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# Efficient silver nanoparticles deposition method on DBD plasma-treated polyamide 6,6 for antimicrobial textiles

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#### Introduction

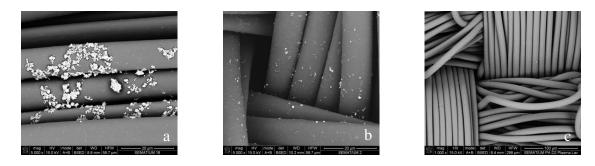
The study of antimicrobial fabrics with silver nanoparticles (AgNPs) incorporation has shown excellent properties in medical, pharmaceutical, cosmetics and electronics applications due to their formidable action against pathogens, preventing and treating infections.[1] The high surface-to-volume ratio from AgNPs promotes an easy release of silver ion, responsible for the antimicrobial effect.[2] The most traditional method for nanoparticles deposition onto fabrics is the pad-dry-cure technique.[3] Other methods were developed such as dip coating, electrochemical methods and layer-by-layer depositions.[4-6]. However, the major methods have several limitations for noble metals. In this work, several methods for AgNPs deposition on Dielectric barrier discharge (DBD) plasma pre-treated polyamide 6,6 (PA66) were tested for the production of durable antibacterial textiles. DBD plasma was previously used for surface modification to increase surface energy through the introduction of polar groups altering wettability and roughness.[7] However, the study for an efficient deposition methods after plasma treatment was disregarded. SEM, XPS, cytotoxicity and antimicrobial tests were performed to evaluate the different deposition methods.

#### Materials and methods

Commercial pre-washed pure PA66 (warp 50, weft 32 threads cm<sup>-1</sup>, areal density of 110 g m<sup>-2</sup>) woven fabrics were used in this study. The DBD plasma treatment was conducted in a semi-industrial prototype (Softal/University of Minho) working at RT and 1 atm with a dosage of 2.5 kW min m<sup>-2</sup>. NPs depositions were performed by dip coating, exhaustion, ultrasonic tip/magnetic stirring or spray. Morphological and chemical analyses were carried out with an Ultra-high resolution FEG-SEM, NOVA 200 Nano SEM, FEI Company. The X-ray photoelectron spectroscopy (XPS) analyses were carried out on the PHI-TFA XPS spectrometer (Physical Electronics Inc.). Antimicrobial efficacy was evaluated by Log reduction. AgNPs dispersions were prepared with 10  $\mu$ g/ml of 30 nm silver nanoparticles by ultrasonic bath or ultrasound tip for 30 minutes each.

#### **Results and discussion**

DBD plasma treatment on PA66 was optimized for 10 passages at the power of 1 kW and velocity of 4 m/min. Four methods for AgNPs deposition onto the PA66 were used: i) simple dip coating; ii) ultrasonic tip/magnetic stirring, iv) spray system v) exhaustion at 30°C and 70°C. FTIR analysis displayed an increase in the number of carboxylic acid groups at 1743 cm<sup>-1</sup> with the increase in the number of plasma passages. XPS results clearly show the new oxygen species on the fabric surface due to plasma treatment and that the higher amount and less aggregate AgNPs were deposited using the exhaustion method at 30°C (Figure 1b). Washing fastness was also evaluated after 5 washing cycles at 75°C for 15 min with 0.1gL<sup>-1</sup> non-ionic detergent and a 1:30 ratio bath. AgNPs deposited by plasma showed higher washing fastness than untreated samples (figure 1c). Preliminary results show a significant reduction in bacterial and biofilm growth. Cytotoxicity of samples is quite high but mostly due to the type of fiber.



**Figure 1.** SEM images of deposited AgNPs on the DBD treated PA66 by a) ultrasonic tip/magnetic stirring b) exhaustion process at 30°C c) tip ultrasound after washing.

# Conclusion

The results clearly show that the exhaustion method at 30°C is the best option for the deposition of a uniform and high amount of AgNPs onto PA66 fabrics.

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# References

[1] Mitra C, Gummadidala P, et al. 2017 Environmental Science & Technology 51 (14) 8085–8093

- [2] Surmeneva M, Sharonova A, et. Al 2017 Colloids and Surfaces B: Biointerfaces 156 104-113
- [3] Toh H, Faure R, Amin L, Hay C, George S, 2017 Nanotecn., Science and Applications 10 147-162
- [4] Budama L, Çakır B, Topel O, Hoda N, 2013 Chemical Engineering Journal 228 489-495

[5] Wildgoose G, Banks C, Compton R, 2006 Small 2 182-193

[6] Dubas S, Kumlangdudsana P, et. Al 2006 Coll. and Surf.: Physicochem. Eng. Aspects **289** 105-109 [7] Seidelmann L, Bradley J, Ratova M, 2017 Applied Sciences **7(4)** 337

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# **Options indication**

1. Oral

2. Nanotechnology, Nanotextiles, Electrospinning