Research Article
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Functionalization of fabrics by Ag-TiO2 Nanoparticles deposition by sol-gel method

Abstract

Nanotechnology's expanding global applications, especially in textiles, integrate various disciplines within textile engineering. Research primarily concentrates on incorporating nanomaterials, like titanium dioxide and silver, into textile substrates for enhanced functionalities such as self-cleaning, UV protection, and antimicrobial properties. Metal oxide nanoparticles, particularly Ag and TiO2, exhibit remarkable efficacy in combating microorganisms. Sol-gel techniques play a crucial role in surface modification, facilitating better adhesion of nanomaterials and enabling diverse applications in materials science. This study focuses on utilizing silver and titanium nanoparticles for antimicrobial and dirt-repellent properties in lightweight fabrics, showcasing a preliminary step towards uniform incorporation for subsequent evaluation.

Keywords: fabrics, nanoparticles, textile industry, shirts

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Artemísia Lima Caldas,[1] Jefferson Sousa,[2] Edgar Carneiro,[3,4] Sandra Carvalho[4]
[1]Department of Design, Fashion and Styling at the Federal University of Piauí, Brazil
[2]Physics Center of Minho and Porto Universities, Physics Department, University of Minho, Guimarães, Portugal
[3]Centre for Mechanical Engineering, Materials and Processes, Department of Mechanical Engineering, University of Coimbra, Portugal
[4]LED & MAT-IPN—Laboratory for Wear, Testing and Materials, Instituto Pedro Nunes, Coimbra, Portugal

Correspondence: Artemísia Caldas, Department of Design, Fashion and Styling at the Federal University of Piauí, Teresina, PI, Brazil, Email artecalda@ufpi.edu.br

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Introduction

Nanotechnology is increasingly being used throughout the world due to its wide range of functionalization and economic potential. In the textile industry, it presents a very comprehensive and potential field, as it brings together many areas of study within textile engineering such as medical textiles, technical, cosmetic, among other emerging technologies involving fibers.

In this particular sector, research is mainly focused on the use of nanomaterials in manufacturing processes, in order to produce nanostructures with special functions or improved properties, as highlighted Joshi and Bhattacharyya.[1] The inclusion of nanomaterials in textile substrates can be made by adding it to the fiber structure itself during the manufacture or through post-production surface treatments and coatings.[2] Nanomaterials often used in the textile industry are mostly inorganic compounds, including titanium dioxide (TiO_2), zinc oxide (ZnO), silver (Ag), copper (Cu), gold (Au),[3] silica (SiO_2) nanoparticles,[4] carbon nanotubes (CNT),[5] clays and nanolayered coatings and also non-metallic nanocomposites.[6] These materials have specific applications, providing textile substrates with features never seen before.

Many metal oxide nanoparticles are reported as having photocatalytic ability, conductivity, UV absorption, photo chemical oxidation and antimicrobial function. These nanoparticles have higher efficiency when compared to particles in the micrometric scale, since they are able to reach a larger surface area when deposited on the surface of a textile substrate.[7]

Several properties identified above, can be applied to textile substrates. According to some authors,[8] the current applications for nanomaterials are diverse and of interdisciplinary nature, providing a single substrate of textile material with properties such as self-cleaning and antimicrobial capacities.

Textiles can be equipped with flame retardant properties, an important requirement as a feature for fire-fighting technical clothing. UV blockers, other possibility, may either be organic or inorganic, its function is to limit the rate of tissue yellowing. Inorganic blockers are usually semiconductor oxides such as TiO_2, ZnO and Al_2O_3. These materials act on tissues, absorbing ultraviolet radiation, preventing it to be transmitted to the skin, an application that can be targeted to a technical level, in uniforms and gloves, for example, but may also be intended for common use in outerwear, as shirts, t-shirts, as well as applied in bathing suits, to protect the areas of the body devoided of any sun protection factor.[8]

Self-cleaning properties, can be incorporated in textile, a property that allows removal of stains more easily. The self-cleaning of materials is based on the incorporation of silver nanoparticles and titanium dioxide , which are capable of reacting with sunlight and promote degradation of organic materials (dirt).[9]

Antimicrobial properties are another example of functionality that can be incorporated into textiles. Studies showed[10] that properties in the tissues are improved in order to protect users from the spread of bacteria and diseases. The level of protection is also extended to fungi, yeasts and microorganisms like dust mites. Ag and TiO_2 nanoparticles are nowadays widely applied in textile industry.

It is observed then that these two materials, Ag and TiO_2, are being applied in the textile sector in various areas and are used to provide textile substrates with functional characteristics.

According to Rai and Yadav,[11] since ancient times, silver has almost always been the material used to fight microorganisms such as bacteria, fungi, yeasts and other eukaryotic genera in antimicrobial applications. Others[12] complement stating that their use in nanometric dimensions increases the number of particles per unit area that maximizes its effects.

Ag salts when transformed into nanoparticles, have their role enhanced, becoming insoluble in aqueous medium, an important feature for application in textiles.[13] Note that there are various techniques for the preparation of silver nanoparticles, such as photo catalytic reduction, chemical reduction process, micelles methods and even biological methods. Titanium dioxide has stable properties, is considered non-toxic[14] and is appropriate for UV protection applications,[15] for their optical properties,[16] and also for biological applications.[17]

Literature emphasizes that TiO_2 is a semiconductor with optical absorption in the UV (<400 nm). This enables its use for UV protection and can also be incorporated into textiles. Other applications for material lay in the areas of health, such as antimicrobial functionality, but is also used in the areas of security, home textiles and cosmetics. There are three known crystalline phases of TiO_2 (anatase, rutile and brookite), the largest surface area is in the anatase phase, which is more applicable in photocatalytic properties. The rutile phase is considered more stable than anatase and brookite, at higher temperatures, which allows its use, especially for pigment production in the industry.[13]

The surface modification of textile materials are of great importance. It allows for a greater deposition rate and adhesion of nanomaterials deposited on textile surfaces. It also provides mechanical resistance to wear caused by the washing processes, becaming an essencial step for functionalizing textiles. In addition the development of fibers with increasingly smaller diameters also enabled the development of finishes at the nanoscale.[3] Various technologies can be used to promote adhesion of nanoparticles, fibers, composites, polymers and nanofibers onto substrates. Sol-gel coating of textile with nanometric silica (particles less than 50 nm), chemically or physically modified, for example, enable the change in mechanical, optical, electrical and biological properties by incorporating different compounds together.[18]

Sol-gel technique is one of the methods widely used in materials science. It is a process used in the textile field, where the surface coating occurs through immersion.[19] The process of sol-gel dip is applied almost exclusively for the manufacture of transparent layers. This method is based on the preparation of colloidal suspensions and nanosols, from precursors suitably selected.[20]

The functionalization of tissues through the deposition of silver and silica by using the sol-gel method, becomes an increasing widely used procedure, since it is a low-cost method, simple in its application and can be uses with a wide range of substrates.[21]

This study investigated the use of silver and titanium nanoparticles to achieve both antimicrobial properties and dirt repellency. Combining them leveraged silver‘s well-known antimicrobial activity and TiO2‘s self-cleaning properties, potentially leading to enhanced UV protection as well.[22] This is a preliminary work where it is intended to validate a method to achieve a better uniformity in the incorporation of NP‘s in the selected substrates. Posterior evaluation of these features occurs only for samples that demonstrate better results in this first stage. The fabrics are light weight, suitable for clothe manufacturing.

Materials and methods

Nanoparticle preparation

Based on work by Deliang Chen[23] titanium oxide nanoparticles doped with silver were synthesized by the addition of 0.2 g of Degusa P25 to 0.186 mL of a 0.1M solution of $AgNO_3$ (Titrisol® - Merck) After constant stirring for 10 minutes, 2mL of ethanol were added. The solution obtained was then exposed to sunlight for 5 hours, having undergone a change in coloration from white to dark gray. Finally, the activated solution was diluted in ethanol until a volume of 100 ml obtained.

Surface preparation - textile activation

Two types of cloth: cotton (100% CO) with a weight of 141 gm^{-2} and mixed cotton with polyester (50% CO, 50% PES) with a weight of 122 gm^{-2}.

Prior to alkali activation procedure, several samples of both substrates were cut into small rectangular pieces of 20×30 cm. The substrates were immersed for 24 hours in alkaline solution (10 g of sodium hydroxide (NaOH) in 1L of water). After the immersion period, the samples were removed, washed, and held dried at a temperature of 60 °C for a period of 2 hours. Reweighing results obtained were: 7.79 gm^{-2} for the 100% CO cloth and 7.81 gm^{-2} for the mixed one.

Textile functionalization

With the previously described activated fabrics in hand, a dipping process was carried out in two different procedures (S1 and S2) to access the most suitable method for functionalization of the cloths with nanoparticles:

In S1 procedure, the samples were reduced to the size of 10×10 cm, in order to increase the number of samples. A set of samples of 100% CO and a set of CO/PES were weighed before dipping in the solution. The samples were then subjected to bath stirring for 10 minutes. After dipping CO and CO/PES samples were dried in an oven at 80 °C for 15 min and 10 min respectively.

For S2 procedure, it was decided to reduce the sample size to 5×5 cm. Samples were also weighted. The solution was heated at 60 °C during 20 min. Before dipping the solution was stirred and heated for 5 min, while standing at rest for 15 min for deposition of NP’s in the fabric. After, they were placed inside an oven at 60 °C during 30 min for CO samples and 15 min for the CO/PES samples.

Air permeability

ISO 9237: 1997 “Textiles: Determination of the permeability of fabrics to air.” was followed to determine air permeability in the cloth. The apparatus used in the experiment was a Textest FX 3300 Air Permeability Tester and for each sample were made 10 trials and 100 Pa pressure accordingly the standard.

Kinetic friction coefficient

The FRICTORQ apparatus developed in UMinho University was used.[24] Its operating principle is based on rotating actuation which allow measuring the coefficient of dynamic friction between the sample surface and the metalic surface (standard contact element), thereby enabling the evaluation of smoothness or roughness degrees in a textile sample.

Hydrophilicity

The contact angle of distilled water drop deposited on the surface of the textile substrate was measured with a goniometer. By measuring the absorption time of the water droplet, according to the standard AATCC - 70-2000, we evaluate the wettability parameters and absorption in the textile tissues. It allows us to estimate the hydrophilicity or hydrophobicity of the pre-treated samples and in the ones subjected to S1 and S2 procedures.

Scanning electron microscopy

By means of scanning electron microscopy (NanoSEM – FEI Nova 200) the morphology of the various stages of sample production were observed. Since the substrates were non-conductive a thin coating of Au-Pb (80-20 wt %) was deposited on top of the samples in order to facilitate image acquisition, (208HR - Cressington Company, linked to a high resolution MTM - 20 Cressington and with thickness controller).

Results and discussion

For a better understanding and readability, hereinafter, the samples 100% CO and CO/PES will be named A and B respectively. Furthermore, each stage of functionalization will have and equivalent suffix after the sample name. 1 – for non-treated (NT) or as-bought samples; 2 – for samples pre-treated (PT) in alkaline solution; 3- for samples subjected to procedure S1, and finally; 4 – for samples submitted to procedure S2.

Air permeability

Textile fibers are impermeable to air, the passage of air through a fabric can only take place through space among fibers and yarns.[25] Air permeability is mainly affected by the characteristics of pore in the fabric.[26]

Air permeability results are strictly related to the degree of porosity in the samples which is also related to nanoparticles deposition onto the fabrics. More specifically it can be said that when lower porosity is obtained, that indicates a successful pre-treatment with highest deposition of nanoparticles on the surface of the textile.

The data, in Figure 1, shows a consecutive decrease in air permeability coefficient after each step. In view of the desired functionalization, a lower porosity and higher surface area is important as it allow a larger number of nanoparticles to be deposited on the surface of the material. In the pre-treated samples (A2, B2) a decrease 41,26% and 18,28% respectively in permeability is noted when compared to the as-bought samples (A1, B1). Past this step, we verify that samples exposed to procedure S2 (A4, B4) show a lower air permeability coefficient 15,28% and 30,89% respectively when compared to S1 (A3, B3). Meaning that, S2 provides to the fabrics a larger surface area for nanoparticle deposition than S1.

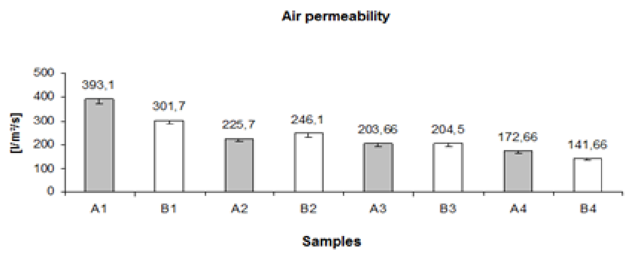

Figure 1 Air permeability samples.

A1 - (NT), A2 - (PT), A3 - (S01), A4 - (S01), B1 - (NT), B2 - (PT), B3 - (S01), B4 - (S02)

Figure Caption: Authors (2014)

We observed that after alkaline pre-treatment a reduction in the quantity of pores and interstices was obtained. Consequently, there has been an increase in surface area of cellulose fibers, specifically CO for both samples, due to modification of its geometry, which explains the decrease of air permeability.

For polyester fibers such treatment only promotes the removal of dirt. This treatment also indorses a degree of temporary hydrophobicity in cellulosic fibers. And, since it reduces the space of the interstices, the fibers have lower absorption power.

Kinetic friction coefficient

The surface of a textile fabric is not uniformly flat and smooth.[27] The coefficient of kinetic friction alone may be insufficient for surface characterization generally a smooth fabric is the one that possesses a low coefficient of kinetic.[28]

Firstly, in Figure 2, for both sets of samples, A and B, the kinetic friction coefficient, measured with FRICTORQ, increases after alkaline pre-treatment. Also, one can observe that samples made of 100% cotton show a more visible increase in kinetic friction coefficient between pre-treatment and S1 than the mixed fabric samples. In the later, the increase, although noticeable, presents itself smaller. These results prove procedure S1 as more efficient for cotton samples, since a greater kinetic friction coefficient indicates a higher fixation rate of nanoparticles in the textile material.

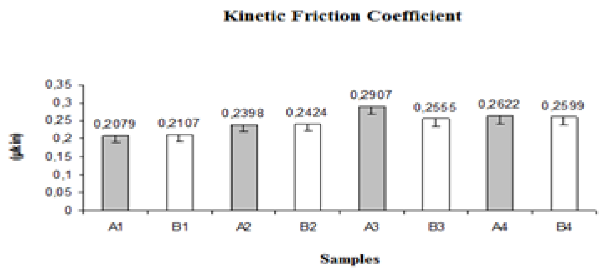

Figure 2 Kinetic friction coefficient.

A1 - (NT), A2 - (PT), A3 - (S01), A4 - (S02), B1 - (NT), B2 - (PT), B3 - (S01), B4 - (S02)

Figure Caption: Authors (2014)

S2, proves itself less efficient for pure cotton (A4) samples with a lesser 9,8% of value of KFC when compared to A3 from S1 treatment, thus showing a lower ability to nanoparticles adhesion. Both S1 and S2 procedures, in mixed CO/PES samples show a very similar value of KFC. One can affirm that S1 is in general more appropriate for induce nanoparticle fixation in the fabrics.

Hydrophobicity

Hydrophilic, water-loving materials are substances with Contact Angle lower than 90° while hydrophobic ones are water hating, displaying a Contact Angle higher than 90°. As stated earlier, super-hydrophobic characteristics are attained when the water contact angle is greater than 150°.[29] The wettability of a solid surface is a characteristic property of materials and depends strongly on both the surface energy and roughness.[30]

The contact angle measurements, Figure 3, obtained for cotton only samples A3, a decline in the degree of hydrophobicity is noted after pre-treatment due to the increase in the hydrophobic effect even if temporary. A3 is 6% more hydrophobic than A4, but the end result was nanoparticle deposition for both. In S2 we verify an increase 2,7% from B2 to B3, as expected due to the already higher degree of hydrophobicity of PES. Mixed CO/PES, B2 samples, contact angle increases after S1, but diminishes after S2. This demonstrates the ineffectiveness of S2 to nanoparticle deposition, because of the appearance of a lower contact angle relative to pre-treatment, showing that nanoparticles do not fixate in the substrate. In this case S1 is more effective for both samples. S1, allows a higher quantity of nanoparticles to be deposited in less functionalization time. Hydrophobicity tests do not reveal much differences between samples subjected to S1.

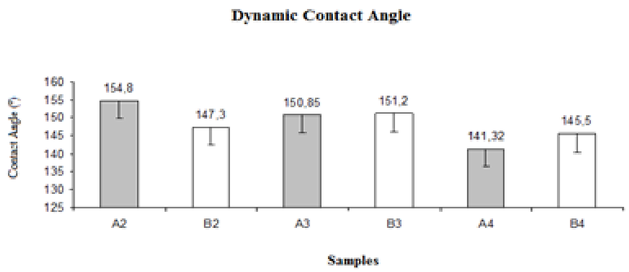

Figure 3 Dynamic Contact Angle (°).

A2 - (PT), A3 - (S01), A4 - (S02), B2 - (PT), B3 - (S01), B4 - (S02)

Figure Caption: Authors (2014)

Scanning electron microscopy

Observing Figure 4, we can see how the nanoparticles cover the surface of 100% CO fibers after S1. When compared to A2 (Figure 4a) we verified a uniform coverage of the substrate material as well as the interstitial spaces between them.

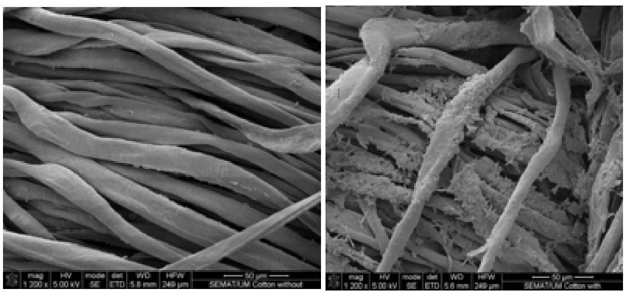

Figure 4 SEM samples – A – Control and B - A3 (S01).

Figure Caption: Authors (2014)

In Figure 5, SEM micrographs of mixed CO/PES fabrics show a less efficient deposition for S2 procedure, despite this being the procedure with lower air permeability. In fact, the CO material in the mixed fabric incorporated the majority of the nanoparticles while the PES polymer does not retain as much as its counterpart. Also, the spaces between the polyester fibers are smaller the ones in the cotton areas which explains the lower results of air permeability and the increase in hydrophobicity.

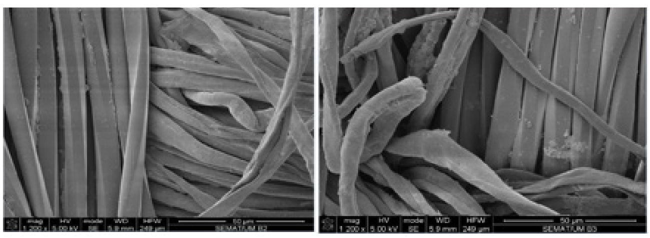

Figure 5 SEM samples – A- B2 (PT) and B - B3 (S02).

Figure Caption: Authors (2014)

Conclusion

This work has uncovered important data to understand the dynamics of cotton and mixed CO/PES functionalization with nanoparticles. It was possible to incorporate silver and titanium oxide nanoparticles via two different procedures. However, S1 was more effective for this particular textile substrates and final application in common clothing.

An important requirement in textiles is the durability of the embedded functional effects. Thus, for future studies we intended to give continuity to the work. To figure out the minimum period in which the effects remain active we will initially perform bending tests, draping, photobleaching, and resistance to washing in samples to be functionalized with the conditions of the S1 method as well as bioactivity testing to assess the antimicrobial behavior.

In conclusion, incorporating silver-doped TiO2 nanoparticles onto these fibers presents a versatile material with promising real-world applications. The combined antimicrobial prowess of silver and photocatalytic self-cleaning abilities of TiO2 is an interesting material to be used in hospital gowns or self-cleaning sportswear. The authors believe that functionalized fibers hold the potential to revolutionize various sectors and further research will unlock the full potential of this exciting material towards smarter and more sustainable textiles.

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Conflicts of interest

Authors declare that there is no conflict of interest.

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