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Copper deposition on fabrics by rf plasma sputtering for medical applications

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Abstract. The present work is about preparation and characterization of RF sputtered Cu films on cotton by the usage of a Magnetron Sputter Source and 99.995% purity Cu target at room temperature. Cotton fabric samples of 1, 2 and 4 min of sputtering time at discharge pressure of 1×10^{-2} Torr and distance between target and sample of 8 cm were used. The main goal was to qualitatively test the antimicrobial action of copper on fabrics. For that purpose, a reference strain of *Escherichia Coli* ATCC 35218 that were grown in TSA plates was implemented. Results indicated a decrease in the growth of bacteria by contact with Cu; for fabric samples with longer sputtering presented lower development of *E. coli* colonies. The scope of this research focused on using these new textiles in health field, for example socks can be made with this textile for the treatment of athlete's foot and the use in pajamas, sheets, pillow covers and robes in hospital setting for reducing the spread of microorganisms.

1. Introduction

The plasma technology has been used to modify a huge range of material surfaces. This occurs because studies have been developed to apply plasma for the processing of textiles. Some important characteristics acquired by the implementation of plasma in fabrics are: the acquisition of hydrophilic or hydrophobic character of the material, the increase in dye absorption capacity [13][17], fiber surface cleaning without chemical reagents or wet treatments, as well as the increase in the absorption and adhesion of other materials like polymers and metals on the fabrics that provide specific finishes allowing coatings with fireproof character, with UV protection, with an increase on the surface hardness and antimicrobial property [1].

The recent development of antimicrobial textiles has become very important in the medical field because it's known that the fabrics provide an excellent medium for the adherence, transfer and propagation of microbial species. The effect of microbial growth on textiles produces generation of body odor, affecting human health and wears out textiles; therefore, different agents are used to provide antimicrobial finishes: dyes, polymers like chitosan or polyethylene glycols, also metal and metal salts where silver, copper and mercury compounds are the most effective biocides [4].



Since March, 2008, US Environmental Protection Agency declared copper as a natural and single metallic antimicrobial material. An important characteristic that copper has is its long lasting antimicrobial property, which after only a few hours of exposition it can significantly reduce pathogenic bacteria. Additionally copper is able to donate and accept electrons, thus developing oxidation and reducing reactions that alter the microbial cells. The concentration of copper would determine how many microorganisms are multiplied or eliminated [9][2].

A research [4] has demonstrated the effectiveness of biocidal properties in the copper-oxide impregnated fabrics. Cotton tests showed a 99 percent reduction of organisms for species like *Staphylococcus aureus*, *Escherichia coli*, *Candida albicans* and a 100 percent reduction in the case of a mite named *Dermatophagoides farinae*. Besides the implementation of plasma sputtering techniques on fabrics has resulted in the inactivation of *E. coli* bacteria. In these cases, where the optimal ratio of Cu-loading/cluster size for *E.coli* inactivation was due to the surface and inside Cu-clusters in the fabric and because of the ionic copper species interacting with the bacteria. Also it has been reported that the modification of the cotton by Cu-depositions changes the surface from hydrophilic to hydrophobic with the increase of the sputtering time [5][15].

Thus the main objective of the present study is to qualitatively test the antimicrobial action of copper on cotton fabrics based on the amount of copper deposited with different sputtering samples prepared by RF Magnetron sputtering.

The importance of this research lies in the application of plasma technology to confer antimicrobial properties in textiles. The main impact would be manufacturing antimicrobial fabrics in medical applications. For example, socks can be made with this textile for the treatment of athlete's foot, the use in pajamas, sheets, pillow covers and robes in hospital setting for reducing the spread of microorganisms or on a daily basis in the house to eliminate dust mites in mattresses, quilts, carpets and pillows, thus helping and improving the quality of life in people that suffer asthma and allergies [8].

2. Methodology

2.1. Materials

The fabric used in the experiment is 100% cotton. Cotton fibers are composed 99% of cellulose and non-cellulosic constituents such as: proteins, wax, organic acids, sugar, and others [10]. The cotton samples had an initial size of 10x10 cm and were cleaned by isopropyl alcohol immersion for about 1 hour and were dried in vacuum. For this study, 3 samples were prepared with different sputtering times and a control sample without deposition was used.

2.2. Copper Deposition on Cotton samples

Copper layers were deposited on cotton substrate by RF magnetron Sputtering Gun at room temperature, in a cylinder chamber with an automatic vacuum station. This technique consists on the bombardment of a solid target using energetic particles, mostly ions, applying a radio frequency power supply [6][7][11]. The important parameters established were: the distance between the Cu-target and the cotton substrate was ~8 cm, Argon as gas, a discharge power of 50 W, with a base pressure of 1×10^{-4} Torr, a discharge pressure of 1×10^{-2} Torr, also a Cu-target with 99.995% purity and with 1 inch of diameter, and the deposition times were 1, 2 and 4 min. After the deposition, the edges of cotton samples were eliminated and their sizes shrank to 3x3 cm and finally these samples were stored in sterilized containers. The experimental configuration of the plasma discharge is shown in figure 1.

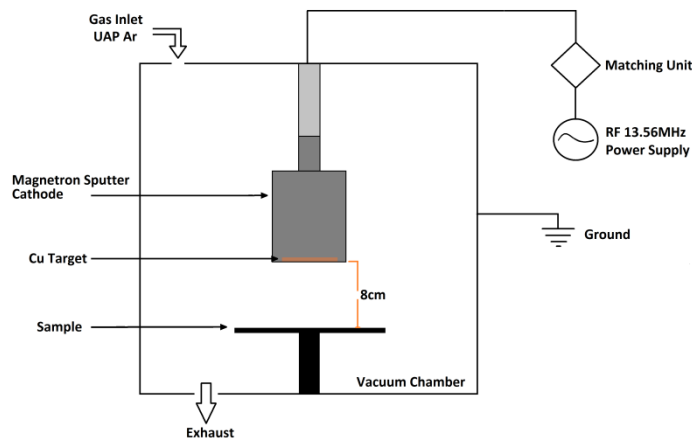


Figure 1. Experimental set up.

2.3. Fiber Characterization Technique

The Scanning Emission Microscopy (SEM) was used to investigate the surface condition and structure of cotton fabrics. This technique consists on the scanning of the sample surface with a beam of electrons and obtaining a three dimensional image [3][14][16].

2.4. Antimicrobial Susceptibility Test

The bacterium used for testing the cotton samples was *Escherichia coli*; its characteristics are: it belongs to the *Enterobacteriaceae* family, it is gram negative, it doesn't form spore, it is motile by means of peritrichous flagella or may be non-motile, facultatively anaerobic, chemo-organotrophic microorganism with an optimum growth temperature of 37°C [20]. Also it is one of the predominant enteric species in the human gut and part of the normal intestinal flora; some of these species provide benefits to the host; however, there is a small group known as pathogenic *E.coli* that can cause severe diarrheal disease in humans [21].

This study used a strain of *E. coli* ATCC 35218. The bacteria were inoculated into a TSA (Tryptic Soy Agar) plate. The cotton fabric samples were placed on a petri dish immediately after the inoculum was done. The provided petri dishes were covered with a lid to prevent contamination and evaporation, and were incubated at 35 °C for 24h. After 24 hours at the incubator the fabric was removed from each plate and the contact area of the fabric was observed. For a better visualization of the bacterial growth after incubation, a stereoscopic microscope (Leica EZ4) was used.

3. Results

3.1. Cotton Fibers

Figure 2 shows cotton fibers without Cu sputtering. The initial cotton structure is braided thus resulting in a nonuniform surface. Also, fiber thicknesses are different with an average of 24,15 μm . Braiding can affect the uniformity of the deposited copper layer fabric. But some investigations reported that a rougher surface of the deposited copper fibers improves the adsorption and stabilizes copper ions thus contributing to the inactivation of the bacteria [5][15].

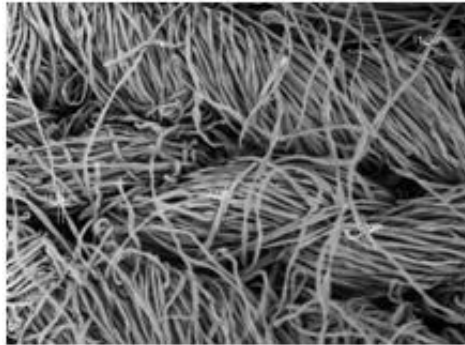


Figure 2. Cotton fibers without Cu sputtering.

3.2. Copper Deposition Cotton

Figure 3 shows sputtered cotton samples for 1, 2 and 4 minutes. Cotton shows light grey color because Cu-content increases with time. The layer color is uniform throughout the sample.

The possible reactions between Ar-plasma and cellulose have been studied. The suggested mechanism for argon plasma induces molecular fragmentation of cellulose and the generation of free radicals through the oxidation process [10]. Therefore this activation process of the cotton fabric may help the adherence of copper ions and the formation of the film [18][19].

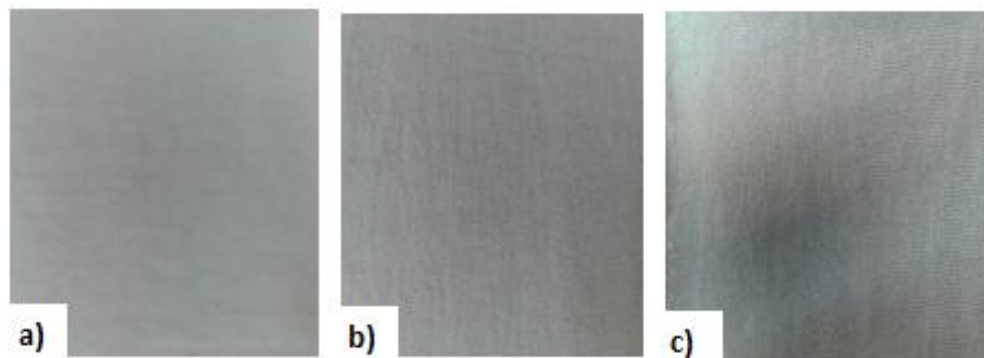


Figure 3. Samples of Cu-cotton sputtered for: a) 1 min, b) 2 min, c) 4 min.

3.3. E.coli Bacterial Inactivation

As shown in figure 4, the bacterial growth appeared as the formation of individual and agglomerated colonies. The picture below shows where the fabric was placed in each test. The fabric was placed on the center for the images on the left whereas the ones on the right show the trace of the edge of the fabric.

As for the pictures on the right, the bacteria did not cross the edge area as the samples show longer time of sputtering. In picture a), growth of bacteria in and out of the edges was observed; in pictures b) and c) there was a bit of bacterial growth beyond the edge, but the concentration of bacteria colonies is focused around the border, while in picture d) there is insignificant bacterial growth within the area.

On the left side images, the brown regions representing lower growth of bacteria decrease as the sputtering time of samples increases. In picture a) there are more brown regions and they are distributed uniformly in the area where there may be bacteria colonies. In picture b) lower brown regions are observed, but there is still a significant group of bacteria colonies. In picture c) and d) this area becomes clearer with lower brown regions that represent the decline in the colony growth.

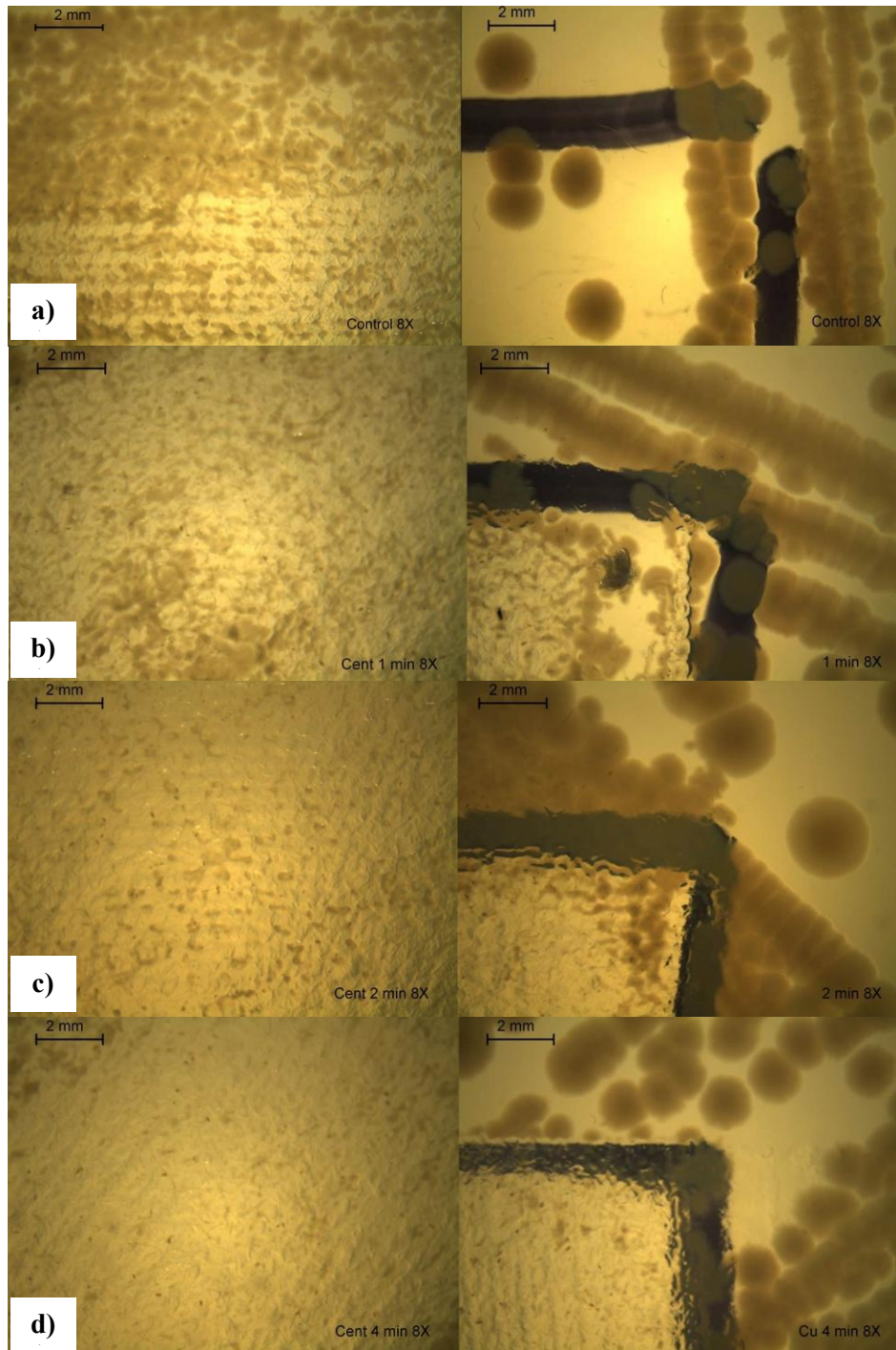


Figure 4. Bacterial growth results in the contact area for each sample:

a) control, b) 1 min, c) 2 min, d) 4 min.

The mechanism by which copper inhibits *E. coli* is not exactly known but theory explains some possible reactions that may help copper against this bacteria. Copper toxicity to the bacteria is due to the reactions that can be performed by its tendency to alternate their ions, mainly its cuprous Cu(I) and cupric Cu(II) oxidation states [22].

The conditions under which the antimicrobial test is developing affect the inhibition rate and destruction of the bacteria. If the medium is under anaerobic conditions, the contact between the cells and copper surface may cause the reduction of Cu(II) to Cu(I) and the oxidation of Cu(I) to Cu(II), where Cu(I) is more toxic than Cu(II) and stable to these conditions. While if the aerobic condition is presented, the effect of copper is amplified because a redox cycling is produced and leads to the generation of reactive hydroxyl radicals and oxygen species that can damage biomolecules, which is the case studied [22][23][15]. An important aspect to consider is that probably the copper inactivation mechanism is due to the joint action of both ions, not just one.

4. Conclusions

A decrease in the growth of bacteria on contact with Cu was observed; the fabric samples with longer sputtering presented lower development of *E. coli* colonies due to the higher concentration of copper.

The center of the fabric tends to be the best part to analyze bacteria, however, good results in the edge with respect to bacterial growth are presented as well.

The antimicrobial property of copper was evidenced on cotton fabrics, but it was not possible to identify a sample with a sputtering time and optimum concentration of copper to achieve total inactivation. Hence, a subsequent study regarding the quantitative characterization of the observed effect must be applied.

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