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# A Green Dry Route for Antibacterial Nanofinishing of Textiles

**Esther Hontañón, Einar Kruis**
Institute for Technology of Nanostructures, University of Duisburg-Essen
Bismarckstr. 81, 47057 Duisburg, Germany
esther.hontanon@uni-due.de; einar.kruis@uni-due.de

**María Blanes**
Department of Technical Finishing and Comfort, AITEX Technological Textile Centre
Pl. Emilio Sala 1, 03801 Alcoy, Alicante, Spain
mblanes@aitex.es

**Laura Santos**
Foundation for the Promotion of the Textile Industry of the Valencian Community, FOMENTEX
C/Els Telers 20, 46870 Ontinyent, Valencia, Spain
laura@fomentex.eu

**Linus Ludvigsson, Axel R Persson, Maria E Messing**
Solid State Physics, Lund University
Box 118, 221 00 Lund, Sweden
linus.ludvigsson@ftf.lth.se; maria.messing@ftf.lth.se

***Abstract*** **-**In this paper, an aerosol-based process is proven for imparting antibacterial property to textiles. Nanoparticles of copper and silver are produced by means of glow discharge between two electrodes in nitrogen at atmospheric pressure and passed through fabrics of cotton and polyester which act as filter media. The bactericidal performance of the nanofinished fabrics is assessed against Gram-positive *Staphylococcus aureus* and Gram-negative *Klebsiella pneumonia* bacteria. Nanoparticle loads of about 0.3 g/m$^2$ (~ 0.2 wt %) give strong antibacterial activity to the fabrics regardless of particle composition and size. Higher loads do not enhance the bactericide property but affect the colour and hand feel of the fabrics. Cotton and polyester fabrics with low loads (<0.3 g/m$^2$) of small silver nanoparticles (<10 nm) show different bactericidal behaviour. Polyester fabrics attain good antibacterial activity for SA and KP, which is retained (KP) or decreases (SA) after washing. Cotton fabrics show hardly bactericidal property but, in some cases, it increases after washing.

***Keywords*****:** glow discharge, metal nanoparticles, aerosol filtration, textile nanofinishing, antibacterial activity, washing durability

## 1. Introduction

Antimicrobial nanofinishes rely on dispersions of the active nanoparticles, generated by using chemical precursors, in solvents containing surfactants, stabilizers and binders. Nanoparticles are applied to fabrics commonly by using a dip-pad-dry-cure method which has several disadvantages. The chemicals are expensive and drying and curing incur high energy consumption and increase costs. The dispersions lead to effluents carrying harmful chemical compounds and nanoparticles, thereby enhancing waterwaste treatment costs. Besides, surfactants cover the nanoparticle surface reducing its activity and polymeric stabilizers and binders degrade air permeability and lead to stiffer hand feel of the treated fabrics.

An aerosol-based process was investigated for antibacterial nanosilver finishing for cotton fabrics in a previous work (Hontañón et al., 2014a). Here, the process is used to apply nanoparticles of copper and silver to fabrics of cotton and polyester and the antibacterial activity of the fabrics is assessed. Washing tests are performed to gain insight into the strength of the adhesion of metal nanoparticles to fabric fibers. The aim is to optimize the size and load of metal nanoparticles imparting the fabrics relevant and durable bactericidal property without affecting other fabric properties. Hazardous chemicals and solvents are neither used nor produced in the aerosol process, and the gas is cleaned and recycled.

## 2. Methods

The setup consisting of an aerosol generator and a gas recycling line is sketched in Fig. 1. This has been described elsewhere (Hontañón et al., 2014b). A fabric sample (20 cm x 35 cm) is installed on the inner surface of a hollow cylindrical HEPA filter enclosed in a vacuum tight housing. The particle size distribution of the aerosols is measured by using a scanning mobility particle sizer (SMPS) together with a condensation particle counter (CPC).

Fig. 1 shows SEM images of the textiles used here. They consist of microfibres (10-20 µm) of natural cellulose (cotton) and of a polymer (polyester) and have different structure and weight (140-150 g/m$^2$ cotton and 200 g/m$^2$ polyester). The bactericidal activity of the textiles treated with metal nanoparticles is assessed by means of the standard ISO 20743 quantitative method (Hontañón et al., 2014a). Values of the antibacterial activity between 2 and 3 indicate *"significant"* antibacterial property and values above 3 correspond to *"strong"* bactericidal property.

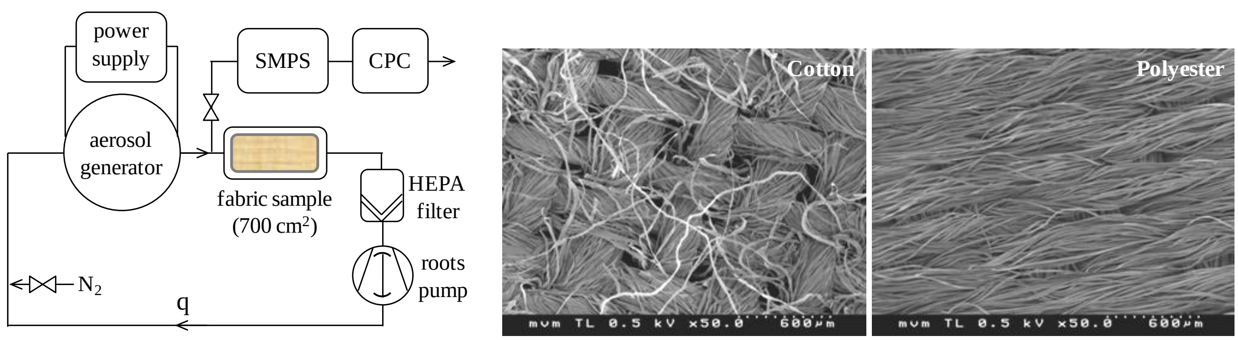


Fig. 1. Setup for applying metal nanoparticles to textiles and SEM micrographs of textiles.

## 3. Results

The flow rate of the gas carrying the nanoparticles is a key parameter, since the particle retention efficiency of the fabric decreases with increasing filtration velocity (gas flow rate / fabric surface area). Nanoparticles of copper are produced by glow discharge in nitrogen at flow rates of 500 lpm and 100 lpm and applied to cotton fabric samples (700 cm$^2$). Filtration velocities are 12 cm/s and 2.4 cm/s, respectively. No change in either the colour or hand feel of the cotton fabric is noticed in the experiment at a flow rate of 500 lpm. After treatment with nanocopper in the experiment at a flow rate of 100 lpm, the cotton fabric is dark grey and rougher to the touch than the untreated fabric.

Fig. 2 displays the aerosol particle size distributions measured in the experiments and SEM images of copper nanoparticles deposited on the fabrics. An even layer of nanoparticles less than 30 nm is observed in the fabric treated at a flow rate of 500 lpm, whereas lower surface coverage and larger agglomerates are found in the fabric treated at a flow rate of 100 lpm. The estimated load of nanocopper is 0.3 g/m$^2$ and 1 g/m$^2$ in the first and second experiments. The bactericide property of the treated cotton fabrics was assessed against *Staphylococcus aureus* (SA) and *Klebsiella pneumonia* (KP). Both samples attain high antibacterial activities of 5.54 for SA and 5.52 for KP (the threshold is 2).

Then, a series of experiments is conducted to determine the load of nanosilver imparting the fabrics strong bactericide property without affecting their colour and hand feel. To that aim, the process parameters are adjusted to attain the highest particle retention efficiency. Table 1 shows the mean size of the aerosol silver particles and the load of nanosilver in cotton (CO) and polyester (PES) fabrics. The colour of fabrics with loads >0.5 g/m$^2$ (CO5, CO6 and PES7) turns out orange and brown.

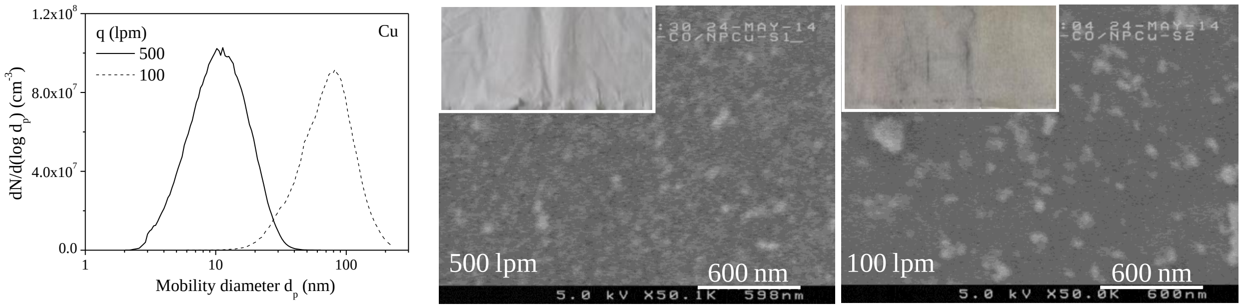


Fig. 2. Particle size distribution of copper aerosols and SEM micrographs of cotton fabrics treated with nanocopper by aerosol filtration (pictures of the treated cotton fabrics are shown in the top left corner).

Table 1. Mean size and load of silver nanoparticles into fabrics of cotton and polyester.

| | CO 1 | CO 2 | CO 3 | CO 4 | CO 5 | CO 6 | PES 1 | PES 2 | PES 3 | PES 4 | PES 5 | PES 6 | PES 7 | PES 8 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Particle size (nm) | 15 | 8 | 6 | 6 | 6 | 6 | 6 | 6 | 4 | 6 | 4 | 10 | 15 | 6 |
| Particle load ($g/m^2$) | 0.15 | 0.29 | 0.10 | 0.26 | 0.51 | 1.56 | 0.02 | 0.30 | 0.02 | 0.32 | 0.02 | 0.18 | 2.2 | 0.36 |

A high resolution SEM (Hitachi SU8010) equipped with a field emission gun was used to image the particle-coated textile samples. To enable imaging, the samples were coated with palladium and imaged at an operation voltage of 4 kV. As displayed in Fig. 3 a reasonably homogenous coating of silver particles was found on the samples. From the images it is also clear that the particles have a certain spread in size.

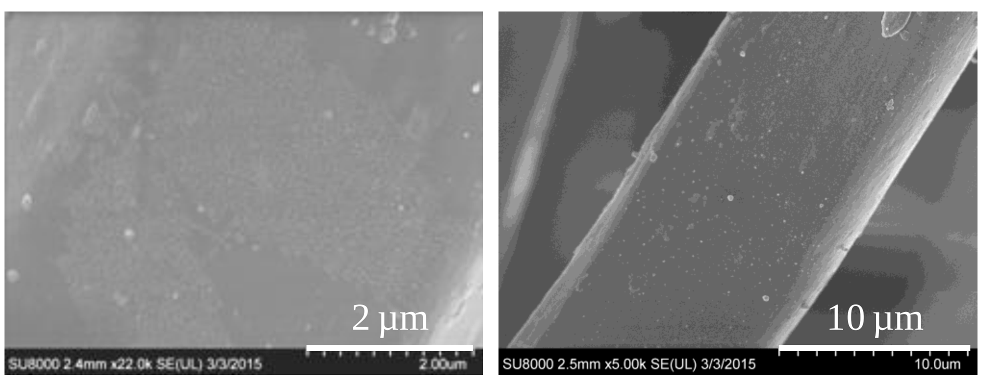


Fig. 3. SEM micrographs displaying a reasonably homogenous coating of silver nanoparticles on polyester fabric (PES8).

Fig. 4 displays the values of the bactericidal activity measured for the fabrics coated with nanosilver. Washing tests were addressed for a few samples; the values of the antibacterial activity of the washed fabrics are shown in Fig. 4 too. Polyester fabric performs quite well against KP. The antibacterial activity does not increase with nanosilver load above 0.02 $g/m^2$ and high antibacterial activity (3.5 - 5) is retained after 10 washes. The antibacterial activity of polyester fabric against SA is more sensitive to nanosilver load and falls down to values close to 2 for loads below 0.1 $g/m^2$. It decreases significantly after washing. Cotton fabric shows poorer bactericidal function than polyester fabric. Higher nanosilver loads are needed for cotton to attain strong antibacterial activity; values in the order of 4.7 are attained for both bacteria with loads above 0.5 $g/m^2$, which are retained after 10 washes. Cotton fabrics with nanosilver loads less than 0.3 g/m² do not show meaningful antibacterial activity. The different bactericidal performance of cotton and polyester fabrics is attributed to the different filtration behaviour and/or fibre composition. Also, in some cases, the antibacterial activity of the fabrics increases after washing. The explanation is that fabrics contain substances like starch used for stiffening fabrics which favors the growth of bacteria. These substances are easily removed by washing and bacteria are then more effectively killed by remaining nanosilver.

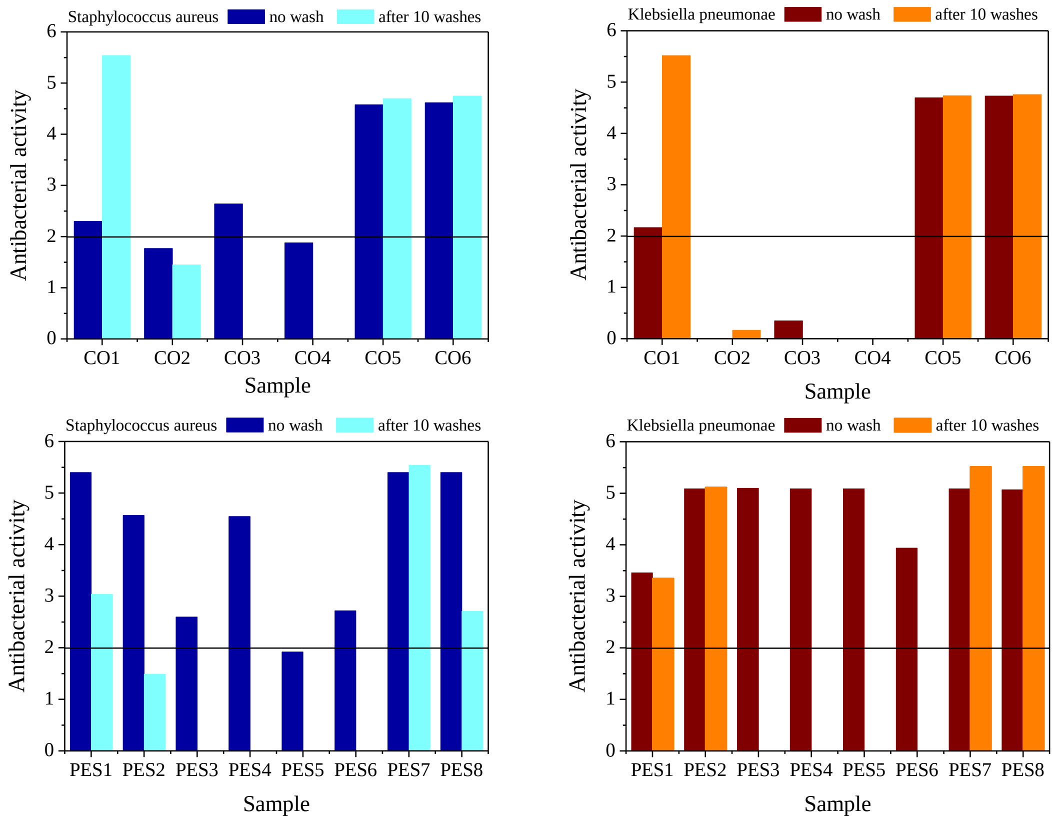

Fig. 4. Antibacterial activity of cotton (top) and polyester (bottom) fabrics treated with nanosilver

## 4. Conclusion

An aerosol process has been proved for antibacterial nanofinishing of textiles. This environmental friendly dry route may be a suitable alternative to wet routes for nanofinishing of textiles.


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