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Tailoring Surface Properties of Cotton Fabric with Polycarboxylic Acids for High-Performance Inkjet-Printed Conductive Patterns

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ABSTRACT

The design of new inks is such that they create the desired properties on the substrate while maintaining the characteristics of the ink. The need to produce conductive and smart fabrics with inkjet printers is one of the challenges of today. In this study, an ink was created that creates an electrically conductive layer on cotton fabric after printing. To create the aforementioned layer, first a chemical reduction process is performed using an inkjet printer, and the electrical conductivity is created by the printed layer. For this purpose, in the first stage, a silver nitrate-based ink was formulated, and to increase the electrical conductivity of the printed layer, biodegradable carboxylic acids with different functional groups were used to increase the crosslinking process of cotton fabric. After treating the prepared fabrics and printing with silver nitrate and sodium hypophosphite-based ink, the electrical resistance of the printed layer and the effect of the number of functional groups on it were investigated. Also, the thermal properties, visible/ultraviolet spectroscopy, and infrared analysis of the prepared ink and the morphology of the printed nanoparticles were investigated. The results showed that the surface tension of the synthesized ink was 38.6±1.37 mN/m. The best sample was butane tetracarboxylic acid, which showed a wrinkle reversibility angle of 157 degrees and a decrease in tensile and force at break point values of 11.38 and 12.48 %, respectively. The lowest electrical resistance was also found in this sample, which was 0.10 megaohm after 5 printings. Prog Color Colorants Coat. 19 (2026), 247-259© Institute for Color Science and Technology.

1. Introduction

With the growing trend of technology, we are witnessing new developments every day. The printing process is no exception, and we are witnessing the replacement of traditional printing with new technologies [1, 2]. With the advent of inkjet printing technology, due to its advantages such as higher speed, higher quality, lower cost, and reduced waste, old methods have been gradually abandoned, and this technology is growing rapidly [3, 4].

Inkjet printers print the desired design by spraying ink onto the substrate. These inks can be designed to carry other materials, such as nanoparticles, polymeric materials, and even biological cells, with them and print on the substrate. Depending on the type of material, special and new properties are created in the printed

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surfaces, ranging from paper to polymeric surfaces and even fabrics and glass [5].

In many cases, we need to have electrically conductive surfaces. Conductivity can be created on surfaces in various ways. Today, inkjet printers create this easily with the presence of conductive particles embedded in the ink. Therefore, it is possible to create surfaces with multiple layers and different substrates, sensors, and flexible conductors. Conductive inks must have high stability and proper dispersion of the conductive material and high electrical conductivity [6, 7].

Since the conductive ink is sprayed onto the surface through the printer nozzle, it must have properties such as easy exit from the nozzle, which requires appropriate viscosity and surface tension, and uniform conductivity, which is related to proper dispersion of the conductive particles. These properties are present in silver nanoparticles, which are a good option despite their high price. The type of substrate also has a great impact on ink absorption, and factors such as type, porosity, hydrophilicity, chemical structure, and morphology can be mentioned, which affect ink absorption and surface and deep conductivity [8]. There is another type of inkjet that is cured with ultraviolet light and usually uses diluted acrylate resins in its formulation that are based on epoxy and urethane [9, 10]. In this system, silane coupling agents or other dispersants are used to disperse nanoparticles [11, 12].

Thus, substrate pretreatment and tailored ink formulations are vital for achieving uniform deposition, strong adhesion, and mechanical resilience of printed circuits. Prior studies have examined screen printing on cotton and polyester, observing that while cotton's porous nature can hinder conductivity with single prints, overprinting and selecting optimal mesh densities reduce electrical resistance and improve performance [13]. Emerging composite inks combining silver nanoparticles with graphene have demonstrated remarkable conductivity and cost benefits, achieving sheet resistances from 0.08 to 4.74 Ω/sq on pretreated cotton at low sintering temperatures [14, 15]. These hybrids exploit graphene's mechanical strength and silver's conductivity, reducing print layers and costs. This research builds on these insights by developing and optimizing silver-based conductive inks specifically for inkjet printing on cotton. It systematically examines diverse substrate pretreatments, ink formulations, and low-temperature curing protocols to produce flexible textiles exhibiting excellent conductivity, mechanical durability, adhesion, and environmental stability. Addressing these challenges, cotton fabric pretreatment plays a crucial role in modifying surface chemistry to enhance ink adhesion, reduce ink penetration into fabric pores, and improve printed pattern durability. Among pretreatment methods, biodegradable polycarboxylic acids combined with sodium hypophosphite as a catalyst have emerged as sustainable, formaldehyde-free textile finishing alternatives [16].

This research systematically explores the use of biodegradable polycarboxylic acids with different numbers of carboxyl groups, combined with sodium hypophosphite as a catalyst, for the pretreatment of cotton fabric. This surface modification improves ink adhesion and enhances conductivity. Additionally, we detail the formulation of a silver nitrate-based ink suitable for inkjet printing, coupled with a low-temperature chemical reduction process to generate conductive silver nanoparticle patterns directly on the textile. In addition, the formulation was designed to form nanoparticles directly on the fabric during the printing process to create conductivity on the smart fabric with one or more printing passes.

2. Experimental

2.1. Materials

1,2,3,4-Butanetetracarboxylic acid (99 % [-CH(CO_2H) $CH_2CO_2H]_2$), citric acid (≥99.5 %, FCC, FG HOC (COOH)($CH_2COOH)_2$), sodium hypophosphite (99.5 % $NaPO_2H_2$), propionic acid (99 % CH_3CH_2COOH), silver nitrate (99.5 % $AgNO_3$) and formic acid (99.5 % HCOOH) were purchased from Merck (Germany). Also, maleic acid (99% HOOCCH=CHCOOH) and ammonium hydroxide (28.0-30.0 % NH_3 basis, NH_4OH) were obtained from Sigma Aldrich (USA) and Dr. Mojallaly Company (Iran), respectively. The ink used for the inkjet printer was purchased from Rafraf Company (Iran). Cotton fabric was obtained from Borujerd Textile (Iran) with a specific weight of 98 g/m^2.

2.2. Methods

2.2.1. Preparation of cotton fabric with biodegradable carboxylic acids

The preparation process used the pad method (with a pressure of 5 kg/cm^2, pad speed of 3.5 m/min, and wet extraction of 80 %). The amount of carboxylic acid in the pad solution was 80 g/L and the amount of sodium hypophosphite was 60 % by weight of carboxylic acid. The curing operation after the pad process was carried out for 3 minutes at a temperature of 180 °C [17]. The carboxylic acids used were formic acid, acetic acid, propionic acid, malic acid, citric acid, and butane-tetracarboxylic acid (Figure 1). The process of modifying cotton fabric is due to the ability of the fabric to absorb more ink. Schema 1 demonstrates the difficulty of inkjet printing on cotton and the solution to overcome it.

2.2.2. Printing of silver nitrate-based ink on fabric

After treating the fabrics with a reducing agent, the prepared silver nitrate-based inks were placed in an inkjet printer cartridge, and the desired design was printed on fabric with a desktop printer at 1200 dpi. To remove the reducing agent and unreacted silver nitrate, wet extraction was performed. In the wet extraction operation, the printed layer was rinsed with distilled water [18]. After the synthesis of nano silver essence, an electrical resistance test was performed with a multimeter. The experimental procedure and equipment are demonstrated in Schema 2.

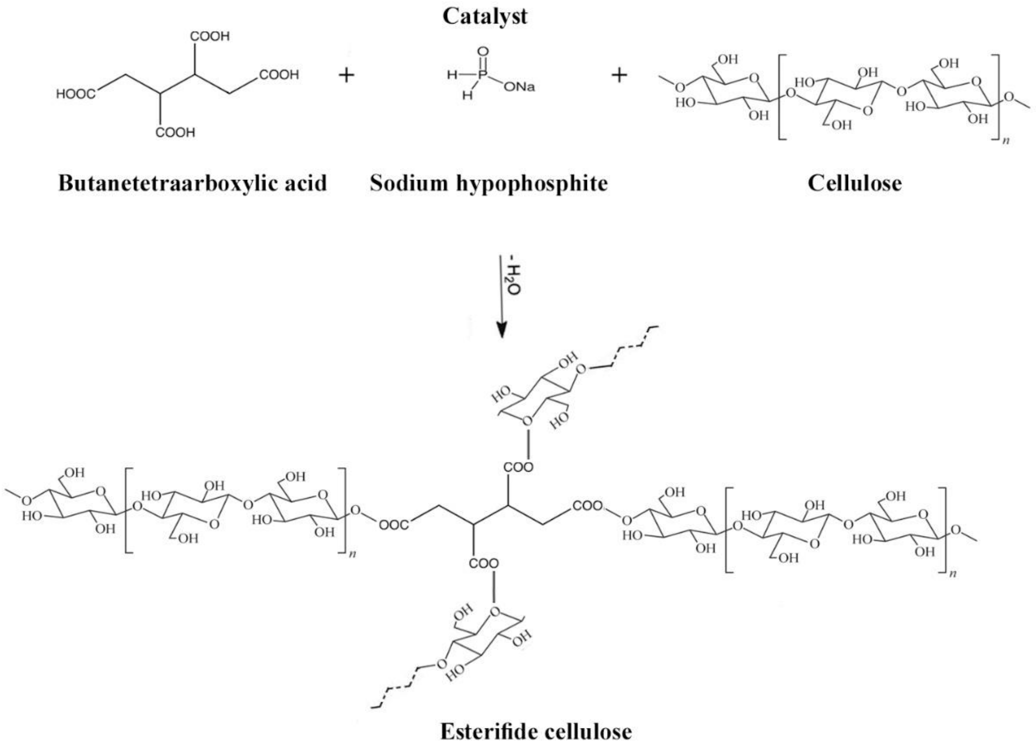
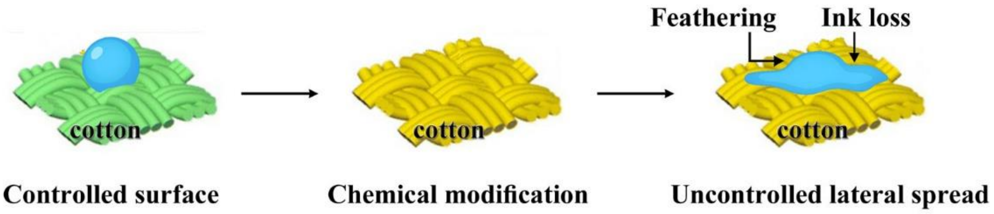
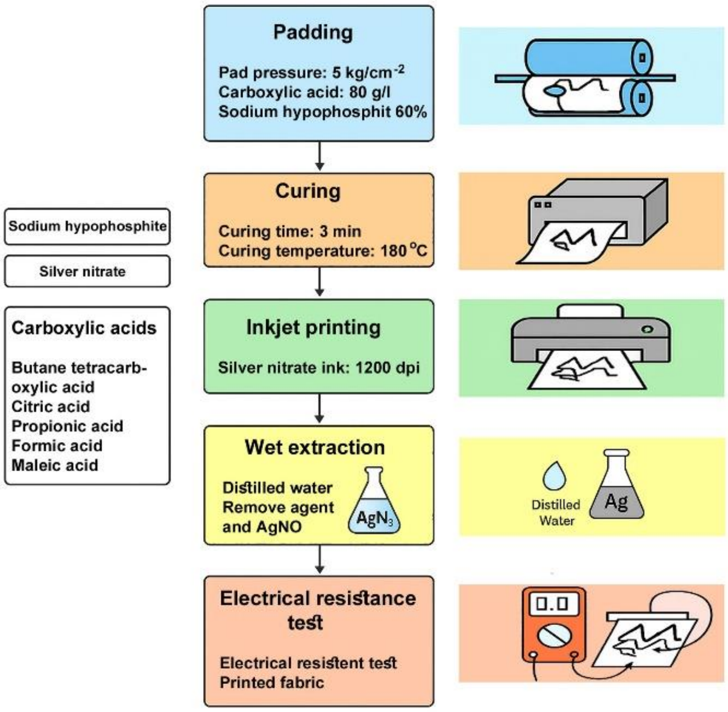


Figure 1: The process of treating cotton fibers (cellulose) with carboxylic acid and sodium hypophosphite catalyst.



Schema 1: Schematic of the difficulty of inkjet printing on cotton and the solution to overcome it.



Schema 2: The experimental procedure and equipment.

2.3. Instrumental

Fabric preparation was performed using a pad machine (Ernst Benz - Germany). The prepared fabrics were then dried in an oven (Azar Kore Company - Iran). The visual and color characteristics of the prepared and untreated fabrics were measured using a spectrophotometer (Xrite Company, USA) in the wavelength range of 400 to 700 nm. The thermal properties of the prepared and untreated fabrics were examined using thermogravimetry analysis (TGA) and derivative thermogravimetry (DTG) (PerkinElmer, USA) under an ambient air atmosphere with a heating rate of 10 °C/min. The wrinkle reversibility angle of the samples was determined according to the ISO 2313 standard. The strength and tear point of the fabric were examined using an Instron device (USA) in accordance the ASTM D5035 standard. In order to understand the chemical changes on the surface of the samples, the Fourier-transformed infrared spectroscopy in attenuated total reflection mode (ATR-FTIR) device (Nexus, USA) in the range of 1400-4000 cm^{-1} and Raman analysis (XploRA, Japan) in the range of 150-3500 cm^{-1} were used. The viscosity and rheological behavior of the inks were also measured by a rheometer (Anton Paar, Germany). Dynamic light scattering (DLS) analysis (Horiba, Japan) was performed to determine the size of the ink particles. A transmission electron microscope (TEM) (Zeiss, Germany) was used to study and examine the size and morphology of the particles. The printing operation on the fabric was performed by a Hewlett-Packard (HP) desktop thermal inkjet printer, model 5150. The printed fabrics were subsequently dried and fixed in an oven (Azar Kore, Iran). The electrical resistance of the printed layers was measured by a multimeter (Victor, China), After washing according to the AATCC 61-2001 standard with non-ionic detergent at 60 °C for 10 minutes, the electrical resistance of the printed fabrics was measured. The morphology was studied by a PHILIPS scanning electron microscope (SEM) XL30 FEG.

3. Results and Discussion

3.1. Surface tension, rheological properties and pH

The surface tension of the ink plays an important role in the jetting ability of the printer nozzle and the wetting of the substrate (fabric). The surface tension of the synthesized ink was 38.6±1.37 mN/m after three tests, which is considered suitable for industrial printers [19].

The ink's rheological properties are usually determined by its viscosity, which affects its movement in the printer's thin nozzles. In view of this, ink with high viscosity does not jet properly from the printer nozzle, and ink with low viscosity causes the ink to ooze from the nozzle and reduces the quality of the printed image, so the rheological properties of the synthesized ink were investigated by measuring the viscosity at different shear rates. Figure 2 shows the rheological behavior of the ink at different shear rates. The synthesized ink exhibits Newtonian behavior at shear rates above 600 (1/s) and is within the appropriate range.

The pH of the ink also affects its properties and printability. A high acidic pH of the ink will damage the cartridge, and a high alkaline pH will dissolve the cartridge adhesive. The pH of the synthesized ink was 8.5, which is considered appropriate.

3.2. Particle size analysis of synthesized ink

Figure 3 shows the particle size distribution and electron microscope images of the ink in solution and after drying on the fabric and scraping. As can be seen, nano silver particles are formed in near-spherical shapes in various dimensions, and the largest size of silver nanoparticles is in the range of 100-200 nm. This particle size is suitable for being placed in printer ink and passing through the nozzle and is usable. In order to prevent clogging of the printing machine nozzle, the prepared inks were filtered using a 200 nm syringe filter.

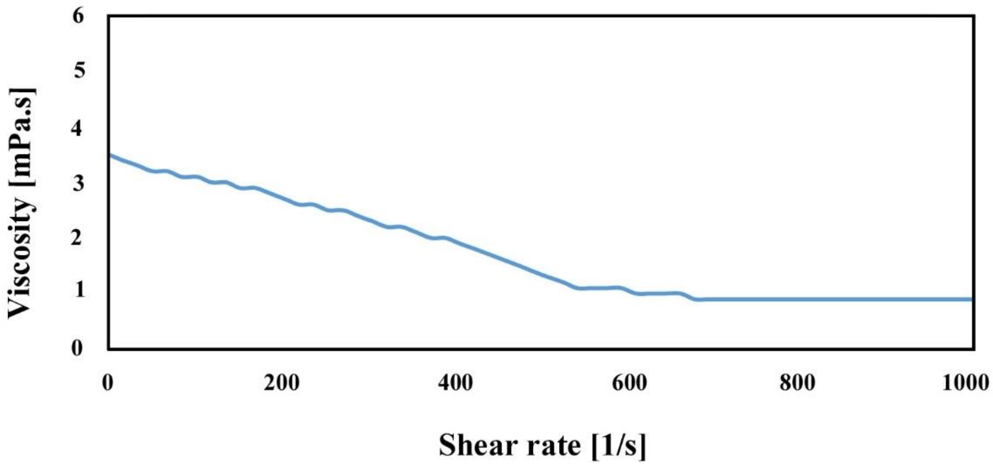


Figure 2: Rheological properties of the synthesized ink based on silver complex.

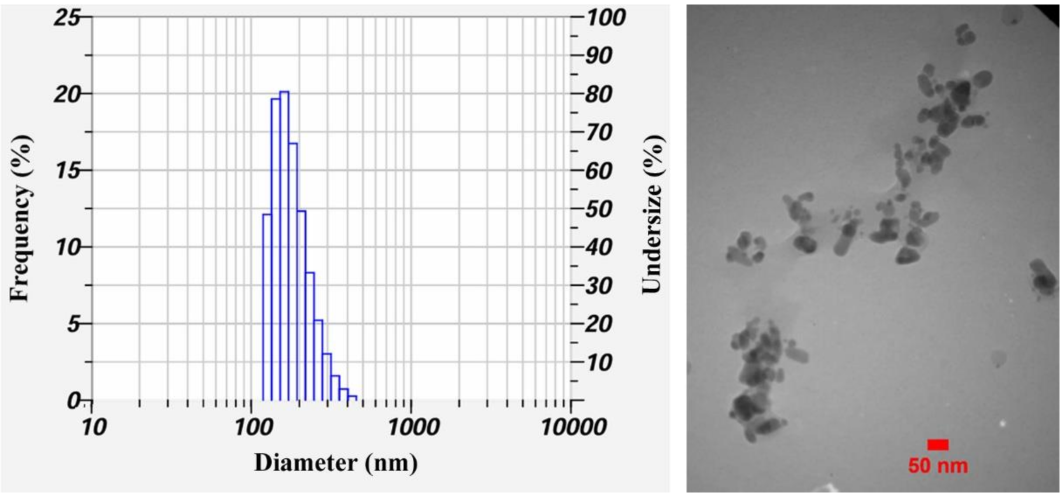


Figure 3: Particle size distribution of nano silver in ink and its transmission electron microscope images.

3.3. Morphology and EDS analysis

In Figure 4, images of samples of untreated cotton fibers (Figure 4a) and treated cotton fibers and silver nanoparticles (Figure 4b) after inkjet printing with the prepared ink are seen. In these images, the formation of silver nanoparticles on the fibers and the dispersion of silver are clearly observed.

Table 1 shows the quantitative results of EDS analysis of the modified sample, which includes the elements carbon, oxygen, chlorine and silver. The weight percentages of the elements were 40.25, 12.33, 1.27, and 46.15 % also the atomic percentages were 73.09, 16.81, 0.78 and 9.33 %, for carbon, oxygen, chlorine, and nano silver, respectively. As can be seen, the amount of nano silver is very high, which firstly shows the successful formation of silver on the cotton fibers of the fabric, which is observed in the electron microscope image. Secondly, this amount of nano silver is good evidence of the electrical conductivity created in the cotton fabric. The presence of oxygen and carbon is related to the cellulose structure of the fabric and the carboxylic acid groups used in the modification of the fabric. The measurement error for all elements is low and is about 2.4-1.6, which indicates high measurement accuracy and greater confidence in the data. The atomic, absorption and fluorescence (ZAF) corrections calculated and presented in the table are also very low (less than 1), which is another confirmation of the high accuracy of the analysis. However, the amount of chlorine in the sample, which is much lower than other elements, is related to the residual impurities from the salts used in the reduction process. In Figure 5, the energy-dispersive x-ray spectroscopy spectrum is also observed, where the presence of element peaks is evident, and the strong peak formed at an energy of about 2.98 kEv and in the Lα region is related to the predominant presence of silver nanoparticles on the surface of the cotton fabric sample.

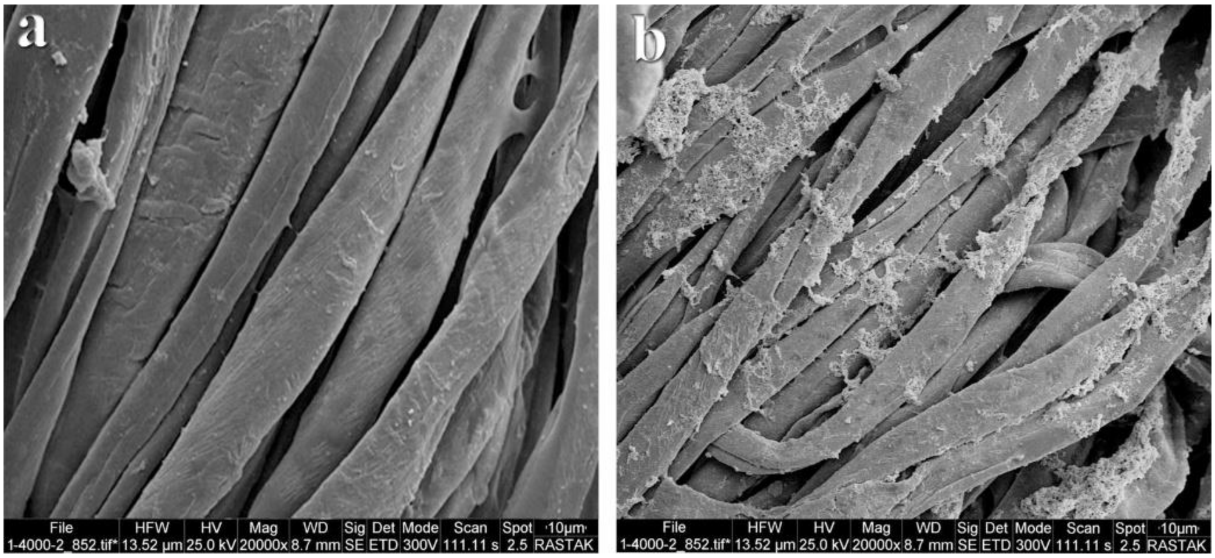


Figure 4: Untreated cotton fibers (a) and treated cotton fibers and silver nanoparticles (b).

Table 1: The quantitative results of EDS analysis of the modified sample.

Elt	Line	Int	Error	K	Kr	W%	A%	ZAF	Ox%	Pk/Bg
C	Kα	229.7	2.4155	0.3609	0.2360	40.25	73.09	0.5862	0.00	32.57
O	Kα	40.6	2.4155	0.0317	0.0208	12.33	16.81	0.1683	0.00	7.76
Cl	Kα	45.6	1.6191	0.0186	0.0121	1.27	0.78	0.9575	0.00	5.48
Ag	Lα	618.6	1.6191	0.5888	0.3850	46.15	9.33	0.8343	0.00	25.99
				1.0000	0.6539	100.00	100.00		0.00	

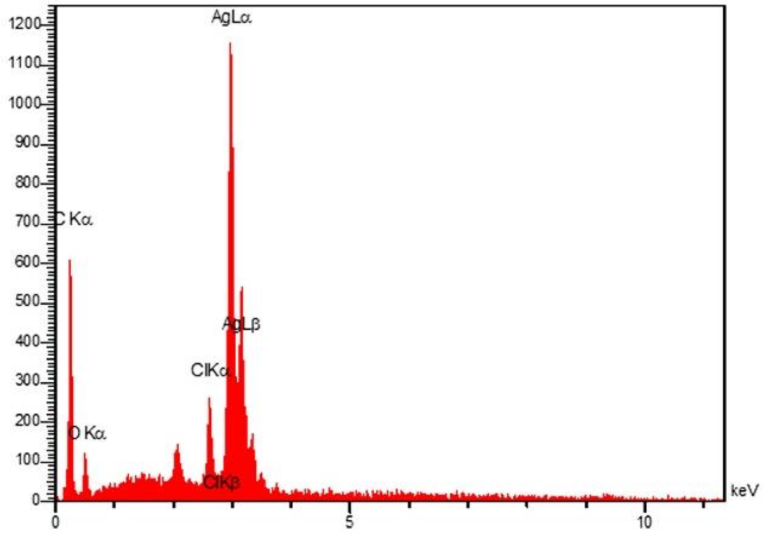


Figure 5: The energy-dispersive x-ray spectroscopy spectrum of the modified sample.

3.4. Thermal properties of ink

According to the thermal analysis for ink (Figure 6), an initial peak is visible, which is related to the evaporation of water and possible impurities; with an increase in temperature to about 400 °C, we reach the evaporation point of silver nitrate, after which the only remaining phase will be silver. In addition, the analysis shows that due to the high melting point of silver, there is a limitation on the application of temperature for heat treatment due to the use of fabric as a substrate, and the application of high temperatures may lead to discoloration and destruction of the fabric. In fact, the advantage of using the chemical reduction method is the production of nanoparticles at low temperatures and mild conditions, which is why this method was used.

3.5. UV-visible spectroscopy of synthesized silver complex-based ink

UV-visible spectroscopic analysis was performed on the silver complex solution in the range of 200 to 800 nm, the results of which are shown in Figure 7. This analysis can be used to investigate the structure of the existing silver, as different silver structures have different absorptions. Generally, the absorption peaks of Ag^+ ions are determined in the region of 190-230 nm. The peaks related to silver atoms Ag^0 show themselves in the region of 270-330 nm. The peaks related to silver clusters Ag_n are determined in the regions of 330-360 and 440-540 nm. The absorption related to the phenomenon of surface plasmon resonances also occurs at 380-450 nm [20, 21].

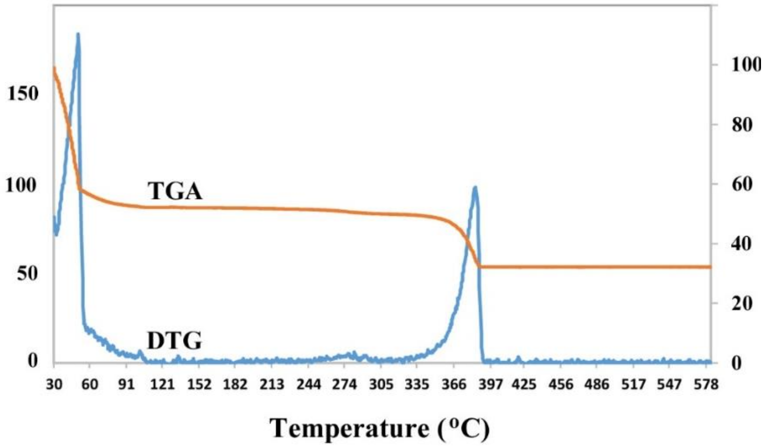


Figure 6: Thermogravimetric analysis on silver complex-based ink.

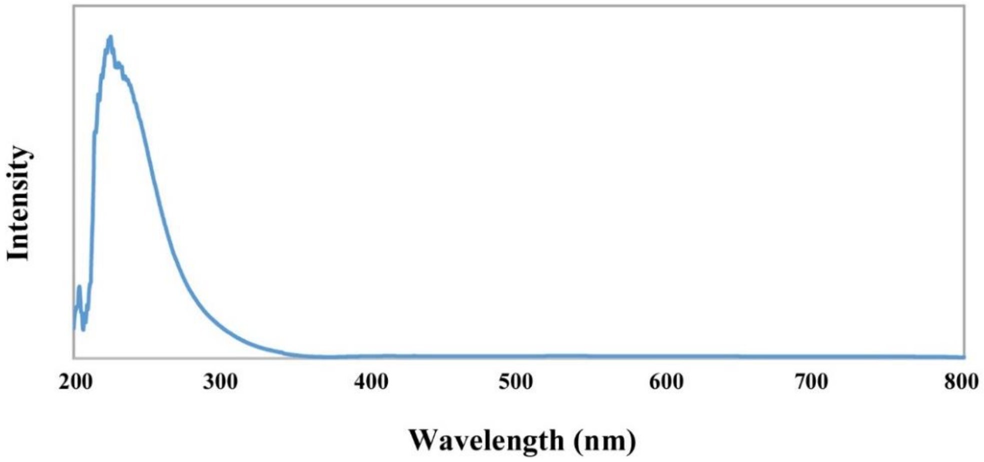


Figure 7: UV-Visible spectroscopic analysis of the silver complex-based ink.

According to the above explanation and the analysis of the silver complex, no absorption peaks are observed in the range of silver nanoparticles, which indicates that the prepared ink is particle-free. Absorptions due to the presence of silver ions occur at about 250 nm.

3.6. Fourier transform infrared analysis of cotton fabrics treated with carboxylic acids

To investigate and understand the ester linkage formed in the prepared samples, Fourier Transform Infrared Analysis was performed on fabrics treated with formic acid, malic acid, butanetetracarboxylic acid, and citric acid, and the results are shown in Figure 8.

The Fourier transform infrared analysis spectra obtained from the untreated cotton sample and the cotton prepared with different carboxylic acids are shown in Figure 8. As can be seen, the peaks related to glucose are in the range of 800 to 1250. In the above spectrum, the cotton fabric has a broad peak at 13263 cm^{-1}, which indicates the presence of hydroxyl (OH) groups in the cellulose structure of the cotton, and the difference in this region in different samples is due to the change in the number of OH groups and their reaction with carboxylic acid. A clear peak at 1730 cm^{-1} is seen, which is related to the carbonyl stretching (C=O) in the structure, which in unprepared cotton is due to the ester bond of the carboxylic group of lignin [22] or the stretching of the C=O group in the hemicellulose structure [23, 24], and its significant increase compared to the prepared samples confirms the formation of an ester bond between cellulose and carboxylic acid, which is more pronounced in the

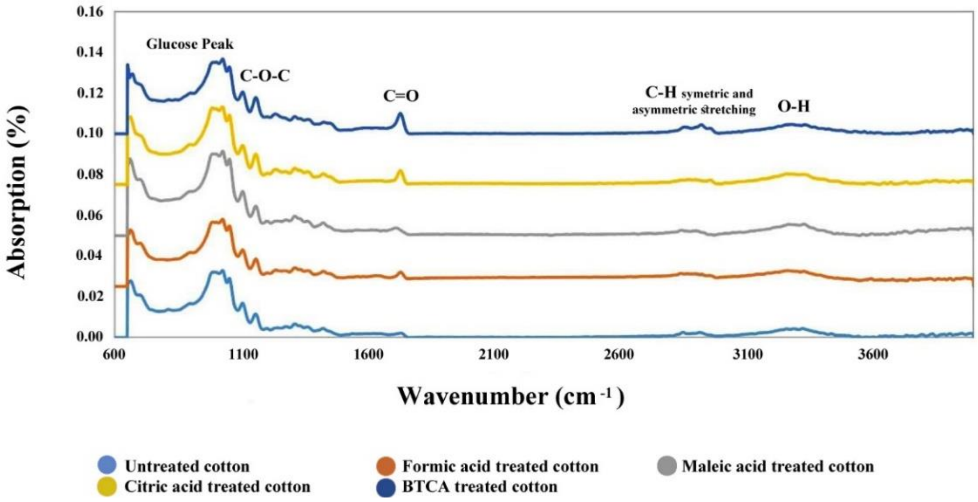


Figure 8: Fourier transform infrared analysis of the sample prepared with different carboxylic acids.

citric acid and butane tetracarboxylic acid samples. The peaks at 12872 and 12915 cm^{-1} are related to the symmetric and asymmetric C-H stretching, and the weak peak at 11312 cm^{-1} is related to the vibrations of the hydroxyl groups (-OH). The sharp peak at 11157 cm^{-1} confirms the presence of C-O-C stretching in the cotton structure [25]. The peaks at 1053 and 11025 cm^{-1} are related to C-O, -C-C- or -C-C-O groups [26]. The peak at 1425 cm^{-1} is related to O-C-H and H-C-H conformational changes [27].

3.7. Effect of carboxylic acid-based treatment on physical properties of fabric

The process of esterification of cotton fabric with carboxylic acids creates a network of cross-links in the fabric and can affect its physical properties. For this purpose, to understand the effect of carboxylic acid-based treatment, tensile/force at break tests and wrinkle reversibility angle tests were performed on butane tetracarboxylic acid and citric acid samples according to ASTM D5035 and ISO 2313 standards, respectively. The results (average of 5 repetitions) are given in Figures 9 and 10.

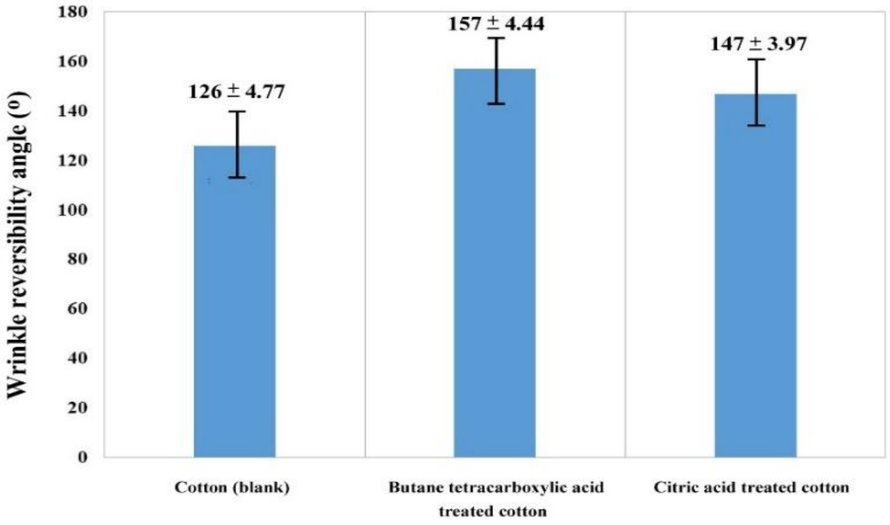


Figure 9: Wrinkle reversibility angle of fabrics treated with carboxylic acids.

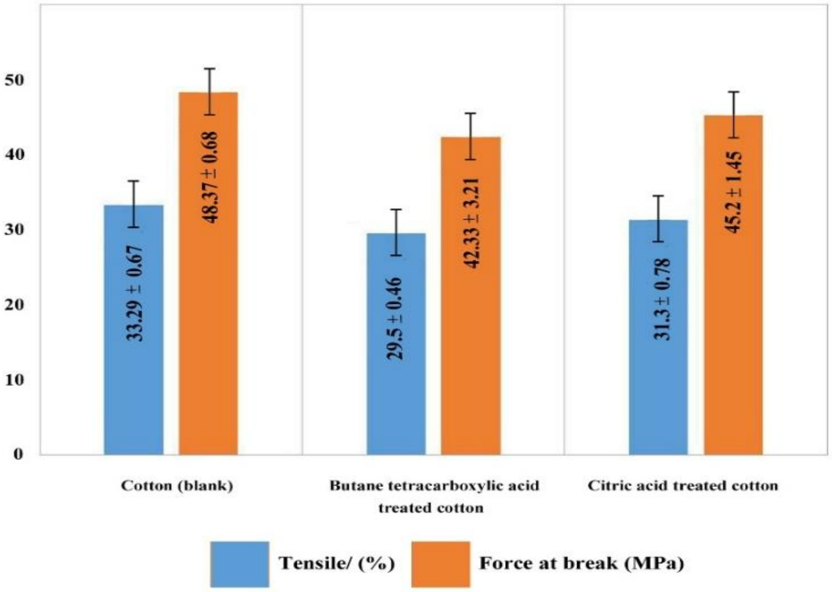


Figure 10: Tensile and force at break point test of fabrics treated with carboxylic acids.

As can be seen from the figures, the wrinkle reversibility angle value for the butane tetracarboxylic acid-modified sample is 157 degrees, and the citric acid-modified sample is 147 degrees, which is an increase of 24.6 and 16.6 % for butane tetracarboxylic acid and citric acid, respectively, compared to the unmodified cotton fabric sample. The reason for this increase is due to the increased degree of crosslinking. The greater increase in the wrinkle reversibility angle value in butane tetracarboxylic acid compared to citric acid is due to the greater number of carboxylic acid groups.

The results of the tensile and force at breakpoint tests showed a decrease in both cases, so that the tensile value decreased by 11.38 and 5.97 %, respectively, in butane tetracarboxylic acid and citric acid compared to the unmodified cotton fabric sample, which is due to the hardening of the sample with the increase in cross-linking. Also, the hardening of the samples caused a decrease in the force at the breakpoint by 12.48 and 6.55 % in butane tetracarboxylic acid and citric acid, respectively, compared to the unmodified cotton fabric sample.

3.8. Effect of preparation based on carboxylic acids on the conductivity of the printed design

The preparation process was carried out using biodegradable carboxylic acids and sodium hypo-phosphite as a catalyst. In addition, carboxylic acids without sodium hypophosphite were also used to prepare the fabric, but due to the absence of a catalyst, the crosslinking reaction was not successful. In the absence of catalyst, the esterification process is not well carried out and the crosslink network is not formed, which as a result does not reduce the penetration of the ink into the fabric [28]. In samples without a catalyst, electrical conductivity is not observed. The results of the electrical resistance of the printed layer with silver nitrate-based ink were measured with a multimeter (with 5 repetitions and a distance between the probes of 1 cm) which are summarized in Table 2. Figure 11 shows images of fabrics printed 1, 3, and 5 times with different carboxylic acids.

Table 2: Electrical resistance results for samples treated with different carboxylic acids.

Cotton	Fabric	Carboxylic	Resistance
Kind of treatment	Number of printed layers	functionality	(MΩ)
Formic acid	1	1	6.00
	3		1.10
	5		0.45
Acetic acid	1	1	5.50
	3		2.45
	5		0.55
Propionic acid	1	1	5.70
	3		2.25
	5		0.65
Maleic acid	1	2	4.50
	3		0.60
	5		0.42
Citric acid	1	3	4.50
	3		0.35
	5		0.15
BTCA	1	4	0.31
	3		0.22
	5		0.10

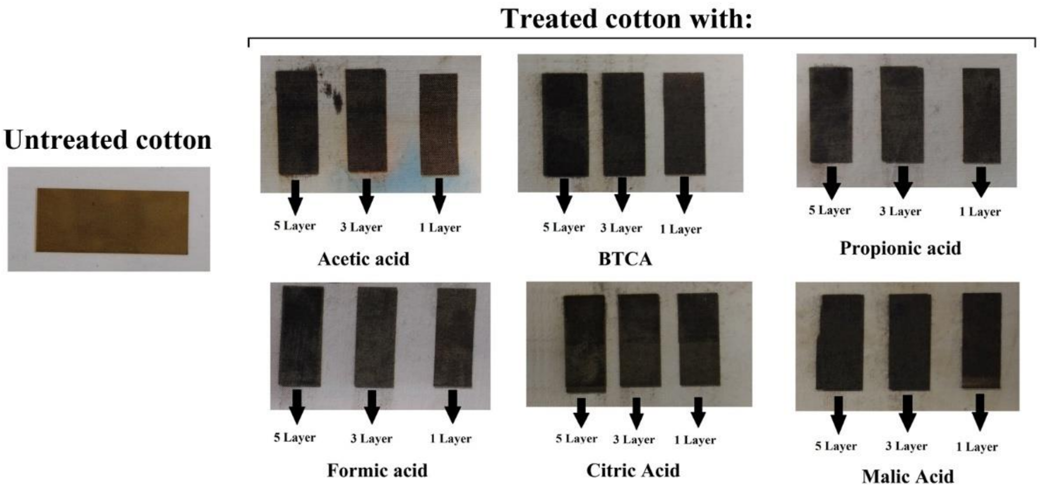


Figure 11: Image of samples treated with carboxylic acid and then printed with silver nitrate-based ink.

As is clear from Table 1, the electrical resistance has decreased with the increase in functional groups, so that for propionic acid, maleic acid, citric acid, and butane tetracarboxylic acid with 1, 2, 3, and 4 functional groups for 5 times of printing, it has become 0.65, 0.42, 0.15, and 0.10 megaohms, respectively, which means higher conductivity. Also, the number of printing times of 1, 3, and 5 times of printing has decreased the electrical resistance and increased the conductivity. This observation is because silver nanoparticles acquire a positive charge in acidic environments [29], which creates an affinity for the hydroxyl and carboxyl groups of the crosslinking agents, which ultimately leads to greater adsorption of nanoparticles onto the fiber. The use of carboxylic acid, due to the creation of a network of crosslinks in cotton as shown in Figure 12, traps silver nanoparticles between the chains and increases the stability of the nanoparticles. In addition, when carboxylic acid is used as a fabric preparation, it reduces penetration into the fiber due to crosslinking of cotton fibers, and easier printing, a more uniform design, and higher conductivity are created. In order for the complete esterification process of cotton fabric to be carried out, the carboxylic acid used must have three or more carboxyl groups. Therefore, in the case of fabrics prepared with formic acid and malic acid, the crosslinking process is not complete, and not all functional groups have reacted. As a result, the cotton fabric preparation process is not well completed [30]. The toughness and impermeability of cotton fabric increase with the increase of functional groups, resulting in decreased ink penetration and increased electrical conductivity. On the other hand, carboxylic acid and sodium hypophosphite themselves can act as a

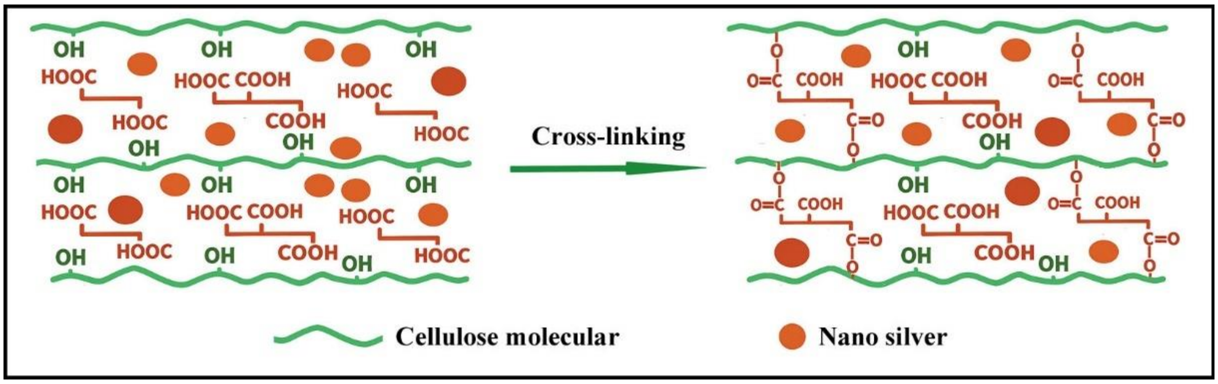


Figure 12: Immobilization of silver nanoparticles in cellulose chains in the presence of carboxylic acid.

reducing agent and increase the reaction efficiency. According to Yang's findings, for an effective esterification reaction, carboxylic acids with more than three carboxylic groups should be used. The higher the number of carboxylic groups, the greater the degree of crosslinking, decreased ink penetration, and improved electrical conductivity, with the best results in this area for citric acid and butane tetracarboxylic acid, which have three and four functional groups. In addition, with the increase in the number of printing cycles, the synthesis of nanoparticles on the surface increases and the electrical conductivity increases.

As mentioned, the electrical conductivity of the printed design increases with the increase in carboxylic groups in the preparation process. On the other hand, the conductivity decreases with the increase in the length of the carbon chain for carboxylic acids with equal functionality, which is due to the steric hindrance of the side chain in the process of esterification of cotton fabric with carboxylic acid.

4. Conclusion

The effect of carboxylic acids as a pretreatment agent for cotton fabric was studied. Six different carboxylic acids were used in the presence of a catalyst. The physical properties of the fabric after pretreatment were examined, and pretreatment with carboxylic acids improved the wrinkle reversibility angle but reduced its tensile strength. The best pretreatment grade was butane tetracarboxylic acid, which was observed by Fourier transform infrared analysis to have a carbonyl group resulting from its esterification reaction with cotton. After pretreatment, the fabric was printed with silver nitrate-based ink. The best sample was tetracarboxylic butane with a wrinkle reversibility angle of 157 degrees and elongation values of 29.5% and 42.33 MPa. The electrical resistance of this sample after 1, 3, and 5 printings was 0.31, 0.22, and 0.10 megaohms, respectively. Changing the silver nitrate-influencing agents, reducing the number of prints, using other conductive particles suitable for inkjet printing, using other modifiers, and investigating printing on non-cotton fabrics are some of the issues that require future research.

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