Antimicrobial activity of bleached cattail fibers (*Typha domingensis*) impregnated with silver nanoparticles and benzalkonium chloride.

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Abstract: Lignocellulosic fibers are renewable and sustainable materials, and their use can be expanded when modified. This study aims to investigate the chemical modifications in cattail fibers (*Typha domingensis* - *Taboa*) pretreated with alkaline hydrogen peroxide (bleaching) and subsequently chemically modified by Tollens' reagent for the deposition of silver nanoparticles (NP's) on the surface fibers and with commercial quaternary ammonium salts (PolyCLBZ 50®, PolyQuat 08® and PolyBac QT 80® from POLYORGANIC® Tecnology LTDA, São Paulo, Brazil), in order to provide antimicrobial properties for these materials. After the treatments, they were characterized by scanning electron microscopy, Fourier transform infrared spectroscopy (FTIR), thermal properties (TGA/DTG) onset temperature (T_{onset}), maximum degradation temperature (Tmax) and residual mass, in addition to the profile of antimicrobial susceptibility by means of disk-diffusion, against *Staphylococcus aureus* (ATCC 25923), *Escherichia coli* (ATCC 25922), *Salmonella typhimuruim* (ATCC 14028) and *Salmonella enteritidis* (ATCC 13076). As a result, through SEM it was possible to observe the silver nanoparticles on the surface of bleached fibers, using FTIR, the presence of benzalkonium chloride was confirmed with the reduction of the 3,400 cm^{-1} region, as well as by the presence of defined peaks in the region of 2,921 - 2,851 cm^{-1} that can be attributed to characteristic stretches of alkanes binding (CH bonds), and the increase in intensity by 1,383 cm^{-1}, referring to tertiary amine. The treatments applied influenced the thermal properties of the samples, with a small reduction in stability when compared to the control fiber, as well as showing an increase in residual mass. Regarding the susceptibility profile to microorganisms, it was found that AgNP's are efficient for gram negative bacteria *E. coli, S. typhimuruim,*

S. enteritidis. Samples with different concentrations of benzalkonium chloride showed better activity against gram-positive bacteria *S. aureus*.

Keywords: Lignocellulosic fibers; Physical-chemical modifications; Immersion; Impregnation; Tollens reagent; Benzalkonium chloride; Thermal properties; Antimicrobial activity; Disc-Diffusion; Gram-Positive/Gram-Negative Bacteria.

1. Introduction

The use of cellulose is an area in constant expansion, not only for the production of high-volume commodities, such as textiles and paper, but also for the discovery of new materials with high added value, such as functionalized fibers and reinforcement elements based on from natural fibers to composite materials (Belgacem & Gandini, 2005; Gandini & Belgacem, 2011), biocomposites and nanocomposites.

Considering the growing public health awareness about the pathogenic effects caused by some microorganisms, there is a growing need to obtain antibacterial materials, a strand with several areas of application, such as medical devices, hospital materials, surgical equipment, hygiene applications household, textiles, food packaging and storage (Goldade & Vinidiktova, 2017).

Cattail (*Typha domingensis*) is an infesting aquatic macrophyte, considered a perennial and emerging herbaceous that belongs to the *Typhaceae* family (Apfelbaum, 1985) is a perennial herbaceous plant), which develops spontaneously throughout tropical and subtropical America (Bajwa et al., 2015).

Silver is the best known of the various heavy metals' toxic to microorganisms (Borsa, 2012). Silver nanoparticles have efficient antimicrobial properties compared to other salts due to their extremely large surface area, which provides better contact with microorganisms. It is assumed as a mechanism of action that the nanoparticles adhere to the cell membrane and penetrate inside the bacteria. The bacterial membrane contains proteins with sulfur and the silver nanoparticles interact with these proteins in the cell, as well as with compounds containing phosphorus, such as DNA. When AgNPs enter the bacterial cell, they form a low molecular weight region at the center of the bacterium, in which the bacterium clumps together, protecting the DNA from silver ions. Thus, they preferentially attack the respiratory chain, affecting cell division

and, ultimately, leading to death. Nanoparticles release silver ions in bacterial cells, which still increase their bactericidal activity (Rai et al., 2009).

Quaternary ammonium salts are cationic surfactant compounds with long alkyl groups, considered to be well-known disinfectants. These carry a positive charge on the nitrogen atom (N^+) in solution. The antimicrobial effect of these, in general, is due to an electrostatic rupture of the cell wall after invasion by a lipophilic chain of specific length. The agent is not consumed in the process; therefore, the antimicrobial activity is continuous, if this bioactive material is adhered to the surface. It is considered unlikely that microorganisms will become resistant to this type of attack, as this would involve an important change in the structure of their cell wall (Borsa, 2012; Thomas et al., 2009).

Surface modifications in natural fibers are widely studied and have shown promising results in several fields of research. Cruz and Fangueiro (2016) did a bibliographic review on the changes in natural fibers (Cruz & Fangueiro, 2016). Examples include the study by Kulpinski et al., (2012), which obtained luminescent cellulose fibers (Kulpinski et al., 2012). Xiao et al., (2017) developed an easy strategy for the preparation of cellulose fibers functionalized with alkynes with click reactivity (Xiao et al., 2017). Sabzalian; Alam; Van De Ven, (2014) investigated through the application of chemical processes the transformation of hydrophilic cellulose fibers into non-hygroscopic hydrophobic fibers (Sabzalian et al., 2014). Chauhan and Mohanty (2015) studied an easy one-step hydrothermal method for coating titanium (TiO_2) nanoparticles (diameter of about 40-250 nm) on the surface of cellulose fibers that were used to prepare paper matrices and showed excellent activity photocatalytic for degradation the methyl orange dye in the presence of ultraviolet light and also showed promising antibacterial activity against *Escherichia coli* (Chauhan & Mohanty, 2015). Li et al., (2015) prepared cellulose/silver nanocomposite fibers by immersing the cellulose fibers in an aqueous solution of $AgNO_3$, to synthesize Ag nanoparticles *in situ*, in this study the experiment antibacterial has proved to be excellent antibacterial activity of cellulose nanocomposite fibers against *Staphylococcus aureus* (Li et al., 2015). At the forefront, Lazić et al., 2020, developed silver nanoparticles coated with dextran to improve the barrier and control the antimicrobial properties of nanocellulose films used in food packaging (Lazić et al., 2020).

In this sense, to obtain versatile materials with antimicrobial activity, this study investigated physical-chemical processes of incorporation of silver nanoparticles and

impregnation of commercial quaternary ammonium salts in bleached cellulose fibers. These modifications of modifications appear as promising alternatives to obtain sustainable materials.

2. Material and methods

2.1 Raw material and chemicals

Cattail fibers (*Typha dominguensis*) were obtained directly from farmers in Dourados (Lat: 22 ° 13'18 " South, Lon: 54 ° 48'23 " West), Mato Grosso do Sul - Brazil, in July 2016 The samples were dried in an oven at 50ºC with air circulation for 24 hours and crushed in a knife mill with mesh> 100 mesh and stored in plastic containers.

The reagents used in the chemical treatments (mercerization and bleaching) such as Sodium Hydroxide (NaOH) and Hydrogen Peroxide (H_2O_2 50%) were acquired from Sigma Aldrich® and Dinâmica® in an analytical degree. Silver nitrate ($AgNO_3$), ammonium nitrate (NH_4NO_3), analytical grade dextrose acquired from Merck Co® were used to prepare the Tollens reagent.

The commercial quaternary ammonium compounds used were donated by the company POLYORGANIC® Tecnology LTDA (São Paulo, Brazil). Whose trade names are: PolyCLBZ 50®, PolyBac QT 80® and PolyQuat 08®, both obtained from high purity coconut oil fatty acids through reactions with benzyl chloride, following strict specifications of the international pharmacopoeia. According to the manufacturer, the PolyCLBZ 50® product has a 50% concentration of active ingredient, the rest represented by solvent and water. The PolyBac QT 80® product has a molecular weight of 353.90 g/mol, amine salt 1.0% maximum and 80% concentration of active ingredient. The PolyQuat 08® product is a solution containing dodecyl dimethyl benzyl ammonium chloride, solvent and water, 80% in concentration, which has a molecular weight of 340.0g/mol, with a chain distribution (Typical) in 70% C12 and 30% C14 (PolyorganicTecnologiaLTDA, 2012, 2014a, 2014b).

For the disc-diffusion antimicrobial analysis, Brain hearth infusion broth (BHI) Muller Hinton (MH) from Sigma Aldrich® (Bauer et al., 1966) was used.

2.2 Alkali and alkaline/hydrogen peroxide treatment

The mercerization and bleaching of natural fibers is an important process for the purification of cellulosic material, as, among benefits, it increases the availability of cellulose molecules for interaction with the active agents used.

The first treatment is carried out in an alkaline solution of sodium hydroxide (NaOH) at 5% in the proportion 1:20 $g.mL^{-1}$ for 4 hours in the form of 190 rpm in a shaker at room temperature. Subsequently, the fibers were washed in water until neutral pH and dried in an oven with air circulation for 24 hours at 50 ° C (Negawo et al., 2019; Vijay et al., 2019).

A further bleaching step was carried out in an alkaline/hydrogen peroxide solution in the proportion of 1:20 $g.mL^{-1}$ of solution. This solution was prepared in a 1: 1 ratio with 50% hydrogen peroxide and 5% sodium hydroxide at 50°C under continuous preparation for 1 hour. Afterwards, the fibers were washed in water until neutral pH and dried in an air circulation oven at 50 ° C for 24 hours. With the dry fiber, the third and fourth stages were carried out containing only 1:1 oxidizing hydrogen peroxide and distilled water under preparation at 50 ° C for 1 hour. After this process, the fibers were washed with distilled water and again dried in an oven with air circulation at 50 ° C for 24 hours, then stored in plastic containers for later stages.

2.3 Tollens reagent

The Tollens reagent was prepared based on the methodology proposed by Montarez, *et. al.,* (2012), and Michalcová *et al.,* (2018) with adaptations. First, silver nitrate (0.5M) ($AgNO_3$), ammonium nitrate (1.5M) (NH_4NO_3), and dextrose (2M) ($C_6H_{12}O_6$) solutions were prepared. Both prepared at room temperature and atmospheric pressure. The ammonium nitrate solution (NH_4NO_3) was used as a precursor, in which the second silver nitrate solution ($AgNO_3$) was added slowly until the ratio 1:1 mL, with stirring (500rpm).

Initially, there was the formation of a brown precipitate, silver amine (complex ion $[Ag(NH_3)^2]$ + (Tollens' reagent), which disappeared after the complete mixing of the solutions. Then 3mL of the dextrose solution was added, and the Tollens solution was heated to 100ºC. Afterward, 10g of bleached cattail fiber were added, which remained in constant agitation (500rpm) at 100ºC for 30 minutes. At the end of the process, the fiber, initially white, showed a reddish-brown color. With the addition of dextrose (aldehyde group) the Tollens reagent oxidizes the aldehyde group to a carboxylic acid

and, at the same time, induces the reduction of silver, in metallic silver, deposited on the fibers. Process can be simplified in Equation 1.

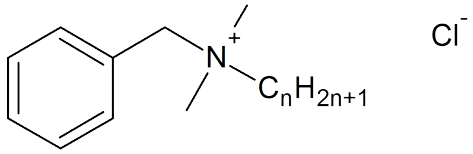
$$Ag(NH_3)_2{}^{+}(aq) + RCHO_{(aq)} \rightarrow Ag_s + RCOOH_{aq} \quad (1)$$

Fonte: (Yin et al., 2002).

Subsequently, the fibers were washed with 500 mL of distilled water and dried in an oven with air circulation at 50ºC for 24 hours. After synthesis, the effluents generated were acidified with dilute acid before disposal, to avoid the formation of explosive silver nitride (Michalcová et al., 2018; Montazer et al., 2012).

2.4 Quaternary ammonium salts solutions

The commercial quaternary solutions used had dodecyl dimethyl benzyl ammonium chloride (benzalkonium chloride) – Fig. 1, solvent and water, with a molecular weight of 340 g/mol. After the bleaching process, the dried fibers were immersed in different solutions containing quaternary ammonium salts.



n = 8, 10, 12, 14, 16, 18

Fig. 1 Simplified typical chemical structure of benzalkonium chloride.
Source: (Álvarez-ros, 2018).

The solutions were prepared with the following concentrations of active ingredient: 03.PolyCLBZ 50® [4%], 04.PolyQuat 08® [8%] and 05.PolyBac QT 80® [10%], 06.PolyQT 08® [8 %]. In reagent glass flasks, solutions 03, 04 and 05 were heated to 50ºC, while solution 06 was heated to 85ºC, after which bleached cattail fibers [1g for each 50 mL] were added and the flasks were closed, 03, 04 and 05 remained immersed and under constant magnetic stirring (500 rpm) for 24 hours, while solution 06 remained in the previous conditions for 6 hours (Adapted from He et al., 2014; Wei et al., 2011). After the entire immersion process, the fibers were washed

with 500 mL of distilled water and dried in an oven for 24 hours at 50ºC. Subsequently stored in plastic containers for future analysis.

2.5 Scanning electron microscopy (SEM)

The microstructure and homogeneity of the modified fibers were observed using SEM in a device called Quanta 200 FEI (Everhart - Thornley detector). Before observation, samples were coated with gold. The sections were observed under an acceleration voltage of 10 kV. Analysis performed at the LGP2 of the Polytechnic Institute of Grenoble-France.

2.6 Fourier transform infrared spectroscopy (FTIR)

The analysis was performed using spectrophotometer model FT/IR-4100 type A (JASCO®, Japan), transmission in KBr tablet. The spectra were recorded from 4000 to 500 cm − 1 of wave numbers, with a resolution of 4 cm^{-1} and 32 scans, the analysis was performed at the UFGD Optics Laboratory.

2.7 Thermal properties

Fiber samples of approximately 4 mg were placed in the platinum sample port and subjected to a temperature range of 30 to 600°C at a heating rate (β) of 10ºC.min^{-1} and nitrogen atmosphere with a flow of 20 ml/min. The equipment used was the simultaneous thermal analyzer STA 449 F3 Jupiter® (STA449 F3, NETZSCH®, Germany), an analysis was carried out at the UFGD Thermal Analysis Laboratory. Mass loss (TG curve) and first derivative (DTG curve) were compared as a function of time/temperature.

2.8 Antimicrobial susceptibility test

To analyze the antimicrobial activity of the fibers after the modification, the disk diffusion test (BAUER et al., 1966) was used, in which the standard species of pathogens such as Staphylococcus aureus (ATCC 25923), Escherichia coli (ATCC 25922) were tested , Salmonella enteritidis (ATCC 13076) and Salmonella

typhimurium (ATCC 14028). It is a test that produces a qualitative result, such as the classification of organisms in resistant, intermediate, or susceptible (ANVISA, 2005; Sader & Pignatari, 1994; Performance Standards for Antimicrobial Disk Susceptibility Tests, 13th Edition, 2018).

The microorganisms were reactivated in BHI broth (Brain hearth infusion) for 24h in an oven at 37°C, sown on Muller Hinton agar (MH), and incubated for another 24h at 37°C, to obtain pure cultures. The test is carried out by applying a bacterial inoculum of approximately 1–2 × 10^8CFU/mL to the surface of a large Mueller-Hinton agar plate (150 mm in diameter), in which up to 12 discs can be placed on the surface of the inoculated agar. Subsequently, the plates are incubated for 16–24 h at 35 ° C. The results are obtained by forming growth inhibition zones around each of the disks used and measured with millimeter precision. The diameter of the zone is related to the susceptibility of the isolate and the diffusion rate of the active agent in the agar (Jorgensen & Ferraro, 2009).

2.9 Statistical analysis

The Statistica® 7.0 (StatSoft) software was used to calculate variance analyzes (ANOVA) applied Tukey's test to determine differences between fibers properties in the 95% confidence interval. To measure the diameters of the AgNP's, the free software ImageJ® was used.

3. Results and discussion

3.1 Initial characteristics, morphology, and infrared spectroscopy with Fourier transform (FTIR)

To contextualize the initial characteristics, Table 1 presents some data referring to cattail fiber, before and after the bleaching process, in which it is possible to observe improvements in the physical-chemical properties, such as increased cellulose content, luminosity, crystallinity, onset temperature, maximum degradation temperature and reduction in waste.

These chemical modifications referring to the purification of the cellulosic material (mercerization and bleaching), are necessary to increase the availability of

hydroxyls (-OH) for the interaction with the compounds impregnated during the functionalization of the material (Gandini & Belgacem, 2011). Some literature records also show increases in cellulose content, crystallinity after mercerization/bleaching procedures, such as cotton fibers (Adel et al., 2016), rice straw (Johar et al., 2012) and *Cocconia grandis* L. fibers (Senthamaraikannan & Kathiresan, 2018).

Table 2 presents a brief description of the fiber samples with a summary of their process and the code used for their identification.

As shown in Table 2, samples 04. CB + PQuat08 [8%] and 06. CB + PQT [8%] 85ºC have the same concentration of active principle, differing only in the method of obtaining, since fiber 06 was obtained with heating to 85ºC and 6 hours of immersion under agitation. Silver and other metals (and their ions) can be linked to cellulosic fibers by several methods (Borsa, 2012), as examples the use of silver salt incorporated in dressings with hydro cellulosic fibers used for wound healing, which presented promising results, in helping to eliminate pathogens and wound healing (Newman et al., 2006), as well as obtaining cotton fabrics padded with colloidal silver nanometer solution (Lee et al., 2003).

In the images obtained by SEM – Fig. 2, it is possible to observe the presence of silver nanoparticles deposited on the surface of the cattail fibers - Figure 2-02. While the samples treated with different concentrations of quaternary ammonium remained with homogeneous structures similar to the control fiber - Figure 2-01.

Fig. 2 - 02 shows the spherical structure of the AgNP's, with the aid of the ImageJ® software, it was possible to measure their size, whose average area measures 204 ± 0.06 nm. Montazer et al. (2012), in his study on the direct synthesis of silver nanoparticles by Tollens' reagent in cotton fabric observed the formation of nanoparticles that measured an average of 88 nm (Montazer et al., 2012). Thus, the variation in the size of the nanoparticles is related to the synthesis process, studies indicate that smaller particles were formed in lower concentrations of ammonia (Pingali et al., 2008; Sharma et al., 2009). The size of the nanoparticle is important, as it directly influences antimicrobial activity.

The FTIR spectra for the different modified and unmodified samples are contained in Fig.3. The widest peak around 3,400 cm^{-1} for samples 01. CBC, 02. CB + AgNp's, 03. CB + PCLBZ [4%] and 06. CB + PQT [8%] 85ºC - 6h is due to elongation of the cellulose -OH groups (Amroune et al., 2015), in samples 03. CB + PCLBZ [4%], 04. CB + PQuat08 [8%] and 05. CB + PBacQT [10%], there was a narrowing in this

region indicating that new interactions arose from immersion in different concentrations of quaternary ammonium salts. The sample 06.CB + PQT 85% [8%] has the shortest reaction time, 6 hours, in comparison to the other samples, a fact that may justify maintaining the presence of the enlarged region at 3,400 cm^{-1}.

Comparing the spectra of the modified fibers with quaternary in relation to the control fiber, the presence of the active principle (benzalkonium chloride) is observed, which was confirmed through the greater definition of the two peaks present in the region of 2,921 - 2,851 cm^{-1} that can be attributed to characteristic stretches of CH-type alkanes (Carvalho et al., 2009; Le Troedec et al., 2008; Ventura-Cruz et al., 2020), evident especially in samples 04. CB + PQuat08 [8%] and 05. CB + PBacQT [10%], in addition to the reduction of the extended band around 3,400 cm^{-1}. In the region of 1,626 cm^{-1} there is a characteristic peak of cellulosic materials that can be attributed to the C=C aromatic vibration (Mahltig et al., 2004). This peak was more intense in samples 04. CB + PQuat08 [8%] and 05. CB + PBacQT [10%] treated with quaternary ammonium.

Yue et al., (2019) in their study on the addition and controlled release of benzylammonium chloride (BAC) in nano-clays, as well as in this experiment also observed the appearance of two new bands in the range of 2,900–2,850 cm^{-1}, after the addition of BAC, which were also attributed to the symmetric and asymmetric C - H_2 elongation vibration of the alkyl group in BAC (Yue et al., 2019)

In the region of wave number 1,383 cm^{-1}, the signal amplification for samples 04. CB + PQuat08 [8%] and 05. CB + PBacQT [10%] can be attributed to the angular deformation C—H of the methyl group (CH_3) , as well as, can be defined as a characteristic region of tertiary amines, intensified due to modification with benzalkonium chloride (Liu et al., 2013). The C-O-C vibration of cellulose can be observed by the prominent peak in the 1,057 cm^{-1} band (Adel et al., 2016). At 664 cm^{-1}, it's possible to see peaks referring to C-OH outside the plane referring to the cellulose molecule (Le Troedec et al., 2008).

There are many -OH groups and in the cellulose fiber, which can be used to reduce Ag^+ in Ag. After the fibers are immersed in the Tollens reagent, it is considered that it has penetrated their micropores, being then reduced by the -OH groups of the cellulose. at 100°C, and simultaneously the cellulose was oxidized, a similar fact was observed in the study by Li et al., (2015).

For the 02.CB + AgNp's sample, no characteristic bands were identified in its spectrum, only that it remained similar to the 01.CBC control fiber. Li et al., (2015) attributed the similarity of the modified spectra with silver nanoparticles to the control fiber, the low concentration of $AgNO_3$ that would have oxidized few cellulose molecules was oxidized, maintaining largely the cellulose structure.

With the data obtained, it appears that both the silver nanoparticles and the samples with different concentrations of commercial ammonium quaternary were successfully impregnated.

3.2 Thermal properties and evaluation of the antimicrobial activity of fibers

The thermal properties of a material are important to help define parameters such as its use limitation. Fig. 4 shows the thermogravimetric (TG) and derived thermogravimetric (DTG) curves obtained for the modified and unmodified fiber samples.

The data obtained through the thermogravimetric analysis are compiled in Table 3. Thus, it is observed that the control sample 01.CBC has the greatest thermal stability, or 336ºC in Tonset, followed by the sample with 02. CB + AgNp's with 326ºC, and subsequently the sample 03.CB + PCLBZ [4%] and 05. CB + PBacQT [10%] at 321ºC, samples 04. CB + PQuat08 and 06. CB + PQT 85ºC [8%] at 311ºC and respectively the concentrations of 8% of active ingredient. Observing the DTG curves - Figure 4-B, it can be seen in the samples with quaternary, the formation of a first peak around 219ºC, with the following values of mass loss of about 5% for samples 02. CB + AgNp's, 03.CB + PCLBZ and 06. CB + PQT, about 7.5% for sample 04. CB + PQuat08 and about 10% for sample 05. CB + PBacQT. Another event can be noticed in the region of 282ºC, with a loss of mass of about 10% for sample 02. CB + AgNp's, about 14% for sample 06. CB + PQT, approximately 17% for sample 03. CB + PCLBZ e, 22% for sample 05. CB + PBacQT. These events are related to the degradation of benzalkonium chloride in the samples.

The quaternary concentration influences the thermal stability of the samples, for the item maximum temperature of degradation, methodological differences for obtaining samples 04 are still considered. CB + PQuat08 [8%] and 06. CB + PQT 85ºC [8%] as the temperature and time of obtaining differ in the number of final residues sampled, being 5.5% to 3.5%.

In quantitative terms, when we try to better understand the concentration of active principles in the samples, we can, with the help of the residual mass, calculate the percentages of non-decomposed and non-volatile inorganic materials present in the samples of AgNP's and commercial benzalkonium chloride. Thus, in Table 3 the last column is reserved for the concentration of inorganic solids, corrected in relation to the control sample 01.CBC.

As an estimate, it can be said that the sample 02. CB + AgNp's, has a concentration of 3% of AgNP's. As for samples with quaternary ammonium, the number of insoluble solids at 558ºC varies according to the concentration and process of application applied. These values may be related to the purity of the reagents used, since these are commercial compounds.

The susceptibility profile to microorganisms was traced using standardized strains and using the disk-diffusion technique - Figure 5. In Table 4, it is possible to verify the values of inhibition halos obtained for the different samples, as well as their control with known antibiotics. For the disk-diffusion test, 6 mm diameter discs obtained by mechanically compacting the fibers in a metallic mold were standardized.

Figure 5 shows the zones of inhibition of the fibers impregnated against *Staphylococcus aureus* (ATCC 25923) (gram-positive bacteria), *Escherichia coli* (ATCC 25922), *Salmonella typhimuruim* (ATCC 14028) and *Salmonella enteritidis* (ATCC 13076) (gram-negative bacteria) respectively.

Pure bleached cattail fibers and antibiotics such as Oxacillin (1mg) and Amikacin (30µg) were tested as control samples, for bleached cattail samples, no zone of inhibition appeared, which illustrated that the fibers in question had no antibacterial activity. The positive controls showed inhibition halos of about 18.5 mm. The sample 02.CB + AgNp's, showed activity only against gram-negative microorganisms, *E.coli, S. typhimuruim, S. enteritidis*, with medium halos of 8mm, we consider the microorganisms tested for this sample condition resistant. The sample 03.CB + PCLBZ, showed inhibition halos for the samples of gram-positive and negative microorganisms, with clear inhibition zone for *S. aureus* 15mm, considered intermediate, *E.coli*, 7mm, and *S. typhimuruim*, 8mm , considered resistant. Samples 04.CB + PQuat08, 05.CB + PBacQT and 06.CB + PQT 85ºC, presented inhibition halos against gram-positive and negative microorganisms, *S. aureus*, values close to 15 mm, considered intermediate and *E. coli, and S. enteritidis*, averages of 8.5 mm considered resistant.

Regarding the results, its possible consider that all the modified samples of this study showed relevant and significant antibacterial activity.

In the study by Mahltig et al., (2004) silica materials embedded with layers of silver, silver salts and biocidal salts of quaternary ammonium (cetyltrimethylammonium bromide and octenidine) were obtained and it was found that the growth of fungi and bacteria could be inhibited (Mahltig et al., 2004).

Li et al., (2015) cellulose fibers were treated with silver nanoparticles and tested for antibacterial activity against strains of *Staphylococcus aureus* and *E. Coli*, the diameter of the inhibition zone and the photos taken were measured. It was found that with the increase in the content of silver nanoparticles and the size of the particles in the composite fibers, the antibacterial capacity increased. However, the inhibition halo obtained for *E.coli* (21.0 mm) was smaller than that obtained for *Staphylococcus aureus* (28.5 mm) for others silver became antibacterial through the destruction of the bacterial cell wall, considering that the cell wall of these two bacteria was quite different. The cell wall of *Staphylococcus aureus* is thicker with less protein content than the cell wall of *E. coli*, therefore, *Staphylococcus aureus* is more easily (Li et al., 2015).

In this study, however, there was no halo of inhibition *for Staphylococcus aureus*, among the justifications it is mentioned that the antimicrobial effects of silver NPs depend on their size, shape and concentration (Rai et al., 2009).

Son et al., (2006) in his study on the transmission of durable antimicrobial properties to cotton fabrics using quaternary ammonium salts, tested the antimicrobial activity of the cotton sample treated against Staphylococcus aureus according to the AATCC 100- 1999, and found that the treated tissue showed greater antimicrobial activity compared to the untreated tissue (Son et al., 2006).

Pinto et al., (2009) in their study to obtain cellulose nanocomposites/Ag found about the antibacterial activity carried out for Bacillus subtilis, Staphylococcus aureus and Klebsiella pneumoniae that the silver nanoparticles present in cellulosic fibers in concentrations as low as 5, 0 × 10^{-4}% by weight make these nanocomposites effective antibacterial materials (Pinto et al., 2009).

Finally, Volova et al., (2018) describes the production of bacterial cellulose (BC) composites with silver nanoparticles and antibiotics and compares their properties, the disk diffusion method and the agitated flask culture method were used and showed that all experimental composites had activity antibacterial pronounced against *E. coli,*

Ps. eruginosa, K. pneumoniae and St. aureus, and BC/antibiotic compounds were more active than BC / AgNp; S. aureus was the most susceptible to the effect of BC composites (Volova et al., 2018).

4. Conclusions

Bleached cattail fibers were modified by physical-chemical processes of impregnation of silver nanoparticles using Tollens' reagent, and commercial quaternary ammonium salts were successfully obtained. The nanoparticles were adhered to the surface of the fibers, and had, on average, an area of 200 nm. Modifications in FTIR and TGA/DTG were identified that confirm the impregnation of benzalkonium chloride in different proportions in the samples. Regarding antimicrobial activity, positive and satisfactory responses were obtained to control contamination by everyday microorganisms. AgNP's are effective for gram negative bacteria *E. coli, S. typhimuruim, S. enteritidis*. Samples with different concentrations of benzalkonium chloride showed better activity against gram-positive bacteria *S. aureus.* We anticipate that the versatile use of these cellulosic materials can bring a promising strategy in obtaining a wide range of materials with important antimicrobial activities.

Acknowledgments

The Brazilian institutions for research promotion: CAPES, CNPQ, FUNDECT. CAPES/BR: Marivane Turim Koschevic/Sandwich Doctorate Program abroad - PDSE - Notice no. 47/2017 - Selection 2018/Process Number: 8881.189683/2018-01.

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