inkjet printing of wool fabrics. Furthermore, high color strength can be obtained by CMHPC-treated wool fabrics with lower CMHPC concentration, less urea consumption, and shorter steaming time[36]. The color strength was reported that the CMHPC-treated sample obtained the highest K/S value of 28.4. This implies that the CMHPC-treated fabric exhibited the darkest and most vivid colors. However, their maximum absolute values were obtained after CMHPC treatment, thereby signifying that the purest color was obtained by CMHPC-treated fabric because cyan is a mixture

of yellow and green. **Table (7)** also shows that CMHPC-treated fabrics exhibited the lowest permeability of 2.4%, indicating that the ink droplets tended to be fixed on the surface of the fabric rather than penetrating the back of the fabric. Conversely, SA-treated fabrics obtained the poorest color performance. Furthermore, the color performance of the CMC-treated fabrics was between those of CMHPC and SA-treated fabrics. These data are consistent with the results of ink drop spread and penetration. This further proves the close relationship between the rheological properties of the treatment solution, film structure, and inkjet printing performance[36].

**Table 7. Colorimetric values of the inkjet-printed wool**

Treatment	$h^\circ$	K/S	Permeability (%)
SA treated	217.7	21.0	6.7
CMC treated	218.0	24.5	4.1
CMHPC treated	218.1	28.4	2.4

### Indirect printing

It is the process of dyeing the fabric first, after which a design is printed using a discharge or resist agent that dissolves the dye where the design is located. In this process, the pre-treatment processes for wool could include scouring and bleaching, followed by acid dyeing, which is a common practice for wool. The discharge paste, which usually contains a reduction agent (such as sodium hydrosulfite or thiourea dioxide) and a thickener, is applied to the cloth after it has been colored. The design area loses color as a result of the reduction agent's breakdown of the dye molecules there, leaving a white or light-colored pattern on the colored background. Resist printing and discharge printing are categories for indirect printing.

### Discharge Printing

In discharge printing, A pre-dyed cloth is printed with a paste containing a reducing (discharge) agent and a dye (the illuminating color) that is resistant to reduction in discharge printing, as illustrated in Table (8). In the printed portions, the ground shade is simultaneously eliminated and replaced by the 'illuminating' color. The production of intricate designs, usually on sharply contrasting grounds, with exceptional clarity, sharpness, and fit has become the trademark of this style, which can only be accomplished with extreme effort using direct printing methods[39-41]. The original dye in the printed areas is chemically destroyed during discharge printing. Ground-shade dyes are invariably azo dyes, with discharge ability ratings of at least 4-5 according to the Color Index system. Many acid, metal-complex, and reactive dyes are dischargeable to white, thus choosing them is often not an issue. Because the most readily reduced component is discharged first, it is possible that a less readily

reduced component, even if present in modest quantities, will be poorly discharged under adverse conditions[39, 42].

Formaldehyde sulphonylates are the most often used reducing agents in the discharge printing of wool. Sodium formaldehyde sulphonylate is a water-soluble substance that produces nice white discharges and is easy to handle. It is stable in alkaline print pastes but not in acidic environments; it reaches its full redox potential when the print is steamed, resulting in significant fiber damage and shrinking unless extreme caution is applied. As a result, it is rarely used on its own; pH correction leads to decreased stability[39].

Calcium formaldehyde sulphonylate is water-insoluble; although stable, it can lead to problems of screen blockage and unsatisfactory penetration of the discharge. BASF has subsequently developed a 30% dispersion of the compound; greater stability, fewer problems with haloing, less fiber damage, and less shrinkage than with the sodium salt are claimed. Calcium salt is quite often used in combination with sodium salt[39].

**Table 8. The printing paste for discharge printing of wool**

Chemical	Quantity (g dm <sup>-3</sup> )
Dye	x
Urea	30-50
Thiodiethylene glycol	30-50
Water	y
Sodium/zinc/calcium formaldehyde sulphonylate	30-180
Thickening	500
Sodium <i>m</i> -nitrobenzenesulphonate (Revatol S, Ludigol, Matexil PA-L)	5-20
Ammonium chloride	5-20
Zinc oxide 1 : 1	20-50
Water to bulk to	1000

### Resist Printing

Resist printing produces similar effects to discharge printing, but the processing method is different. The resist agent is printed and placed on the white cloth, preventing the ground shade, which is applied using a suitable dyeing or printing procedure, from being fixed. A colored resist is created by incorporating an illuminating dye in the print paste that can fix in the presence of the resist agent. There are two types of resist techniques:[22]

Reactive resistance is followed by piece dyeing, usually from long liquor.

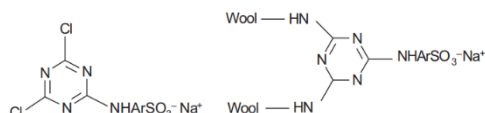
Mechanical/chemical resist, where the resist agent is printed and the ground shade is applied by wet-on-wet printing with a blank screen.

Many attempts have been made over the years to produce multicolored effects on wool fibers, with varying degrees of success.



### Chemical Resist Processes

Several methods have been suggested for rendering wool non dyeable with acid dyes, the first practical proposition being the use of a reactive resist agent resembling a colorless reactive dye. One such product was (Sandospace R)[43], which is a highly reactive water-soluble anionic product, as noted in the **scheme (6)** by which this compound can react with amino groups in the wool, to produce **scheme (7)**. The resistance thus arises both from the blocking of reactive amino sites on the fiber and by anionic repulsion.



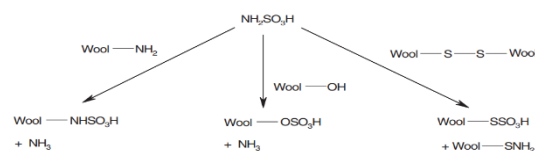
**Scheme (6-7). the chemical reaction between (Sandospace R) dye and wool**

This product's use to create resist effects on wool became well-established, and it also found some use in the printing of wool garments. When Sandospace R is padded or long-liquor dyed onto wool, treatment levels corresponding to mass increases of 10-12% result in a resist effect of roughly 80-90% when the goods are dyed to a 2% shade. A perfect white resist is attained at pale depths. Sandospace S is less reactive than Sandospace R because it is most likely a monofunctional chloro-s-triazine and cannot be properly attached to wool to produce the mass gains required for effective resists[43].

### Sulphamic Acid Resist Printing

The majority of the techniques discussed so far have one major flaw: the difficulty to generate a flawless white resist except in pale depths of ground shade. These disadvantages prompted the invention of a more effective wool-resist technology. Sandoz patented the use of sulphamic acid as an anionic dye-resist agent for wool in 1955. It has been observed that this compound has dye resistance when applied using cushioning procedures. Bell et al. were the first to report its usage in wool printing, and the resist was discovered to be especially effective against reactive dyes. According to Elliot et al., the most likely reactive sites on wool for sulphonation with sulphamic acid include basic amino groups, serine hydroxyl groups, and cystine linkage. As shown in **scheme (8)**, the reactions between sulphamic acid and wool have been thoroughly researched, as have the optimum reaction conditions. For maximum reaction with sulphamic acid, urea is essential in the pretreatment liquor/print paste. the fabric is cured at 150–160 °C for 4–5 minutes; under these conditions, a maximum uptake of 8.5% bound sulphamic acid is achieved. No further uptake is achieved by increasing either the curing time or the temperature. The conditions employed in printing are very similar as shown in **Table (9)**. A colored resist is achieved by including

an illuminating dye in the print paste; only reactive dyes, selected from the Lanazol and Drimarene R and K ranges, fix covalently to the fiber under these conditions; these may be added directly to the white resist paste as solids. two-step of fixation process, involving: dry heat fixation, 4–5 minutes at 150–160 °C, to fix the sulphamic acid, followed by; steaming, 30 minutes at 100–102 °C, to fix the illuminating dye[44].



**Scheme 8. Sulphonation of wool with sulphamic acid**

**Table 9. Printing paste of white resist**

Chemical	Quantity (g dm <sup>-3</sup> )
Sulphamic acid	150–200
Urea	150–200
Thickener 301 Extra RF 12% (Grünau) or Indalca PA40 Cesalpinia)	500
Alcopol 650 (Huntsman) (non-ionic wetting agent)	2
Antifoam	1
Water to bulk to	1000

### Reactive-Under-Reactive Resist

The technology utilized for resist printing on cellulosic fibers is utilized in this chemical resist procedure for chlorinated wool. The illuminating colors are generally monochloride-triazine reactive dyes printed from a mildly alkaline print paste containing sodium sulfite (pH 7.6 with sodium acetate); the ground shade is printed wet-on-wet with vinyl sulphone (VS) dyes printed from a mildly alkaline paste (pH 7 with caustic soda). After reasonable drying, the print is steamed for 20 minutes at 102 degrees Celsius and washed off in ammonia at 50 and 70 degrees Celsius. The presence of sodium sulfite, which reacts quickly with the VS group, prevents the 'ground-shade' print from appearing in the printed areas. Under these conditions, The monochloride-s-triazine illuminating color is capable of repairing [39].

### Conclusion

Conclusively, the different treatments of printing wool fabrics techniques led to enhancements in the printability, and from all these studies the results indicate that CMHPC-treated wool obtained the highest K/S value of 28.4

The improvement in the fastness properties is governed by the kind of additive and the type of substrate.

The improvement in the imparted functionalities such as antibacterial, UV protection, soft-handle, or water-repellency.

The obtained outcomes have significant practical ramifications for the implementation of concurrent functional finishing and printing in a one-step process and using eco-friendly pretreatment, taking into consideration both the environmental and economic concerns.

### Conflicts of interest

There are no conflicts to declare

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There is no fund to declare

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## تأثير المعالجات المختلفة على تقنيات طباعة الصوف

أحمد جمعه حسبو<sup>1</sup> \* ، نادين خالد<sup>2</sup>، سهيلة شاكر<sup>2</sup>، نعمة عبد السلام<sup>2</sup>، نورهان محمد<sup>2</sup>، نهاد جودة<sup>2</sup>، عبد الله يحيى عبد الله<sup>3</sup> وحنان علي عثمان<sup>2</sup>

<sup>1</sup> المركز القومي للبحوث (60014618 ID Scopus) ، معهد بحوث وتكنولوجيا النسيج ، قسم التحضيرات والتجهيزات للألياف السليلوزية - الجيزة - مصر  
<sup>2</sup> جامعة بنها - كلية الفنون التطبيقية - قسم طباعة المنسوجات والصباغة والتجهيز - بنها - مصر  
<sup>3</sup> قسم الكيمياء، كلية العلوم والآداب، جامعة الملك خالد، محابيل عسير، المملكة العربية السعودية  
 \*المؤلف المراسل: البريد الإلكتروني [aga.hassabo@hotmail.com](mailto:aga.hassabo@hotmail.com) :

## الملخص

يهدف هذا البحث إلى دراسة تقنيات الطباعة متعددة الوظائف على الأقمشة الصوفية باستخدام عمليات طباعة المنسوجات التي تصنف على أنها طباعة مباشرة وطباعة غير مباشرة. تشمل الطباعة المباشرة طباعة الشاشة باستخدام الأصباغ الطبيعية والاصطناعية وطباعة الأصباغ باستخدام إضافات وظيفية مثل  $TiO_2$  - الجسيمات النانوية التي تؤدي إلى صوف تشطيب متعدد الوظائف. ويشمل أيضا الطباعة الرقمية النافثة للحبر عن طريق استخدام البلازما الهوائية كمعالجة مسبقة ، وكذلك المعالجة المسبقة الصديقة للبيئة باستخدام  $H_2O_2$  وغراء واستخدام مٹخنات مختلفة. تتضمن الطباعة غير المباشرة التفرغ ومقاومة الطباعة. كل هذه التقنيات تؤدي إلى تعزيز قابلية الطباعة ووظائف الصوف وخصائص الثبات لجميع مطبوعات الصوف.

**الكلمات الرئيسية:** أقمشة صوفية ، طباعة مباشرة ، طباعة غير مباشرة ، بلازما هوائية ، جزيئات نانوية  $TiO_2$  ، معالجة مسبقة صديقة للبيئة