Antimicrobial nanocomposites: Cellulose fibers loaded with silver nanoparticles in health products

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ABSTRACT

Polymeric nanocomposites based on cellulose fibers, mainly cotton, and loaded with silver nanoparticles have been used mainly in health products (such as endotracheal tubes, bandages, medical dressings, mesh, catheters and filters) thanks to the fact that these nanoparticles have broad-spectrum antiseptic properties. Based on the analysis of scientific publications, this work aims to investigate how these nanocomposites have been prepared and characterized in general, with regard to the characteristics and properties of silver nanoparticles and their synthesis by chemical, photochemical, physical and biological methods and how they are impregnated in cellulosic fibers. Specific considerations will also be made regarding the antimicrobial mechanism of nanostructured silver and its toxicity to humans.

Keywords: Cotton, Silver nanoparticles, Polymeric nanocomposites, Antimicrobial activity.

1 INTRODUCTION

According to Silva, Viana and Mohallem (2009), nanosciences study nanomaterials, which are materials in which at least one of their dimensions has a size between 1 and 100 nm. Nanotechnology, on the other hand, deals with the processes of preparation and characterization of nanomaterials.

It is worth noting that materials prepared by nanotechnological processes do not necessarily have nanometric size, but may have nanometric structures in their composition, resulting in nanostructured materials. In fact, some research advocates that sizes up to 300 nm be considered as belonging to the nanoscale (CADIOLI; SALLA, 2006; GUAZZELLI; PEREZ, 2009).

Esteves, Barros-Timmons and Trindade (2004) define nanocomposites as hybrid materials in which at least one of their constituents has nanometric dimensions.

Analogous to traditional composites, we have in nanocomposites the combination of at least two distinct phases, called reinforcement and matrix, the latter being the phase in which the first is dispersed. The matrices can be polymeric, metallic, ceramic or carbonaceous. The reinforcements can be fibrous or particulate (LEVY NETO; Pardini, 2006). In the case of this work, the nanocomposites are said to be polymeric depending on the class of materials to which their matrices belong.

This work is a bibliographic review and aims to analyze scientific publications on the preparation, characterization and antimicrobial properties of polymeric cellulose nanocomposites (as matrix) and silver nanoparticles (Ag NPs) (as filler).

These nanoparticles have broad-spectrum antiseptic properties and, therefore, are promising in the constitution of health products, such as: endotracheal tubes, bandages, medical dressings, mesh, catheters and filters.

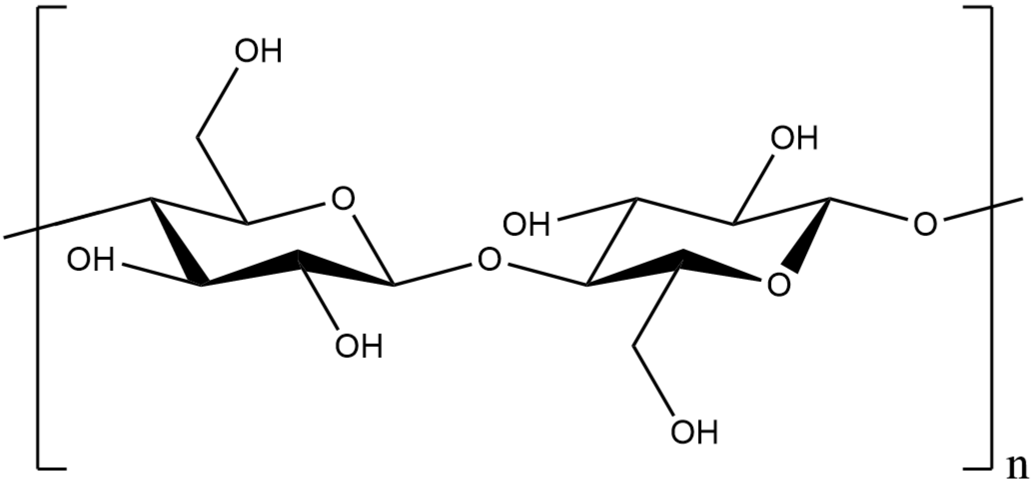
2 CELLULOSE

Cellulose, a constituent of the primary cell wall of green plants, many forms of algae and oomycetes, is the most abundant organic polymer in nature and also the most versatile organic raw material. It is a polysaccharide with molecular formula $(C6H10O5)_n$ that, when hydrolyzed in an acidic medium, produces a large number of monomeric units of β-D-glucose (also known as β-D-glucopyranose). Cellulose was isolated and characterized in 1838 by the French chemist Anselme Payen (SHREVE; BRINK JUNIOR, 1980).

The monomers are joined by glycosidic bonds β-(1,4) in an unbranched chain forming a linear structure (Figure 1). In addition, the hydrogen bonds between the hydroxyl groups of juxtaposed chains contribute to cellulose being fibrous, resistant and insoluble in water and in a large part of organic solvents. It is worth mentioning that the bonds between the monomers are resistant to the action of oxidizing agents, reducing agents and weak bases (FONSECA, 2003).

The pulp used in the production of paper is obtained from the pulp of the wood. The textile industry uses vegetable fibers, such as: hemp, jute, sisal, pita, rami, raffia, linen, paina and cotton (RIBEIRO, 1984).

Figure 1 – Cellulose repeating unit.



Source: Author (2023).

2.1 COTTON

Cotton, the most widely used natural fiber in the world, originates from India (where the oldest pieces were woven, dating back to 3000 BC) and Ethiopia. However, some historians say that cotton was already known and cultivated among the Aztecs (in Mexico) in 5000 BC (CHATAIGNIER, 2006).

Cotton is almost entirely composed of cellulose (with mass content ranging between 88 and 96 %) and contains small portions of protein, pectin, wax, ash, organic acids and pigments. Its fibers are able to absorb up to 32% of the moisture of completely saturated air. They can serve as food for insects and be completely decomposed in the environment, by certain fungi and bacteria, in about three months, and are therefore biodegradable. (RIBEIRO, 1984).

The cotton fiber grows in the seeds of several plants of the genus *Gossypium*, family *Malvaceae*. In each pit, 1200 to 1700 fibers are developed, which have the functions of protecting the grains of the seed and assisting their transport by the wind when ripened. The length of the fibers varies from 13 to 50 mm, while the thickness can measure from 20 to 40 µm from the point of adhesion, from where they thin to the tip (ERHARDT *et al*., 1976).

Cotton is used in the manufacture of a number of textile products and other products, such as: fishing nets, coffee filters, tents, explosives (nitrocellulose) and paper. It is also present in the daily life of hospital environments. The most common items where we find it are: the hydrophilic cotton itself, the gauze, the bedding that lines the beds and the clothes of the professionals who work there.

Since cotton is susceptible to microbial contamination under appropriate temperature conditions, the products based on it undergo physical and chemical treatments in order to remain aseptic (GAO; CRANSTON, 2008).

3 NANOPARTICLESS

The ISO/TS 27687:2008 standard defines a nano-object as a material in which one, two or three of its external dimensions belong to the nanoscale (CEN, 2008). Then this same standard defines nanoparticles as being nano-objects with all three external dimensions at the nanoscale. The other two types of nanoobjects are nanofibers (two nanoscale dimensions) and nanoplates (at least one nanoscale dimension).

According to Holister *et al.* (2003), the transition from microparticles to nanoparticles can lead to a series of changes in the physical properties of a material. One of the factors responsible for this is the increase in the ratio between surface area and volume. For example, gold is a material of yellow color, when it is in bulk (macroscopic) form, and red, when its nanoparticles are dispersed in aqueous medium.

Another issue is that within a matrix or in a liquid medium, nanoparticles aggregate easily. This can be explained by the fact that these nano-objects have a large surface energy. When they cluster, the system acquires lower energy values (LEVY NETO; Pardini, 2006).

Dispersing agents are also used or treatments of the surface of the material are made (polymer encapsulation or functionalization, for example) to ensure the dispersion of the nanoparticles, through mechanisms of electrostatic repulsion and / or steric impediment. Otherwise, agglomeration prevents the special properties of the nanomaterial, related to the nanometric dimension, from being evidenced (LEVY NETO; Pardini, 2006).

At the nanoscale, phenomena arise that do not appear at the macroscale. This is not only due to the reduction in size, but also to the changing importance of fundamental natural forces. Gravitational, frictional and combustion effects give rise to the effects of electrostatic forces and two aspects linked to them: van der Waals forces and Brownian motion (DURÁN; MATTOSO; MOORS, 2006).

4 SILVER NANOPARTICLES

In nature, silver is found in the native form or in the ores argentite (silver sulfide, $Ag2S$), chloroarginite (silver chloride, $AgCl$) and pyrargirite (silver sulfoantimony, $Ag_{3}SbS_{3}$). However, this metal is obtained mainly by application of the Parke process in lead ores containing small amounts of silver or as a by-product in the anodic mud of the electrolytic refining of nickel, copper, gold and zinc (LEE, 2009).

The earliest reference to metal appears in the book of Genesis when it is mentioned that Abraham was very rich in cattle, gold, and silver. Although man is said to have known silver since prehistoric times, portions of the metal's slag have been found in Asia Minor and the Aegean islands, indicating that it was being isolated from lead through surface mining around 4000 BC (WEEKS, 1968).

Pure silver is a white, shiny, glossy metal (depending on the degree of polish). Its ductility and malleability are inferior only to those of gold and palladium. It has the highest electrical and thermal conductivities of all metals and is stable to pure air and water. However, it is attacked by ozone, hydrogen sulfide, and sulfur-containing air. When newly deposited, it is the best reflector of visible light, but it quickly stains and loses its reflection (EMSLEY, 2011; GREENWOOD; Earnshaw, 1997; LEE, 2009).

The main applications of silver are in: photographic emulsions, dental amalgams, jewelry and ornaments, electrical contacts, printed circuits, batteries, mirror manufacturing, welding, brazing, catalysis and control of fission reactions in pressurized water nuclear reactors (KLOPROGGE; PONCE; LOOMIS, 2021).

4.1 PROPERTIES OF SILVER NANOPARTICLES

Some of the properties of macroscopic silver are also presented by Ag NPs, such as: good conductivity (electrical and thermal), chemical stability and catalytic activity. However, these properties can be controlled depending on the size of the nanoparticle, its shape and polydispersity (SIGNORI *et al*., 2010).

According to Kiss, Miotti and Ferraz (2011), the properties of nanoparticles vary with increasing size. When we have larger and larger nanoparticles, the number of atoms on the surface is proportional to the square of the diameter of the nanoparticle, while the number of atoms in the inner region is proportional to the cube of the diameter. For this reason, they present increasingly expressive crystalline characteristics, which did not exist before.

According to Melo Júnior *et al*. (2012), the color of colloidal silver is a result of the absorption of electromagnetic radiation in resonance with surface plasmons. With the increase in the size of the particles, the system starts from yellow and passes through orange and violet until it acquires the characteristic coloration of macroscopic silver.

4.2 ANTIMICROBIAL ACTIVITY OF SILVER NANOPARTICLES

Various heavy metals in very small amounts (which can be expressed in parts per million) can be biocides or antiseptics, such as silver, copper, and zinc. This antimicrobial capacity is called oligodynamic action, resulting from the action of metal ions (PELCZAR JR.; CHAN; KRIEG, 1996).

Still according to the same authors, in antiquity, water was stored in silver or copper containers because people realized over time that the containers of these metals kept the water suitable for human consumption. During the Middle Ages, solutions of silver nitrate (AgNO3), known as "Lunar Caustic", were given to patients with nervous disorders, epilepsy and syphilis.

Moving forward in chronology, we have the use of 3 to 1 % AgNO solutions as antiseptic eye drops in the prevention of neonatal gonorrheal ophthalmia. This prophylaxis was established by Karl Credé in 1884 in the United States. However, antibiotics eventually replaced $AgNO_3$ for this purpose (TORTORA; FUNKE; CASE, 2005).

The authors point out that, recently, there has been renewed interest in the use of silver as an antimicrobial agent, especially in cases where there are antibiotic-resistant bacteria. The most common formula is a topical cream based on silver sulfadiazine for use in burns.

Currently, dressings to which Ag NPs are incorporated are employed in the treatment of open wounds, burn infections and chronic ulcers. Ag NPs are broad-spectrum antimicrobial agents that have action against bacteria (Gram-positive or Gram-negative), filamentous fungi, yeasts and viruses (EL-RAFIE *et al*., 2012).

The first scientific documents reporting the use of colloidal silver in the medical field date from the late eighteenth century. other related research was developed in the period between 1910 and 1920 (GIBBS, 1999).

Sadeghi *et al*. (2012) compared the bactericidal activity against *Escherichia coli* and *Staphylococcus aureus* of different silver nano-objects (nanoparticles, nanorods and nanoplates) stabilized by the polymer poly (N-vinyl-2-pyrrolidone) (PVP), by the methods of disc diffusion and Minimum Inhibitory Concentration (MIC).

Staphylococcus aureus is one of the main Gram-positive bacteria that cause disease and can be found on the skin in regions such as: armpits, inner part of the elbows, between the buttocks, in the groin and in the lower part of the heel. Some of its strains are resistant to methicillin. *Escherichia coli,* a Gram-negative bacterium and also pathogenic, resides in the thin portion of the intestine, and can be found in areas of the body contaminated by feces and urine (GRICE *et al*., 2009).

They found that the antimicrobial effect of the nano-objects is dependent on their shapes and sizes, due to their different surface areas, and that the nanoplates were the ones that showed the best bactericidal activity for the microorganisms tested.

Klemenčič *et al*. (2010) studied the efficiency of different forms of silver in the protection of cellulose fibers against biodegradation in test soil (simulating the biodegradation of the material in the environment), according to ISO 11721: 2003. The samples were buried in the soil for periods of 3 and 12 days.

The cotton fabrics were functionalized by the exhaustion method with: (a) Ag NPs with an average diameter of 30 nm, dispersed with the aid of a dispersing agent and ultrasound, generating the material M1; (b) commercial dispersion of AgCl in water, originating the material M2 and (c) colloidal silver, consisting of nanoparticles with an average diameter of less than 10 nm, resulting in the material M3. The tissue samples were immersed in the respective dispersions and left for 30 minutes and at room temperature under agitation and rotation. Finally, they were squeezed and dried at room temperature as well.

The degree of biodegradation was monitored by: SEM, color measurement (parameter of the uniform color scale CIE L*a*b*, of the International Commission on Illumination), tensile strength, degree of polymerization and Fourier Transform Infrared Spectroscopy (FTIR). The authors concluded that large concentrations of Ag NPs in the M1 material led to insufficient antimicrobial activity, due to the mechanism of release of silver cations (Ag1+) from the agglomerates for the medium to be deficient. In the case of M2 material, the dissociation of AgCl in the presence of moisture was high, providing better protection against degradation than that observed for M1 material. Finally, the M3 material exhibited the best protection, probably due to the synergistic action of the nanoparticles and Ag^{1+} cations released.

The antimicrobial action of Ag1+ cations on microorganisms is already known. However, the action of Ag NPs is not yet well resolved (HEBEISH *et al*., 2011).

Morones *et al*. (2005) reported the presence of small Ag NPs adhered to the bacterial membrane and also inside the cell, which may contribute to a better understanding of the bactericidal action of this nanomaterial. There are two main mechanisms proposed.

4.2.1 First mechanism

The metallic silver (Ag0) in the nanoparticles can be oxidized to the Ag1+ cation, in aqueous medium, by the action of the dissolved oxygen present in it, according to Equation 1:

$$\text{O2 (aq)} + 4\ \text{H3O1+ (aq)} + 4\ \text{Ag (s)} \rightarrow 4\ \text{Ag}^{1+}\ \text{(aq)} + 6\ \text{H2O (l)} \qquad (1)$$

According to Mijakovic *et al*. (2006), the Ag1+ cation has a great affinity for sulfur and phosphorus atoms, which causes two situations to happen. In the first, the Ag1+ cations react extra or intracellularly with the thiol (–SH) groups present in the proteins that make up the bacterial cell membrane, denatured them.

In the second, Ag1+ cations can interact with portions of phosphorus in the DNA, causing damage that prevents the performance of normal metabolic functions, and the cell can no longer divide (GUPTA; BAJPAI; BAJPAI, 2008; MORONES *et al*., 2005).

4.2.2 Second mechanism

Starting from water and dissolved oxygen in the medium, the Ag1+ cation can catalyze the formation of nascent oxygen, which oxidizes the molecules that make up the microorganism. The interesting thing is that, according to this mechanism, it is not necessary that there is a direct contact between the Ag1+ cation and the microorganism, since the radical formed can diffuse through the medium (KIM *et al*., 2007).

4.3 TOXICITY OF SILVER NANOPARTICLES

Although silver is not considered toxic, most of its compounds are. This is because they can be absorbed by the circulatory system, leading to the deposition of Ag0 in various tissues of the body, which can cause argyria, a condition characterized by bluish or blue-gray pigmentation of the skin and mucous membranes. In addition, the ingestion of silver in high concentrations can cause side effects such as: neurological and kidney problems, indigestion and headache (KLOPROGGE; PONCE; LOOMIS, 2021).

Guazzelli and Perez (2009) warn that nanomaterials may be more toxic than the corresponding compounds at the macrometric scale. They have greater chemical reactivity, biological activity and catalytic action, due to the huge surface area they have. For the same reason, nanomaterials with different shapes and sizes can exhibit completely different toxicities.

Also according to the authors, due to their small size, nanomaterials are more easily absorbed by cells and tissues. They are so small that the immune system itself cannot detect them. Some studies have shown that in *vivo* tests with synthetic nanoparticles, there was the formation of free radicals, which caused: inflammation, disturbances in cellular functions and the subsequent development of tumors due to DNA damage.

Ahamed, Al-Salhi and Siddiqui (2010) critically discussed the recent findings related to the risk of human exposure to NP Ag, mainly in the areas of: cosmetics, sunscreens, textiles, food and medical products.

Based on the results of a large number of *in vitro* studies with mammalian cells, one of the authors' findings was that Ag NPs are toxic to cells derived from the skin, brain, vascular system, reproductive organs, and especially the liver and lung.

In addition, in vivo *toxicity studies* in rats and mice demonstrated that after being administered by inhalation, ingestion, or intraperitoneal injection, NP Ags were found in the bloodstream and caused toxic effects in several organs, including the brain.

Beer *et al*. (2012), using the MTT colorimetric assay, investigated the relationship between the viability of A549 cells of the lung and the fraction of Ag1+ cations present: (a) in suspensions of Ag NPs and (b) in their supernatant after ultracentrifugation. The concentration of Ag1+ cations was determined by Atomic Absorption Spectrometry (AAS) after acid digestion.

The results showed that cell viability decreases with the increase of the fraction of Ag1+ cations. Cells exposed to a suspension of Ag NPs containing a fraction of Ag1+ cations of 69 % had a viability of 54 %, while for the case of the fraction to be 39 %, the viability was 92 %.

The study also concluded that there are no statistically significant differences between the toxicities of the suspension of Ag NPs and their supernatant. Only suspensions in which the fraction of Ag1+ cations was less than or equal to 2.6 % were shown to be slightly more toxic than their respective supernatants.

5 CELLULOSIC NANOCOMPOSITES WITH SILVER NANOPARTICLES

The processing of nanocomposites is determined based on the nature and characteristics of the matrix and reinforcement. Therefore, in this work, we will first pay attention to the fact that the matrix (cellulose fibers) is polymeric and the charge (Ag NPs), metallic.

Cellulose fibers are made by living organisms and then extracted and processed. That is, there are no synthetic syntheses involved in obtaining them. They have low specific mass and are biodegradable, non-toxic and recyclable. Additionally, extraction and processing costs are low. These are some of the advantages of using these fibers (LEVY NETO; Pardini, 2006).

Since, in general, cellulose fibers do not tolerate temperatures higher than 200 ° C, care must be taken in choosing the technique of preparation of nanocomposites in which they will be one of the constituent elements.

Ag NPs are often immobilized on the fibers of a range of textile products, giving the fabrics an antimicrobial effect that allows them to be used in the making of bandages, gauze, bed sheets and surgical clothing. The most frequently used fabric is cotton (HANH *et al*., 2014; Kozicki *et al*., 2013).

5.1 PREPARATION OF NANOCOMPOSITES

According to Esteves, Barros-Timmons and Trindade (2004) and Maneerung, Tokura and Rujiavanit (2008), polymeric nanocomposites loaded with metallic nanoparticles can be prepared by three main strategies. They are:

a) The mixture of the components.

b) The polymerization of the monomer *in situ* to generate the matrix, in the presence of metallic nanoparticles.

c) The reduction of cations or metal complexes in a containing medium or in the polymer matrix itself, that is, the *in situ synthesis* of the nanoparticles.

In the case of nanocomposites consisting of cellulose fibers, among the three strategies listed, the second is not feasible. That's because the fibers are extracted and not synthesized.

In most cases, the manufacturing process involves the separate synthesis of the Ag NPs, which are then impregnated in the matrix (first strategy). Cotton fibers have large surface areas containing hydroxyl groups, which assist in the fixation of NPs Ag. However, crosslinking or adsorption agents may be used (BREITWIESER *et al*., 2013; RAVINDRA *et al*., 2010).

However, Dallas, Sharma and Zboril (2011) comment that *in situ* reduction prevents the formation of nanoparticle aggregates (which would drastically reduce the extent of the antimicrobial property) and leads to the obtaining of nanoparticles with a narrow size distribution. That would be the advantage of the third strategy over the first.

Another option is to perform some type of surface treatment to functionalize the surface of the fibers, nanoparticles or both, in order to improve the adhesion between the reinforcement and the matrix at the interface (LEVY NETO; Pardini, 2006).

The process of treating cellulose fibers tensioned with alkaline solution is called mercerization, after John Mercer, who, in 1844, performed the first experiments on the behavior of cellulose in soda bleach or caustic potash. He observed that the cotton fabric samples showed: shrinkage (from 20 to 30 %), increase in tensile strength (in the order of 25 %) and increase in affinity for dyes (from 30 to 40 %). When the operation is performed in the absence of tension, it receives is called leaching or caustification.

This treatment with alkaline solution is widely used to functionalize cotton fibers. In the same way that there is an increase in affinity for dyes, the modified surface can improve the adhesion of other types of compounds.

5.2 FOULARDAGEM

The foulard is a machine used in the process called foulardagem (or padding, in English), popularly known as impregnation, which has the function of impregnating a wide variety of compounds (dyes, pigments, microcapsules, protectors against ultraviolet rays, bactericides and bacteriostatics) in textile products. Prior to the foulardagem process, a depletion step is carried out, in which, inside a vat, the fabric is immersed in a bath that contains the compound to be impregnated.

In the foulardagem itself, the compound present in the bath absorbed by the fabric is forced to penetrate the interior of the material by the action of two rubber cylinders that keep the system pressed. There are configurations in which several pairs of cylinders can be placed in sequence.

After foulardagem, the tissue is processed, usually in a dryer, to consolidate the fixation of the compound in the tissue. There are some derivations of the foulardagem process. They are:

a) Pad-stream: sequence of: impregnation, vaporization, washing, drying and winding.
b) Pad-batch: sequence of: impregnation, cold rest, washing, drying and winding.
c) Pad-dry: sequence of: impregnation, drying, washing, drying and winding.
d) Pad-dry-stream: sequence of: impregnation, drying, vaporization, washing, drying and winding.

6 SYNTHESIS OF SILVER NANOPARTICLES

The development of new ways of preparing metallic nanoparticles has been extensively investigated. There are two general types of production of these nanoparticles. The first type is characterized by aggregation techniques, in which atoms are aggregated to form the nanostructure (bottom up) and the second is composed by removal techniques, in which there is removal of material to form the nanostructure (top down) (EUSTIS; EL-SAYED, 2006).

Ag NPs can be prepared by a variety of synthetic routes involving chemical, photochemical, physical and biological methods.

6.1 CHEMICAL METHODS

According to Durán, Mattoso and Morais (2006), chemical approaches are the most commonly employed because they provide the synthesis of nanoparticles in a liquid medium. Generally, in addition to the medium, three types of compounds are used: a precursor of the metal (the simplest are the salts that contain the cation of the metal, such as $AgNO_3$), a reducing agent (which will promote the reduction of the cation of the metal) and a stabilizing agent (which will prevent the aggregation of the nanoparticles formed, keeping the colloid stable).

Tran *et al*. (2013) explain that the formation of silver colloids involves two phases, one of nucleation and the other of growth. These phases can be controlled in order to obtain nanoparticles with the desired size and shape, by adjusting some parameters of the synthesis, such as, for example, the temperature (usually high temperatures), the pH and the speed of agitation of the system.

The most commonly used reducing agents are: sodium borohydride ($NaBH_4$), formaldehyde, sodium citrate, hydrazine (N2H4), ascorbic acid, glucose and also the liquid medium itself such as dimethylformamide (DMF) and ethylene glycol (DALLAS; SHARMA; ZBORIL, 2011).

According to Budama *et al*. (2013), the reduction with N2H4, provided that it is stoichiometrically controlled, does not form dangerous by-products, according to Equation 2:

$$4\,\text{Ag}^{1}\text{+ (aq)} + \text{N2H4 (aq)} \rightarrow 4\,\text{Ag (s)} + 4\,\text{H1+ (aq)} + \text{N2 (g)} \qquad (2)$$

As examples of stabilizing agents, Tran *et al*. (2013) cite poly (vinyl acetate) (PVA), PVP and sodium oleates. Some synthetic polymers can act simultaneously as reducers and stabilizers, such as polyacrylamide.

El-Nour *et al*. (2010) say that Ag NPs can also be reduced in microemulsion, which is a biphasic system consisting of an aqueous phase (where the precursor of the metal is usually located) and another organic phase (where the reducing agent is found), under vigorous agitation. The interaction between the reactant species depends on mass transport across the interfaces, mediated by a quaternary alkylammonium salt. Although nanoparticles with uniform size are obtained by this method of preparation, it is a generally costly approach.

6.1.1 Preparations involving chemical methods

Ilić *et al.* (2009) evaluated the antimicrobial efficiency, before and after repeated washing, of cotton fabrics loaded with Ag NPs, with an average diameter of approximately 10 nm, against the bacteria *Escherichia coli* and *Staphylococcus aureus* and the fungus *Candida albicans*.

The nanoparticles were synthesized by reducing Ag1+ cations in an argon atmosphere with $NaBH_4$ and without the use of any stabilizer. For the impregnation stage, the tissue was immersed in the colloids, cured at 100° C for 5 minutes, washed and dried at room temperature. Some samples of the nanocomposite obtained were dyed with the dye C. I. Direct Red 81.

Although the tissues loaded with colloid at a concentration of 10 ppm exhibited good antimicrobial efficiency before washing, the study indicated that they did not have a good durability against them due to the detachment of the nanoparticles from the fibers. The content of silver detached from the nanocomposite was determined by AAS.

This detachment was slower for tissues loaded with colloid at a concentration of 50 ppm, which showed microbial reduction above 99%, even after washing. It is worth mentioning that, in the results of many publications, the presentation of percentage values of reduction of microorganisms is recurrent, which will be referred to in this text only as microbial reduction, calculated by Equation 3:

$$R\,(\%) = \frac{A_0 - A}{A_0} \cdot 100\,\% \qquad (3)$$

In Equation 3, we have that: **"R (%)"** is the microbial reduction; "**A0**" is the number of colony-forming units (CFU) in the control sample (in the absence of a silver compound) and "**A**" is the number of CFU in the sample loaded with a silver compound.

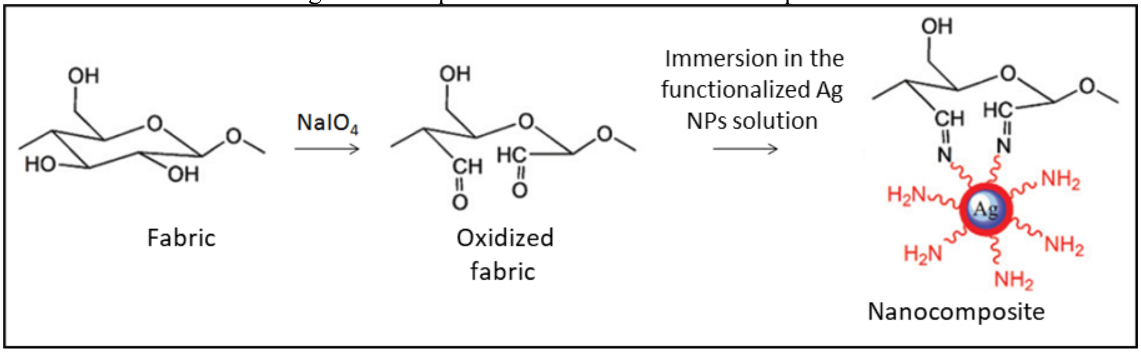
The antimicrobial activity was not affected by the dyeing of the tissue with the dye. Other results showed that, starting from the colloid with a concentration of 50 ppm, there are changes in the color of the dyed tissues that are noticeable to the naked eye, since $\Delta E^* > 1$, according to the CIE color scale L*a*b*.

Zhang *et al.* (2013) studied the durability, against washing, of an antibacterial cotton fabric loaded with Ag NPs. In the first step of the preparation of the material, they used a hyper-branched amino finished polymer (HBP-NH2) to obtain the nanoparticles, through the reduction of Ag1+ cations from an aqueous solution of $AgNO_3$, and also functionalize them, without any other reducing or stabilizing agent. Initially the system was stirred at room temperature and then heated.

Subsequently, the cotton fabric previously oxidized by an aqueous solution of sodium periodate ($NaIO_4$) was dipped in the solution containing the Ag NPs functionalized by the polymer, for a period of 30 minutes and at room temperature. The overall process of nanocomposite preparation can be seen in Figure 2.

Due to the presence of aldehyde groups, it was possible to adhere the functionalized nanoparticles to the oxidized tissue by means of chemical bonds. Thus, after exposure of the nanocomposite to 50 consecutive washes, the silver content (measured by Inductively Coupled Plasma Atomic Emission Spectrometry, ICP-AES) decreased from 149.88 to 116.08 mg/kg, demonstrating good durability against washing. The microbial reduction of *Escherichia coli* and *Staphylococcus aureus*, also after this number of washes, was 96% on average.

Figure 2 – Preparation scheme of the nanocomposite.



Source: Adapted from ZHANG *et al*. (2013, p. 2093).

In an initial step, Shateri-Khalilabad, Yazdanshenas and Etemadifar (2017) prepared Ag NPs by in *situ* reduction of Ag1+ cations (from $AgNO_3$), with the use of ascorbic acid, on the surface of a cotton fabric previously treated with potassium hydroxide (KOH).

Subsequently, the tissue impregnated with the nanoparticles was immersed for 60 minutes in an alcoholic solution of prehydrolyzed n-hexadecyltrimethoxysilane. Finally, it was cured at 110°C also for 60 minutes, forming a high-density, low-energy layer around the tissue. One of the purposes was to produce a super-hydrophobic fabric.

The resulting nanocomposite showed excellent antibacterial activity (against *Escherichia coli and* Staphylococcus aureus*) and protective action against UV-A and UV-B radiation (in the range of 280 to 400 nm, with a protection factor of 296).*

The material also demonstrated the maintenance of its properties after 5 washes, lasting 45 minutes each, with a solution of concentration equal to 5 g/L of a non-ionic detergent (Triton X-100) at 50° C, according to ISO 105-C10:2006. This is because the hydrophobic coating prevented the particles from being removed from the surface of the nanocomposite.

From the bacterial cellulose produced by *Acetobacter xylinum*, Maneerung, Tokura and Rujiravanit (2008) reduced *in situ*, Ag1+ cations (from an aqueous solution of $AgNO_3$ with a concentration of 0.001 mol/L), using as a reducer NaBH4 (at concentrations of 0.001, 0.01 and 0.1 mol/L). First, the cellulosic film was immersed in the nitrate solution for 60 minutes. Then, the reduction began, lasting 10 minutes. Finally, the nanocomposite was washed with ultrapure water to remove excess reducer and by-products, and freeze-dried (frozen at a temperature of – 40°C and vacuum-dried at – 52°C).

The size of the nanoparticles and their distribution were evaluated by Transmission Electron Microscopy (TEM). A higher concentration of the reducing agent provided the formation of nanoparticles with a smaller average diameter (5.47 nm) and a narrow size distribution ($\sigma = 2.20$ nm). The X-ray Diffraction (XRD) technique complemented the observation of the formation of the nanoparticles, through which the planes (111), (200) and (311) characteristic of the cubic structure of the centered face of Ag0 were observed in the diffractograms. It is worth mentioning that the Energy Dispersive X-ray Spectroscopy (EDS) did not identify boron in the elemental composition of the nanocomposite, which is something positive, considering the toxicity of this element.

The resulting nanocomposite exhibited strong antimicrobial activity against *Escherichia coli* and *Staphylococcus aureus*, evaluated by the methods of counting colonies formed and disc diffusion.

The authors emphasize that the prepared nanocomposite can be used as a skin substitute, in the treatment of extensive burns, and in the making of active dressings. It keeps local temperature and humidity at adequate levels, accelerating the processes of epithelialization and angiogenesis, as well as contributing to the relief of local pain and inhibiting microbial contamination.

Initially, Budama *et al.* (2013) synthesized the amphiphilic and block copolymer polystyrene-co-poly(acrylic acid) (PS-b-PAA) by radical polymerization by atomic transfer. Then, the copolymer was dissolved in toluene and heated to 135° C for 20 minutes to form reverse micelles. Subsequently, a solution of AgNO 3 was added to the system containing the micelles, under vigorous agitation, so that the Ag1+ cations would bind to the carboxyl groups in the micellar nuclei. This stage was carried out under the shelter of light for 2 days. Finally, the Ag 1+ cations in the nuclei were reduced to Ag NPs by the action of N2H4. The nanoparticles obtained were characterized by TEM and XRD, revealing that the average diameter of these chemical entities was 20 nm.

Clean and dry samples of cotton fabric were introduced into the system where the micelles containing the nanoparticles were found and left under agitation for 30 minutes. Then, the tissues were dried at room temperature and placed in an oven for 5 minutes and at 135° C, to bind the micelles to the tissue surface, covalently, through the carboxyl groups in the PAA chains and the hydroxyl groups of the cellulose.

At the end, the antimicrobial activity against the bacteria *Escherichia coli* and *Staphylococcus aureus was evaluated* by the disc diffusion method. It was observed that the bactericidal effect remained up to 5 washes for the first microorganism and up to 20 washes for the second.

Osorio *et al*. (2012) synthesized, in *situ*, Ag NPs using as reagents the precursor AgNO3 (in aqueous solution) and the reducing agent butylamine (in alcoholic solution). The concentration of $AgNO_3$ was fixed at 1 nM, while the concentrations of the agent were 1, 2, 4 and 5 nM. The substrates were woven of wool and cotton. The systems were shaken throughout the synthesis, which lasted 120 minutes. The temperature of 45° C and pH 7 were kept constant. A comparison was made using an aqueous and an ethanolic system. At the end, the nanocomposites were: washed twice with diluted detergent, rinsed three times with cold water and dried.

MET and EDS analyses confirmed the formation of Ag NPs on the surface of both textile products. The ICP-AES analyses (with the samples digested in 9 % nitric acid solution and for 60 minutes) indicated that the amount of silver deposited per gram of cotton for the aqueous system was up to sixteen times higher than that found for the ethanolic system. The amount deposited per gram of wool was up to three times higher for the aqueous system. These differences may be due to better solubilization of $Ag^{1+\text{ cations in}}$ aqueous medium, which leads to an increase in the deposition of Ag NPs.

The formation of Ag NPs on the fibers was also shown to be dependent on the sequence of addition of the reactants. The simultaneous addition of salt and reducing agent resulted in a mass (of Ag NPs) three times smaller than that formed when butylamine was added before $AgNO_3$.

Hebeish *et al*. (2011) synthesized Ag NPs with an average diameter between 6 and 8 nm using hydroxypropyl starch (a type of chemically modified starch that therefore will not be considered as a natural extract) as a reducing and stabilizing agent. They were then applied to cotton fabrics in the presence and absence of the liquid Printofix Binder MTB EG binding agent. The samples were dried at 70° C for 3 minutes and cured at 150° C for 2 minutes.

The nanocomposites obtained (with and without the binder) were evaluated for their antimicrobial activities against *Escherichia coli* and *Staphylococcus aureus*, before and after 5, 10 and 20 washing cycles with hot water, sodium carbonate at a concentration of 2 g/L and a non-wetting agent at a concentration of 5 g/L. Emphasizing that each wash lasted 45 minutes.

Prior to washing, the microbial reduction was greater than 95 %. After 5 washing cycles and in the absence of the binding agent, there was detachment of the nanoparticles, which caused the microbial reduction to be below 74%, an unsatisfactory result. However, the presence of the binding agent ensured microbial reductions above 92% even after 20 washing cycles, demonstrating the importance of this agent in the fixation of the nanoparticles to the tissue surface.

Breitwieser *et al*. (2013) prepared nanocomposites of viscose fibers (artificial cellulose fiber, manufactured from wood chips of low-resinous trees or the linter of the cotton seed) and Ag NPs, using microwave and conventional heating (in reflux and sealed pot). Both forms of heating kept the system at a temperature of 80° C for comparison purposes (but other temperatures were also employed), and in the case of microwaves, the synthesis time was 4 minutes, while for conventional heating, this time was 120 minutes. This is one of the advantages of using microwaves to promote the heating of systems.

As a reducing and stabilizing agent, sulphated chitosan was used, a green, biodegradable and biocompatible polymer. According to Laranjeira and Fávere (2009), chitin is a natural polymer extracted from the exoskeleton of crustaceans and insects, while chitosan is obtained by deacetylation of chitin in alkaline medium. In turn, chitosan can be chemically modified through a sulfation reaction with chlorosulfonic acid to obtain sulfated chitosan

The preparation of the nanoparticles took place: *in situ, in* aqueous medium, with agitation and in the absence of light and air. The resulting materials were submitted to antimicrobial assays against *Escherichia coli* and showed high bactericidal action.

Characterizations by Dynamic Light Scattering (DLS), Ultraviolet and Visible Spectroscopy (UV-Vis) and TEM found that both heating techniques led to the preparation of very similar materials, which differed only in the shape of the nanoparticles on the fibers: elongated when using microwaves and spherical for conventional heating. No evidence of specific effects from the use of microwaves was obtained.

6.2 PHOTOCHEMICAL METHODS

According to Tran *et al*. (2013), photochemical methods are those in which nanoparticles are obtained through the reduction of metal ions, by the action of excited chemical entities formed in the irradiation process, such as radicals.

The main advantages of this type of synthesis are:

a) It is a clean process (as it does not use chemical reducing agents) and easy to perform.
b) It allows the reduction of metal ions *to occur in situ*, as it is a process of high spatial resolution.
c) It is quite versatile, since the synthesis can be done in various media, such as: polymeric films, emulsions, micelles, glasses.

6.2.1 Preparations involving photochemical methods

Hanh *et al*. (2014) were able to immobilize Ag NPs, with an average diameter of 12 nm, on the surface of cotton tissues by *in situ* synthesis using gamma irradiation. At 30° C, the Ag1+ cations were reduced using a dose of 13.8 kGy. An aqueous solution containing $AgNO_3$ at a concentration of 1,5 mmol/L and chitosan (stabilizer) at 1 % was started. The presence of nanoparticles formed in the tissue, at a concentration of 1696 ± 80 mg/kg, was confirmed by SEM and XRD.

The microbial reduction of the nanocomposites prepared against *Escherichia coli* and *Staphylococcus aureus* remained above 99.00 % even after 40 washing cycles. In addition, through tests on rabbits, it has been found that impregnated tissues are innocuous to the skin, proving that this approach holds promise for preparing stable and safe antibacterial tissues.

Kozicki *et al*. (2013) used the screen printing method and type C ultraviolet irradiation (UVC), with energy of up to 10000 mJ/cm2, to obtain silver-based antimicrobial finishes on cotton fabrics. The printing folder was composed of: water; AgNO3 at concentrations of 0,01 to 10 % (w/w); a thermally crosslinkable acrylate copolymer (binding agent) and a thickener. UVC irradiation was responsible for the reduction of part of the Ag1+ cations of the system.

Although there was no formation of Ag NPs, this study presented an easy and quite innovative preparation method. In fact, the material obtained was a composite, consisting of cotton fibers coated by a film containing silver products (Ag0, AgCl and silver oxide) with a size of up to 1000 nm, among which, aggregates and cubic particles.

Initially, it was observed that there was a change in the color of the fabric (Figure 3) and that it depended on the energy of UVC radiation and the content of AgNO 3 in the formulation of the printing paste. The techniques of SEM and EDS proved the formation of the films on the wires.

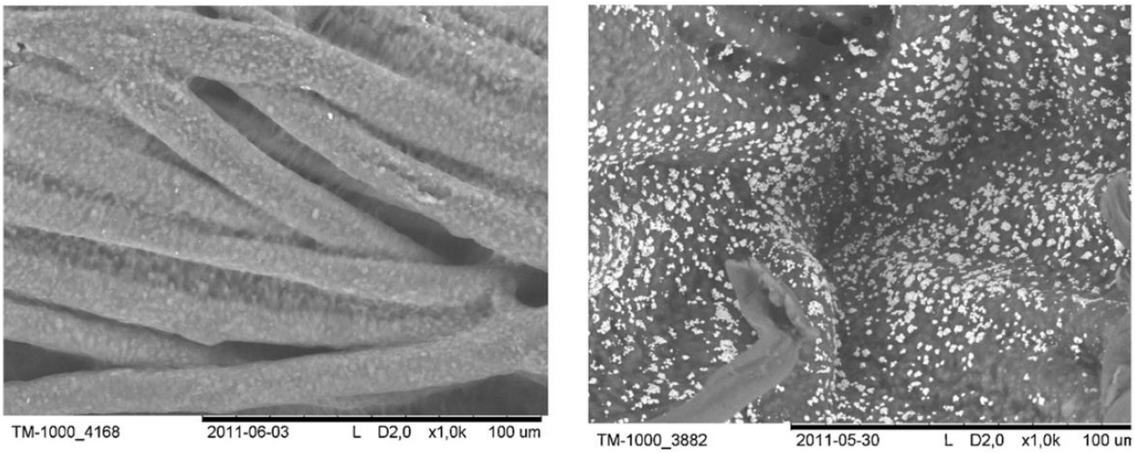
Figure 3 – Uniform before printing, after UVC irradiation and after a wash.

Source: Kozicki *et al*. (2013).

Microbiological assays revealed that the prepared nanocomposites inhibited the growth of the following microorganisms: *Escherichia coli*, *Bacillus subtilis* and *Staphylococcus aureus*. The curious thing is that the antimicrobial activity increased after the washing cycles (50 in total), responsible for exposing the silver products, previously covered by the polymeric film (Figure 4).

Perera *et al*. (2013) investigated two approaches to impregnate cotton fabrics with Ag NPs. In both cases, the precursor was $AgNO_3$. The first of these consisted of the previous synthesis of the nanoparticles using sodium citrate as a reducing agent (under agitation and at 70° C). Then, the tissue samples were immersed in the colloid obtained (for 30 minutes, under agitation and at 50° C), pressed, air-dried and cured in an oven (for 3 minutes and at 120° C).

Figure 4 – Photomicrograph obtained by SEM (magnification of 1000 x).



Source: Kozicki *et al*. (2013).

Note: On the right we have the composite before washing and, on the left, the same after 50 washing cycles. For this sample, the irradiation energy was 10 J/cm2 and the AgNO3 content in the printing paste was 1 % (w/w).

The second was an *in situ* photoreduction, employing ultraviolet radiation to reduce Ag1+ cations. After the tissue samples had been soaked by the nitrate solution, pressed and dried at 50°C for 10 minutes, they were irradiated for a period of 60 minutes, at a distance of 100 mm from the lamp.

The results of UV-Vis and SEM showed that there was agglomeration of the nanoparticles obtained by chemical reduction. On the other hand, the photoreduction provided a uniform distribution of the same on the surface of the tissues. The antimicrobial activity was qualitatively evaluated by the disc diffusion method against *Staphylococcus aureus* and *Escherichia coli strains*. The nanocomposites prepared by photoreduction showed better bactericidal effect, even after repeated washing cycles.

A differential of this study was the comparison between some physical properties of the tissue and the nanocomposites. Tensile strength and elongation at rupture were not affected by the impregnation of the nanoparticles. On the other hand, properties such as the crease recovery angle, the flexion length and the flexion stiffness were not significantly altered only in the nanocomposites prepared by photoreduction.

6.3 PHYSICAL METHODS

Evaporation-condensation and laser ablation techniques are the predominant physical approaches to synthesizing Ag NPs (PERERA *et al*., 2013).

There is also the technique of size reduction by grinding, in liquid medium and in the presence of a surfactant, which can be prolonged for periods of time longer than one month (DURÁN; MATTOSO; MOORS, 2006).

In evaporation-condensation preparations, nanoparticles are synthesized after the starting material is placed in the center of a tube furnace at atmospheric pressure and vaporized by a carrier gas. The drawback is the large amount of energy required for preheating and stabilization of the furnace operating temperature, which, in general, are very time-consuming steps (TRAN *et al*., 2013).

According to El-Nour *et al*. (2010), laser ablation is a process of removing material by irradiation with a laser beam, usually from a solid surface immersed in a liquid medium. It is a way to prepare metallic colloids directly almost always without the addition of chemicals, which implies a purer final material. The characteristics of the nanoparticles formed depend on some parameters, such as: laser wavelength, duration of laser pulses, laser creep, ablation time, liquid medium used and presence or absence of surfactants.

6.4 BIOLOGICAL METHODS

Biological synthesis is nothing more than the reduction of Ag1+ cations by the action of reducing agents, which can also function as stabilizing agents, present in fungi, yeasts, bacteria and plant extracts. These compounds contain functional groups, such as hydroxyl (–OH), carboxyl (–COOH), amine (–NH2), and thiol (–SH). The rate of reduction of metal cations, in general, is high and the process gives in ambient conditions of temperature and pressure (BAJPAI *et al*., 2007; TRAN *et al*., 2013).

The biosynthesis of metallic nanoparticles has been pointed out as a promising green preparation technique, because it employs renewable reagents (instead of chemicals) and non-toxic or very low toxicity solvents and is low polluting (BHATTACHARYA; GUPTA, 2005).

6.4.1 Preparations involving biological methods

Ravindra *et al*. (2010) prepared Ag NPs with an average diameter of approximately 20 nm, using as reducing agents the extracts of the leaves of *Eucalyptus citriodora* and *Ficus bengalensis*, separately.

These extracts, which also aid in the adhesion of nanoparticles to cotton fibers, contain polysaccharides composed of: p-mentane-3,8-diol; β-sitosterol; α-D-glucose and mesoinositol. The functional groups present in the molecules of the enumerated compounds are able to reduce the Ag1+ cations to nanoparticles, which are additionally stabilized by the chains of these molecules.

In aqueous medium (and under normal conditions of temperature and pressure) were gathered: the extract of the leaves of one of the trees, the cotton fibers and $AgNO_3$. The reduction occurred for a maximum time of 5 minutes. For the complete impregnation of the fibers with the nanoparticles formed, the system was stirred for 24 hours. At the end, the resulting material was washed and dried.

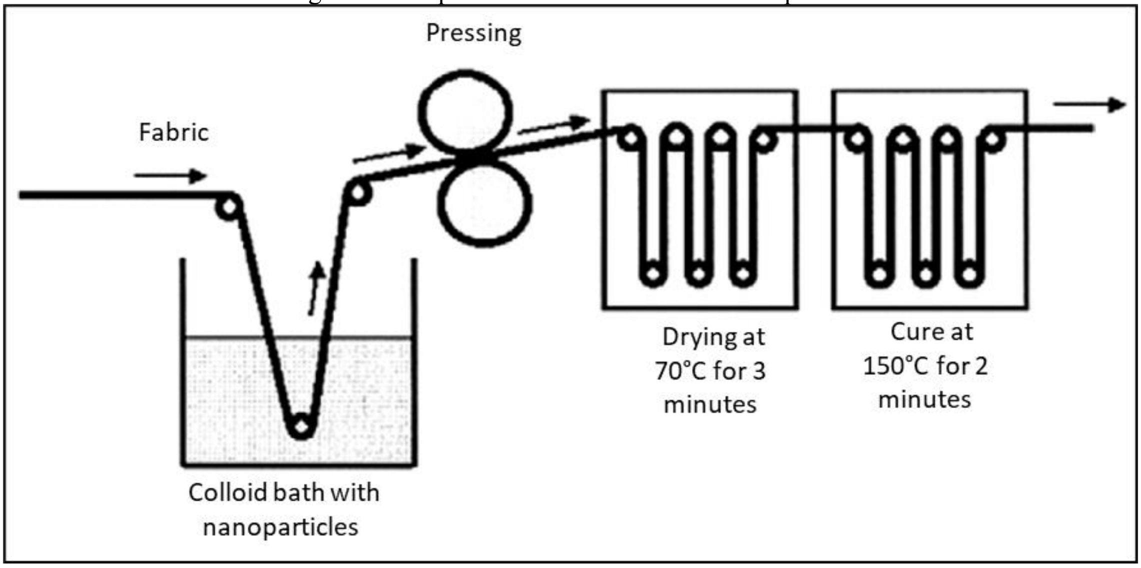
The authors proved that the loaded fibers exhibited excellent antimicrobial activity against *Escherichia coli*. It was also observed that, after an immersion time of around 100 hours, in saline solution, there is the detachment of approximately 25% of the nanoparticles of the nanocomposite.

El-Rafie *et al*. (2010) prepared Ag NPs (with average diameter around 5 nm) using AgNO3 and the biomass filtrate of the fungus *Fusarium solani* as a reducing agent, under ambient conditions and for 48 hours.

Then, the colloid obtained was applied to cotton fabrics, with and without the aid of a binding agent (in this case, the liquid Printofix Binder MTB EG). The preparation scheme of the nanocomposites is shown in Figure 5, where it is possible to observe a process of foulardagem (pressing) and a curing step at 150° C for 2 minutes.

The study determined the antimicrobial activity of the nanocomposites obtained, against the bacteria *Escherichia coli* and *Staphylococcus aureus*, before and after 20 washing cycles. It was found that the average initial microbial reduction was 94 % for the two microorganisms tested. After washing the nanocomposite without the binding agent, this average percentage decreased to 50,85 %. On the other hand, the addition of the binding agent caused the average percentage to decrease to 89.5%.

Figure 5 – Preparation scheme of the nanocomposite.



Source: Adapted from EL-RAFIE *et al*. (2010, p. 780).

El-Rafie *et al*. (2012) deepened the study done in 2010 and optimized the condition for the biosynthesis of Ag NPs with the following parameters: AgNO3 concentration (0.078 g/100 mL), biomass concentration (10 g/100 mL), pH of the reaction medium (12), temperature (25° C) and reaction time (24 hours).

Thus, well-stabilized nanoparticles were obtained at a concentration of 2000 ppm, with an average diameter between 8 and 15 nm. The authors also report that the reduction of $Ag^{1+ \text{ cations}}$ occurs by the action of enzymes and proteins excreted by the fungus and present in the biomass filtrate.

Yang *et al*. (2012) proposed, for the first time, a method of preparation of Ag NPs by hydrothermal synthesis, using bacterial cellulose (cellulose polymer synthesized by some bacteria and consists of nanofibers in network) as the only reducing and stabilizing agent, in the form of purified membranes and soaked with AgNO3 solution.

The synthesis was performed in a sealed container of polytetrafluoroethylene (PTFE), at 80° C, and lasted 4 hours. The hydroxyl groups present on the surface of the nanofibers were responsible for the reduction of the $Ag^{1+ \text{ cation}}$ and subsequent stabilization of the nanoparticles formed. They were able to obtain nanoparticles with an average diameter between 11.2 and 23 nm. The nanocomposite showed a good bactericidal action against *Staphylococcus aureus,* which was evaluated by the MIC assay, in triplicate and with the appropriate controls.

Although cotton was not used as a matrix, but bacterial cellulose, the proposal was interesting because it employed an environmentally correct preparation method. This process can be experimented with other cellulose compounds (perhaps oxidized cotton fibers) to obtain products with bactericidal action, such as self-sterilizing fabrics.

El-Rafie, El-Rafie and Zahran (2013) synthesized Ag NPs using as reducing agents, as well as stabilizers, water-soluble polysaccharides extracted from four marine macroalgae (*Pterocladia capillacae*, *Jania rubins*, *Ulva faciata* and *Colpmenia sinusa*). The synthesis was developed for 60 minutes, under agitation, at a temperature of 70° C and with the pH of the system adjusted to 10. The $AgNO_3$ concentration was 0.1 mmol/L.

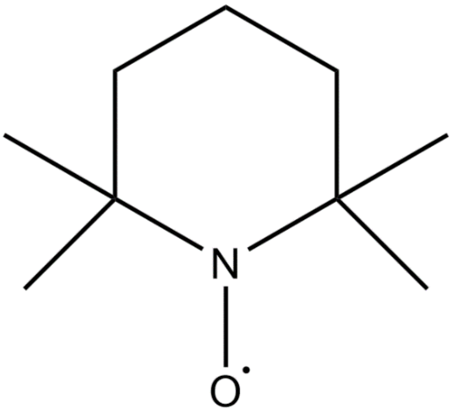
The nanoparticles obtained were characterized by UV-Vis and SEM. The smallest mean diameter was observed for the nanoparticles synthesized using extracts of *Ulva faciata* and *Pterocladia capillacae* (7 nm). The largest mean diameter was observed for those prepared using the extract of *Colpmenia sinusa* (20 nm).

Then, silver colloids were applied to cotton fabrics in the absence and presence of citric acid (2%) or a binding agent (1%). The impregnation of the particles was followed by a drying process (at 70° C and for 3 minutes) and a curing process (at 150° C and for 2 minutes).

The antimicrobial activity of the resulting nanocomposites was evaluated against the bacteria *Escherichia coli* and *Staphylococcus aureus*, before and after a certain number of repeated washing cycles. The study concluded that it is a function of the previous treatment received by the tissue, the size of the Ag NPs and the species of algae used to extract the polysaccharide. The best result obtained was the one in which the nanoparticles were prepared using the extract of *Ulva faciata* and the tissue was previously treated with the binding agent, even after 10 washing cycles.

Cao *et al*. (2013) deposited, *in situ*, Ag NPs on jute fibers previously submitted to the selective oxidation method by the compound known as TEMPO, which is the radical (2,2,6,6-tetramethylpiperidine-1-yl)oxy (Figure 6). The fibers were immersed in an aqueous solution of $AgNO_3$ at a concentration of 0.015 mol/L, sheltered from light, during the night. The system was then heated in a domestic microwave oven. As the heating time increased from 5 to 10 minutes, the average diameter of the nanoparticles obtained was from 50.0 to 90.0 nm.

Figure 6 – Structure of TIME.



Source: Author (2023).

Jute is a very hygroscopic vegetable textile fiber that contains in its composition, on average, 65 % cellulose. It is extracted from the plants of the genus *Corchorus*, widely cultivated in Asia. The oxidation of the fibers (initially treated separately with aqueous solution of sodium hydroxide (NaOH) and dimethylsulfoxide bath) by TEMPO causes the hydroxyl group of position 6 of the cellulose to be oxidized to an aldehyde or carboxyl group, which will act simultaneously in the reduction of the Ag $^{1+}$ $^{\text{cations}}$ and in the stabilization of the nanoparticles formed. This process is carried out under normal conditions of temperature and pressure, with pH adjusted between 10 and 11 and in the presence of sodium bromide (NaBr) and sodium hypochlorite (NaClO).

The authors indicated that the prepared nanocomposite could be useful in applications such as: ultrafiltration, antibacterial dressings, food packaging, water treatment and in the biomedical area.

7 FINAL CONSIDERATIONS

The impregnation of cellulose fibers, especially cotton, with Ag NPs is already a reality to prepare nanocomposites with antimicrobial effect that can be used in health products. Both *in situ* and *ex situ* syntheses are used to obtain Ag NPs.

It is interesting to observe that less aggressive preparation methods are sought to the environment when biosyntheses with various natural reducing agents are proposed. In these, the energy consumption is minimal. Perhaps, for this reason, the physical methods of nanoparticle synthesis have not been prioritized.

Another point that deserves to be highlighted is the concern with the durability of the nanocomposite. Once the nanoparticles are impregnated on the surface of the fibers, they can detach from it, which would lead to a decrease in microbial reduction.

Given this possibility, most publications presented results of tests that verified what happens to the material after a series of washes, either by evaluating the antimicrobial activity or by the silver content in the washing water. Regarding antimicrobial assays, there was a predominance of tests against the bacteria *Escherichia coli* and *Staphylococcus aureus*.

Also to minimize the detachment of the nanoparticles, some binding agents or coating layers were used to better fix the nanoparticles in the fibers. These were also previously treated to improve the adhesion between matrix and load.

It is also possible to emphasize the evaluation of the color of the samples after the impregnation of the Ag NPs, based on the results of color measurements in the CIE scale L*a*b*, even if, compared with the antimicrobial effect, the appearance is not in the foreground.

The techniques that predominated in the characterization of nanoparticles or nanocomposites were: DRX, MET, UV-Vis and EDS. Mechanical assays were not much emphasized in the publications, since they are not determinant for the use of nanocomposites of which the antimicrobial effect is primarily desired.

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