Development and characterization of conductive textile (cotton) for wearable electronics and soft robotic applications

Zuhaib Hassan, Fatma Kalaoglu and Ozgur Atalay

Abstract
This study aims to manufacture and characterize various types of conductive cotton fabrics through the copper metal coating approach. Thus, we selected nine-combed cotton knitted fabrics with different yarn fineness and elastane percentage in order to see the effect of these parameters on conductivity and physical properties of the samples. We also explored the surface morphology of all the knitted cotton fabric samples before and after the coating method via scanning electron microscopy (SEM), which showed a remarkably uniform deposition of copper on the fabric surface, and performed SEM-energy-dispersive X-ray spectroscopy to determine the coated material content on the surface of the fabric after the metal coating process. The results revealed that knitted cotton fabric of 5% elastane with the finer yarn count (Ne = 40/1) showed excellent conductivity compared to the other knitted cotton fabric of 10% elastane with a finer count (Ne = 40/1) or coarser 5% elastane (Ne = 30/1). Therefore, the knitted cotton fabrics of 5% elastane having the finer count (Ne = 40/1) can be considered a suitable candidate for e-textile applications.

Keywords
knitted cotton fabric, electroless copper plating, scanning electron microscopy, conductivity, scanning electron microscopy-energy-dispersive X-ray spectroscopy

Wearable electronics fabricated from stretchable conductive fabrics have gained significant attention in recent years due to their various applications, including medical, soft robotics, sports and security. Electronic textile products are highly suitable for practical use with regards to their inherent properties, such as providing comfort and having warmth, lightness and flexibility. Electronic textiles are also suitable for use in fiber-based or body-attachable platforms to promote industrializing valuable materials with an integrated format. The structural and physical properties of the fabrics, namely flexibility, breathability and absorption, have the essential effect of developing such systems. The advantage of the textile materials is providing comfort and mobility to the user in this respect and that textile-based platforms do not limit the wearer. Therefore, investments and research in this area have dramatically increased in the last decade to produce and improve wearable electronic platforms, which are also called electronic textiles.

The application areas of electro textiles significantly affect the number and type of electronics textile components within the structure. Although, to accomplish the task of the e-textile product, it is necessary to have particular groups of components, such as sensors/electrodes, a power supply and a communication network, within the platform or and having it as an external platform, an actuator and a data processor, which can all be considered the essential elements of an e-textile product.

To produce such components, that is, sensors, signal transmission lines, etc., electrical conductivity is a vital property. Conductive textiles are formed by metal strands woven into the construction of the textile or metal coating over the nonwoven, knitted or woven textile substrate. Textile fibers normally are
non-conductive substrates, which are then either coated or embedded with electrically conductive materials, such as nickel, gold, silver, carbon, carbon nanotubes (CNTs), copper, titanium or poly(3,4-ethylenedioxythiophene) (PEDOT). There are mainly three methods for depositing, namely electroplating, sputtering or electroless plating, to make the fabric. In this work, electroless plating was carried out to form conductive fabrics; the integration of electronic properties directly into the fabric structure carries some advantages, such as increased comfort, reliability, mobility, usability and aesthetic features.

Among the different metallization processes, electroless plating is the most promising process at the industrial level because of its simple and easy operation and low-cost method. Therefore, researches on the fabrication of the fabric coated with various metallic materials by electroless deposition have attracted much attention recently. However, there are still some points that need to be improved in the conventional electroless plating process. For example, in the plating process, if we used formaldehyde as the reducing agent then we need to cover the beaker with stretch film throughout the process, because formaldehyde is a volatile organic compound and it evaporates during the process and effects the plating process.

In previous works, weft knitted strain sensors were developed by using different knitted conductive yarns, that is, silver-plated nylon yarns and polyester-blended stainless steel yarns, with flatbed knitting machines; these kinds of strain sensors have been used for human body motion monitoring and respiratory activity. Textile-based strain-sensing structures can be used to measure the human body motion, monitoring and physiological signals where comfort properties and sensing reliability are required.

Therefore, we envision expanding the usage of our conductive fabric structure in the areas of human motion monitoring as well as soft robotic applications. The main objective of this work is to develop conductive textile fabric using combed knitted cotton fabrics with elastane and investigate the effect of the fabric parameters on the electrical conductivity and physical properties of fabrics. This paper also provides significant information about the characterization of conductive knitted combed cotton fabrics and the most appropriate conductive cotton fabric for the further process of e-textile applications.

**Experimental work**

**Materials**

The nine-combed cotton fabrics (single jersey) with different counts, fabric weight (gsm) values and elastane percentage used in this study are given in Table 1. All reagents used were of analytical grade in this work. Stannous chloride (SnCl2) was acquired from Merck KGaA (Germany) and the HCl solution (37%) was supplied by Riedel-de Haen sigma Aldrich. AgNO3 was purchased from Merck, and ammonia solution (25%) was acquired from Merck KGaA (Germany). Copper (II) sulfate pentahydrate was purchased from Carlo Erba reagents (France), potassium sodium tartrate (KNaC4H4O6-C14H2O) and formaldehyde (HCHO) were acquired from Merck and sodium hydroxide (NaOH) was supplied by Riedel-de Haen sigma Aldrich.

**Fabrication of the conductive fabrics**

There are different methods to make the fabric conductive, for example, electroplating, sputtering, laser-assisted metallization and the electroless plating process. For this study, we have selected the electroless plating process. The electroless plating method was chosen to fulfill this aim, since the process allows the coating of non-conductive textile materials and it is relatively cheap compared to the other available options for making conductive fabric. Electroless plating is used to deposit a coating of a metal on a substrate without using an external power source, such as electricity. Unlike electroplating, usage of direct electric current is not required. Conventional electroless plating involves several heated pre-treatments and activation baths followed by one or more plating baths and then a rinsing bath.

First of all, we washed the fabric samples with 3% non-ionic detergent at 20°C for 40 minutes to remove any oil, dust or impurities from the surface of the fabrics that might have been randomly scattered during the manufacturing process. The machine used for the washing process was an SDL Atlas solution and the standard used for the washing process was 15ISO 5 A 1984.

| Sample | Type             | g/m² | Elastane (%) | Denier (Ne) | Count (Ne) |
|--------|------------------|------|--------------|-------------|------------|
| 1      | Combed cotton    | 250.72 | 10           | 20          | 40         |
| 2      | Combed cotton    | 270.65 | 10           | 30          | 40         |
| 3      | Combed cotton    | 290.64 | 10           | 40          | 40         |
| 4      | Combed cotton    | 136.71 | 5            | 20          | 40         |
| 5      | Combed cotton    | 154.51 | 5            | 30          | 40         |
| 6      | Combed cotton    | 170.42 | 5            | 40          | 40         |
| 7      | Combed cotton    | 176   | 5            | 20          | 30         |
| 8      | Combed cotton    | 171.22 | 5            | 30          | 30         |
| 9      | Combed cotton    | 202.70 | 5            | 40          | 30         |
The electroless plating process consists of three steps, which were carried out in this study. These are sensitization, activation, and the plating process. All three steps were performed by electric automatic controlled temperature heaters IKA C-MAG HS7 and IKA ETS-D5. After the washing process, samples were rinsed with a solution (distilled water plus some drops of HCl) for about 5 minutes. During the sensitization process, washed samples were subjected to a surface sensitizer, and we used a mixture of 5 g/l of tin (II) chloride (SnCl$_2$) and 5 ml/l of HCl solution at 25°C for 10 minutes and pH 1.

After the sensitization process, the second step is activation. The primary purpose of the activation process is to activate the samples for the plating process. Without the proper activation operation, we cannot achieve the required goal of copper coating on the fabric surface. In the activation process, we used a mixture of 10 g/l of silver nitrate and 10 ml/l of ammonia solution at 25°C for 20 minutes. After the activation process, samples were rinsed with the solution (distilled water plus some drops of HCl). The next and foremost step is the plating process. The plating bath is the most important step in the electroless plating process and is composed of 10 g/l of copper (II) sulfate pentahydrate, 45 g/l of potassium sodium tartrate (KNaC$_4$H$_4$O$_6$·4H$_2$O) (complexing agent), 20 ml/l of formaldehyde (HCHO) solution and 10 g/l of sodium hydroxide (NaOH). This solution was used at 30°C for 20 minutes and pH 11. The primary purpose of the complexing agents is to reduce the formation of copper to copper hydroxides (Cu(OH)$_2$) during the process.$^{39-41}$

Having completed the plating process, the next step was the post-treatment process. At this stage, copper-plated fabric samples were rinsed with distilled water at 40°C for 20 minutes by electric automatic controlled temperature heaters IKA C-MAG HS7 and IKA ETS-D5. Thereafter, clean copper-plated fabrics were dried in a Thermo Scientific Heraeus heating and drying oven machine at 80°C for 30 minutes and were spread at room temperature with standard conditions for 24 hours until fully dry. In the electroless copper plating process, copper metal was deposited over the fabric sample and we used formaldehyde solution as a reducing agent in the plating bath.$^{42,43}$ The reactions are shown below

\[
\text{Metal ion (M$^+$)} + \text{Reducing agent (Red)} \\
\rightarrow \text{Metal Deposited} + \text{Oxidized product (Ox)} \tag{1a}
\]

There are two reactions in the electroless plating that are given below

\[
\text{Metal deposition : Metal ion (M$^+$)} \\
+ \text{electron} \rightarrow \text{M (metal)} \tag{1a}
\]

\[
\text{Oxidation : Reducing agent (Red)} \\
+ \text{Water} \rightarrow \text{Oxidized product} + \text{H}^+ + \text{electron} \tag{1b}
\]

In the electroless process, as shown in Figure 1, the reaction is catalyzed by employing a suitable reducing agent (formaldehyde (HCHO)), which supplies the electron for the reduction reaction, and metal (copper) is deposited over the substrate. Here we kept the temperature of the plating bath at 30°C for 20 minutes.

A schematic representation of the electroless metal deposition process is presented in Figure 1, and Figure 2 shows the stepwise process flow of electroless plating for the conductive fabric.

**Characterization**

We performed a list of characterizations to check the properties of the conductive fabric samples after the coating process. Conductivity measurements of the samples were carried out with a Fluke digital precision multimeter device (Fluke 8846A). American Association of Textile Chemists and Colorists (AATCC) test method 76-1995 was used to measure the resistance of the fabric samples.
We calculated the weight gained percentage of all the samples by the following formula

$$\text{Percentage Weight Gain} = \frac{\text{Final Weight}}{\text{Initial Weight}} \times 100\%$$

The thickness test of all of the coated and uncoated samples was carried out with an R&B Cloth Thickness Tester (James Heal Instruments) with parameters of 10 g/cm² with 0.01 mm accuracy. The standard used for measuring the thickness test is D1777-96. The thickness values were taken from five different places on the coated and uncoated fabrics, and the average of these values was accepted as the thickness of the fabric. Washing cycles of all the samples were performed. We washed all the knitted cotton fabric at 40°C for 20 minutes and dried at 80°C for 30 minutes in a Thermo Scientific Heraeus heating and drying oven machine, and then we placed the samples at room temperature in the standard testing atmosphere 65±2% relative humidity (RH) to make it completely dry. After 24 hours, we checked its conductivity and measured the weight.

The abrasion tests for all of the knitted cotton fabrics was performed by Martindale James H. Heal & Co., Ltd. The parameter set for the knitted cotton fabric was 9 kPa, and the standard used for abrasion tests was D4966-12.

The microscopic analysis of all of the coated and uncoated fabric samples was carried out using a Motic SMZ-140-143-FBGG Stereo Zoom Microscope. Scanning electron microscopy (SEM) was used for surface topography and composition. SEM images were recorded by a TESCAN VEGA 3 to study the surface morphology of the controlled and copper-plated samples. Energy-dispersive X-ray spectroscopy (EDX) is an analytical technique used for the elemental analysis or chemical characterization of a sample. It was carried out via the TESCAN VEGA 3.

**Results and discussion**

**Conductivity**

The electrical resistance values of all the samples for this study are reported in Table 2. The results showed that Samples # 4–6 have much lower electrical resistance, meaning excellent conductivity values, as compared to the other samples.
Weight gained percentage results give information about how much weight is gained after the coating process via the electroless plating method. We processed all nine samples of the knitted cotton fabric and calculated their initial and final weights; the results are reported in Figure 3. The results showed that Samples # 4–9 gained good weight after the coating process.

Table 2. Electrical resistance and thickness of all of the knitted cotton fabrics

| Fabric | Resistance (Ω) | Thickness (uncoated) | Thickness (coated) | Thickness increase percentage (%) |
|--------|----------------|-----------------------|--------------------|-----------------------------------|
|        | St. dev (mm) | St. dev (mm) | St. dev (mm) | St. dev (mm) | St. dev (%) |
| Sample 1 | 21.64807 | 0.806 | 0.906 | 0.906 | 12.406 |
| Sample 2 | 77.56284 | 0.814 | 1.026 | 1.026 | 26.02 |
| Sample 3 | 93.81817 | 0.878 | 0.97 | 0.97 | 10.478 |
| Sample 4 | 2.245631 | 0.65 | 0.922 | 0.922 | 41.846 |
| Sample 5 | 3.345606 | 0.678 | 1.03 | 1.03 | 51.917 |
| Sample 6 | 6.904723 | 0.670 | 1.086 | 1.086 | 62.08 |
| Sample 7 | 16.14209 | 0.586 | 0.88 | 0.88 | 50.17 |
| Sample 8 | 30.45518 | 0.538 | 0.7 | 0.7 | 30.11 |
| Sample 9 | 54.88928 | 0.638 | 0.928 | 0.928 | 45.45 |

Figure 3. Graphs of thickness, weight gain percentage and resistance values of knitted cotton.

Weight gained percentage

Weight gained percentage results give information about how much weight is gained after the coating process via the electroless plating method. We processed all nine samples of the knitted cotton fabric and calculated their initial and final weights; the results are reported in Figure 3. The results showed that Samples # 4–9 gained good weight after the coating process.
Thickness

The thickness values were taken from five different places on the coated and uncoated fabrics, and the average of these values was accepted as the thickness of the fabric. The thickness value showed how much of the coating material was deposited onto the surface of the fabrics after the electroless plating process. The results showed that the thickness values are likely to be uniform throughout the surface of the samples, as reported in Table 2, and the graphs of the results are reported in Figure 3.

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**Table 3. Washing cycles of all the knitted cotton fabrics**

| No. of washing cycles | 0 | 1 | 2 | 3 | 4 | 5 |
|-----------------------|---|---|---|---|---|---|
| Fabric | Weight (g) | Resistance (Ω) | Weight (g) | Resistance (Ω) | Weight (g) | Resistance (Ω) | Weight (g) | Resistance (Ω) | Weight (g) | Resistance (Ω) | Weight (g) | Resistance (Ω) |
| Sample 1 | 2.8624 | 43.53428 | 2.8111 | 76.1284 | 2.7455 | 58.4589 | 2.6871 | 198.9421 | 2.6553 | 289.9841 | 2.6193 | 546.9331 |
| Sample 2 | 3.2612 | 112.4411 | 3.4888 | 290.5183 | 3.4433 | 420.5648 | 3.4080 | 653.0237 | 3.3921 | 768.1193 | 3.3813 | 998.4083 |
| Sample 3 | 3.3435 | 162.3488 | 3.2827 | 231.0471 | 3.2687 | 541.8455 | 3.2487 | 782.4381 | 3.2321 | 1208.6341 | 3.2181 | 1497.5821 |
| Sample 4 | 2.5911 | 3.6163 | 2.5111 | 6.84141 | 2.4888 | 8.9348 | 2.4657 | 14.9367 | 2.4497 | 19.53914 | 2.4397 | 22.4624 |
| Sample 5 | 2.7744 | 5.6688 | 2.7127 | 7.22343 | 2.6898 | 11.5493 | 2.6758 | 18.8936 | 2.6638 | 27.73921 | 2.6568 | 37.56389 |
| Sample 6 | 0.3120 | 8.1401 | 0.3081 | 15.7391 | 0.3067 | 26.6391 | 0.3051 | 32.6739 | 0.3039 | 42.87389 | 0.3031 | 57.36479 |
| Sample 7 | 0.2791 | 48.4629 | 0.2714 | 139.9441 | 0.2679 | 198.9847 | 0.2662 | 397.6789 | 0.2626 | 563.8521 | 0.2609 | 784.9371 |
| Sample 8 | 0.2367 | 32.4266 | 0.2301 | 89.8743 | 0.2288 | 153.8331 | 0.2265 | 198.9313 | 0.2251 | 287.6735 | 0.2242 | 309.4827 |
| Sample 9 | 0.3166 | 67.5373 | 0.3097 | 132.8321 | 0.3056 | 178.5392 | 0.3022 | 222.6491 | 0.2987 | 298.6932 | 0.2971 | 367.2681 |

**Table 4. Abrasion cycles of all of the knitted cotton fabrics**

| No. of cycles | 0 | 1 | 2 | 3 | 4 | 5 |
|---------------|---|---|---|---|---|---|
| Fabric | Weight (g) | Resistance (Ω) | Weight (g) | Resistance (Ω) | Weight (g) | Resistance (Ω) | Weight (g) | Resistance (Ω) | Weight (g) | Resistance (Ω) | Weight (g) | Resistance (Ω) |
| Sample 1 | 0.2630 | 30.1223 | 0.2543 | 1716.843 | 0.2521 | 198.9421 | 0.2521 | 289.9841 | 0.2521 | 546.9331 |
| Sample 2 | 0.3328 | 92.4711 | 0.3247 | 987.8691 | 0.3236 | 987.8691 | 0.3236 | 987.8691 | 0.3236 | 987.8691 |
| Sample 3 | 0.3195 | 132.1131 | 0.3093 | 25498.92 | 0.3077 | 76982.87 | 0.3051 | 32.6739 | 0.3039 | 42.87389 | 0.3031 | 57.36479 |
| Sample 4 | 0.2497 | 2.67193 | 0.2289 | 15.83899 | 0.2221 | 55.8421 | 0.2221 | 55.8421 | 0.2221 | 55.8421 |
| Sample 5 | 0.2614 | 3.91631 | 0.2299 | 15.83899 | 0.2221 | 55.8421 | 0.2221 | 55.8421 | 0.2221 | 55.8421 |
| Sample 6 | 0.2925 | 6.98013 | 0.2595 | 21.6569 | 0.2544 | 87.0934 | 0.2544 | 87.0934 | 0.2544 | 87.0934 |
| Sample 7 | 0.2664 | 45.68512 | 0.2418 | 587.1589 | 0.2375 | 1298.8914 | 0.2375 | 1298.8914 | 0.2375 | 1298.8914 |
| Sample 8 | 0.2288 | 26.1983 | 0.2084 | 445.8216 | 0.2053 | 781.8491 | 0.2053 | 781.8491 | 0.2053 | 781.8491 |
| Sample 9 | 0.3016 | 77.7818 | 0.2615 | 998.1278 | 0.2580 | 1809.4721 | 0.2580 | 1809.4721 | 0.2580 | 1809.4721 |

**Thickness**

The thickness values were taken from five different places on the coated and uncoated fabrics, and the average of these values was accepted as the thickness of the fabric. The thickness value showed how much of the coating material was deposited onto the surface of the fabrics after the electroless plating process. The results showed that the thickness values are likely to be uniform throughout the surface of the samples, as reported in Table 2, and the graphs of the results are reported in Figure 3.
Figure 3 shows three graphs, which are the thickness percentage, weight gain percentage and electrical resistance of all the samples. In the thickness graph, all the samples showed good thickness percentage, except for the first three samples. The possible reason for the low thickness percentage could be the percentage of elastane content in the fabric. The first three samples consist of 10% elastane, while the remaining samples are 5% elastane. The electrical resistance of samples in the graph showed that Samples # 4–6 have very low resistance, which means excellent conductivity, as compared to the other samples. The other graph shows the weight gain percentage of the samples. It showed that Samples # 4–6 gained more weight percentage after the

Figure 4. Microscopic morphology of all the uncoated and coated knitted cotton fabrics.
Figure 5. Scanning electron microscopy images of all of the coated and uncoated nine samples at 500 × magnification.
Figure 6. Scanning electron microscopy images of all of the conductive nine samples at 100 × magnification and their close view at a magnification of 5kx.
Figure 7. Energy dispersive X-ray spectroscopy images of all nine conductive samples.
plating process. It also showed that Samples # 7–9 gained good percentages after the coating process, but not as much as Samples # 4–6.

**Washing cycles**

We performed washing cycles on the conductive fabric to check its conductivity and measured the weight. Usually, this method is used to investigate the washing durability property of the conductive fabrics. We checked the washing durability up to five washing cycles. The results showed that after five washing cycles, Samples # 4–6 had a lower reduction in conductivity values compared to other conductive samples, and the values were still excellent. The washing cycle test results of all of the samples for this study are reported in Table 3.

**Abrasion cycles**

The abrasion cycle is an essential test and is used to measure a fabric’s ability to withstand abrasion. We checked the abrasion resistance up to 250 cycles. The results showed that Samples # 1–3 showed less abrasion resistance and lost their conductivity after reaching 60 abrasion cycles, and are likely to show no rubbing fastness, whereas Samples # 4–6 showed excellent abrasion resistance, even after reaching 250 abrasion cycles. Samples # 7–9 lost their conductivity values after reaching 150 cycles of abrasion, and these samples are much better than Samples # 1–3. The abrasion tests results are reported in Table 4.

**Microscopic analysis**

By microscopic examination, we investigated the fabric surface morphology of the knitted cotton fabric before and after the coating process to understand how it is affected by the electroless plating process and check the of the coated and uncoated samples, as reported in Figure 4.

**Scanning electron microscopy**

Images of the knitted cotton fabric treated for 20 minutes with copper plating solution are shown in Figures 5 and 6. By comparing the appearance of coated and uncoated knitted cotton fabric shown in Figure 5, the surfaces of the coated fabrics were found to be covered by a bright copper coating. The results were suggested to be caused by the copper metallic materials used in the plating process. The copper coatings seemed to be very smooth in the observation. No peeled-off path was observed. Images of the coated/conductive and uncoated/non-conductive regions on the fabric made by SEM are shown in Figure 5 and SEM images of the coated knitted cotton fabrics at a specific point showing the coated material on the surface of the fabric with a close view are shown in Figure 6. In Figure 5, all of the SEM images of coated fabric showed that there is a good coating over the surface of the cotton fabrics. The images were obtained at 500× magnification of both coated and uncoated samples. In Figure 6, all of the nine coated sample images have been obtained at 100× magnification with their close view with high magnification of 5k×. It was observed by their 5k× magnification that the plating material is properly deposited on every single yarn of the fabric samples, and they have chains that are interlinked with each other.

**Energy-dispersive X-ray spectroscopy**

The surface structures of the samples with copper plating are observed by EDX. The EDX technique was used for a specific point on the surface of the fabric to check the chemical composition on that specific area. In all the figures, it showed that copper, silver, carbon and oxygen are deposited on the surface of the sample and, among them, copper is at a high concentration (more than 90%) and the others are very much lower. This means that copper is well deposited on the surface of the samples. The results are reported in Figure 7.

**Conclusions**

Knitted combed cotton fabrics with 5% elastane and a fine count showed excellent conductivity compared to the other knitted cotton fabric with 10% elastane with a fine count or coarser 5% elastane. The copper metal was successfully deposited on the surface of the knitted fabrics through the copper metal plating method.

We performed all the necessary tests to check the characterization of all the samples. Samples # 4–6, which have a finer count and 5% elastane percentage, showed excellent abrasion resistance compared to other fabric types; Samples # 7–9 also showed good conductivity values compared to Samples # 1–3. Hence, this study proved that lower the elastane percentage in the fabric structure, the higher the conductivity values, and that this also depends on the structure of the fabric. The denser fabric structure is less conductive, while the finer fabric structure has shown excellent conductivity values. The possible reason for this result is due to the structural properties of knitted cotton fabric, which is thick and dense, and the elastane percentage; thus, the plating solution cannot penetrate deep inside the structure of the knitted fabric surface, which
resulted in weak conductive areas within the structure. Thus, knitted cotton fabric with 5% elastane and a fine count is a suitable candidate for different e-textile applications, such as pressure sensors, etc.

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ORCID iDs
Zuhaib Hassan https://orcid.org/0000-0002-0174-2378
Ozgur Atalay https://orcid.org/0000-0003-1050-0685

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