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Physical and Mechanical Characterization of a Functionalized Cotton Fabric with Nanocomposite Based on Silver Nanoparticles and Carboxymethyl Chitosan Using Green Chemistry

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Citation: Quispe-Quispe, L.G.; Limpe-Ramos, P.; Arenas-Chávez, C.A.; Gomez, M.M.; Mejia, C.R.; Alvarez-Risco, A.; Del-Aguila-Arcentales, S.; Yáñez, J.A.; Vera-Gonzales, C. Physical and Mechanical Characterization of a Functionalized Cotton Fabric with Nanocomposite Based on Silver Nanoparticles and Carboxymethyl Chitosan Using Green Chemistry. *Processes* **2022**, *10*, 1207. <https://doi.org/10.3390/pr10061207>

Academic Editor: Irena Zizovic

Received: 20 April 2022

Accepted: 30 May 2022

Published: 17 June 2022

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Abstract: Cotton is the most widely used natural fiber for textiles but its innate capacity to absorb moisture, retain oxygen, and high specific surface area make it more prone to microbial contamination, becoming an appropriate medium for the growth of bacteria and fungi. In recent years, the incorporation of silver nanoparticles in textile products has been widely used due to their broad-spectrum antibacterial activity and low toxicity towards mammalian cells. The aim of the current study is to synthesize and characterize a nanocomposite based on silver nanoparticles and carboxymethyl chitosan (AgNPs-CMC), which was utilized to provide a functional finish to cotton fabric. The scanning electron microscope (SEM) to produce a scanning transmission electron microscope (STEM) image showed that the nanocomposite presents AgNPs with a 5–20 nm size. The X-ray diffraction (XRD) analysis confirmed the presence of silver nanoparticles. The concentration of silver in the functionalized fabric was evaluated by inductively coupled plasma optical emission spectrometry (ICP-OES), which reported an average concentration of 13.5 mg of silver per kg of functionalized fabric. SEM showed that silver nanoparticles present a uniform distribution on the surface of the functionalized cotton fabric fibers. On the other hand, by infrared spectroscopy, it was observed that the functionalized fabric variation (compared to control) had a displaced peak of intensity at 1594.32 cm⁻¹, corresponding to carboxylate anions. Similarly, Raman spectroscopy showed an intense peak at 1592.84 cm⁻¹, which corresponds to the primary amino group of carboxymethyl chitosan, and a peak at 1371.5 cm⁻¹ corresponding to the carboxylic anions. Finally, the physical and mechanical tests of tensile strength and color index of the functional fabric reported that it was no different ($p > 0.05$) than the control fabric. Our results demonstrate that we have obtained an improved functionalized cotton fabric using green chemistry that does not alter intrinsic properties of the fabric and has the potential to be utilized in the manufacturing of hospital garments.

Keywords: nanocomposite; functionalized fabric; nanocomposite; functionalized cotton fabric; tensile strength; color index

1. Introduction

Cotton is the natural fiber most widely used in the textile industry due to its softness [1,2], low price [3,4], easy production [5], and it is particularly appropriate for manufacturing medical garments [6,7], health care [8], and hygiene products [9,10]. However, cotton fiber's high hygroscopic property [11,12], affinity to oxygen [13,14], and high specific surface area [15] make it more suitable for microbial contamination turning it into an appropriate medium for bacterial and mold growth [16]. Cotton fibers with antimicrobial properties have become highly desirable due to their potential application in health and medicine-related fields [17–30]. The use of biocides such as triclosan [31], quaternary ammonium compounds [32], or organosilicons [33,34] has been reported in combination with cotton. However, these antimicrobial agents have been reported to produce highly toxic or undesirable byproducts [35,36]. In recent years, the use of nanocarrier systems has been widely used in various fields including nutraceuticals [37–47], pharmaceuticals [48–51], which could affect disposition [52–76], lymphatic transport [77], ophthalmic drug delivery [78], and toxicity [79–83]. The incorporation of silver nanoparticles in textile products has been widely used due to their broad-spectrum antibacterial activity and low toxicity towards mammalian cells [84–93]. The antimicrobial properties of silver nanoparticles are size-dependent because silver nanoparticles of different sizes have different surface/volume ratios, producing different antibacterial efficiency during their interaction with microorganisms [94–99]. The instability of silver nanoparticles has been reported because a variation in the size of the nanoparticles when they are applied directly on textiles can cause agglomeration of the nanoparticles, resulting in a decrease in the antimicrobial effect [94,96,97,99–103].

It has been described that the use of stabilizing agents, such as natural polymers to form nanocomposites, significantly improves the stability of the nanoparticles [104,105]. Polymer-based inorganic nanocomposites combine organic, inorganic, and nanomaterial compounds' unique mechanical, optical, and electrical properties. The polymer chains of these nanocomposites can contain reactive groups and, in combination with the inorganic antimicrobial agents, have exceptional advantages such as exhibiting synergistic antimicrobial effects, improving the adhesion to the substrates, avoiding agglomeration, and improving the stability of silver nanoparticles inside the polymer matrix [15,106]. There is only one study that used carboxymethyl chitosan to pad cotton fabric and then soak it in silver nitrate and black rice extract [107]. However, carboxymethyl cellulose has been preferably used as a reductant agent [108]. Other studies report the use of various reductant and stabilizing agents for silver nanoparticles such as acacia gum [109], a bionic mussel-like material named polydopamine (PDA) [90], ethanolamine [110] and carrageenan [111]. Furthermore, recently a tri-component nanoparticle of silver, copper, and zinc oxide was developed using polymethylol compound (PMC) or functionalized polyethyleneimine (FPEI) polymers as both reductant and stabilizing agents [112]. Other approaches have been implemented to improve properties of nanocomposites such as enhancing thermoelectric performance by realigning Fermi level [113], development of polyaniline derivatives towards multi-stimuli responsiveness by plasma activation [114], plasma treatment toward electrically conductive and superhydrophobic cotton fibers using polypyrrole [115], self-cleanable cotton fibers using silver carbamate and plasma activation [18], durable and high-efficient photocatalyst using the diatom template for energy production and environmental remediation of antibiotics [116,117], and the sono-Fenton (SF) process to prepare nanoporous silica through cleaning diatom frustules while preserving their structural features [118].

The novelty of our manuscript is centered around the assessment of the textile intrinsic properties such as tensile strength, texture, and whiteness index of the nanocomposite previously synthesized and characterized by our research group based on silver nanoparticles and carboxymethyl chitosan (AgNPs-CMC) [119]. The nanocomposite obtained from the complex $[\text{Ag}(\text{NH}_3)_2]^+$ was synthesized under the same conditions as AgNO_3 but at a basic pH. UV-vis spectrophotometry verified the plasmon formation of silver nanoparticles at 410 nm for both silver sources [119]. Our results by dynamic light dispersion (DLS) for AgNO_3 , showed a monodisperse distribution of the nanocomposite with an average hydrodynamic size of 166.7 nm [119]. Infrared spectroscopy measurements with Fourier transform (FT-IR) showed the inhibition of the spectral bands at 879 and 723 cm^{-1} indicating the presence of AgNPs in the nanocomposite AgNPs-CMC [119]. The results of scanning electron microscopy (SEM-STEM) showed that the silver nanoparticles in the nanocomposite were spherical in shape and of a size of 5 to 20 nm [119]. Our research group has recently reported the antimicrobial and antifungal properties of the functionalized fabric with nanocomposite AgNPs-CMC against *Escherichia coli*, *Staphylococcus aureus*, *Candida albicans*, and *Aspergillus niger* [120]. The aim of the current study is to continue the assessment of our developed nanocomposite via green chemistry to functionalize a cotton textile with antimicrobial properties, and to evaluate the textile intrinsic properties such as tensile strength, texture, and whiteness index.

The materials and methods are presented in Section 2. Section 3 provides the outcomes and discussion. Conclusions are described in Section 4.

2. Materials and Methods

2.1. Reagents and Materials

The following reagents were used: 100% mercerized ready-to-dye cotton fabric (purchased in a local market), silver nitrate (Merck Millipore, Burlington, MA, USA). Carboxymethyl chitosan (deacetylation degree: 90% was synthesized from chitosan by our research group at the Laboratory of Preparation, Characterization and Identification (LAPCI_NANO) of the Universidad Nacional de San Agustín de Arequipa (UNSA)).

2.2. Equipment

UV-visible spectrophotometer (Thermo Scientific, Waltham, MA, USA, model evolution 201), inductively coupled plasma optical emission spectrometry (ICP-OES) (Perkin Elmer, Waltham, MA, USA, model Optima 8300), scanning electron microscope (SEM) (Hitachi, Santa Clara, CA, USA, model FESEM SU8200), Fourier-transform infrared (FTIR) spectrometer (Thermo Scientific, Waltham, MA, USA, model Nicolet iS50), Spectrometer Raman (WiTec, Knoxville, TN, USA, model ALPHA300 R), Spectrophotometer (DATA-COLOR, Lawrence, NJ, USA, model SF600 Plus), Universal Dynamometer (model M250 3kN) with a constant rate of extension (CRE), Ecodyer (model Rapid Eco-24).

2.3. Green Synthesis of the Nanocomposite, Preparation and Functionalization of Cotton Fabric

The nanocomposite synthesis was performed using 20 mL of silver nitrate solution (1 mM) with the dropwise addition of 30 mL O-CMC (0.025%) with constant stirring (700 rpm) for 30 min at 90 °C. The synthesized nanocomposite was characterized using SEM/STEM and UV/visible spectroscopy. The fabric was washed with a non-ionic detergent (2.0 g/L concentration) at 90 °C with constant stirring (30 rpm) for 15 min. Then, it was rinsed twice with distilled water at 60 °C with constant stirring (30 rpm) for 10 min in an Eco Dyer. One portion was kept at this point as control fabric, and the other portion was ready for functionalization. The fabric was dried at room temperature for 24 h. The fabric was functionalized using the exhaustion method [121] in an Eco Dyer. For this, one gram of fabric was submerged in 20 mL of an AgNPs-CMC nanocomposite solution under the following conditions: 90 °C temperature, constant stirring (30 rpm), liquor ratio of 1:20, and for 15 min. Then, the fabric was rinsed twice with distilled water at 30 °C temperature,

constant stirring (30 rpm), liquor ratio of 1:20 for 15 min using the Eco Dyer. Finally, the fabric was dried at 80 °C for 15 min.

2.4. Characterization of the Functionalized Fabric

Functionalized fabric crystallinity was evaluated from X-ray diffraction (XRD) patterns obtained using a Miniflex 600 diffractometer (Rigaku, Tokyo, Japan) with a Cu-K α radiation, generated at 40 kV and an incident current of 15 mA. The (2 θ) angular region from 3° to 90° was scanned by steps of 0.05° using a step time of 10 s. The amount of silver in the functionalized fabric was quantified by inductively coupled plasma optical emission spectrometry (ICP-OES) using Fourier-transform infrared spectroscopy (FTIR) (Thermo Scientific, Waltham, MA, USA, model Nicolet iS50c) equipped with the diamond crystal. At room temperature, the measurements were performed in the attenuated total reflectance (ATR) mode. The functionalized fabric samples were pressed against the diamond crystal, and data were collected in the 4000 to 600 cm⁻¹ spectrum range. The Raman spectrum was obtained in an alpha300 R-confocal Raman imaging (WITec, Knoxville, TN, USA, model ALPHA300 R). A 532 nm laser was used as a monochromatic light source. All the spectra were corrected and normalized to a 1099 cm⁻¹ band. The morphology of the cotton fabric surface was observed using a STEM (Hitachi, Santa Clara, CA, USA, model FESEM SU8200) with an acceleration voltage of 3 KV, work distance of 8.5 mm, and current of 2.3 μ A. The fabric was placed on a carbon disc, and no coating was added for measurement.

2.5. Physical and Mechanical Evaluation of the Functionalized Fabric

The verification of changes in the physical and mechanical properties before and after functionalization was performed using quality control techniques, such as tensile strength, whiteness index based on the American Society for Testing and Materials (ASTM) D5034 procedure [122] and the American Association of Textile Chemists and Colorists (AATCC) Test Method 110 [123], respectively. The formulas for the calculation of whiteness are based on the International Commission on Illumination (CIE) [123].

2.6. Statistical Analysis

For the tensile strength and whiteness index tests, the Student's t statistical analysis was performed, these tests were performed in triplicate, and the reported variability corresponds to the standard deviation; the analysis was performed using GraphPad Prism Software (GraphPad Software, LLC, San Diego, CA, USA, version 8).

3. Results and Discussion

3.1. Nanocomposite Synthesis

Figure 1a shows the UV-vis absorption spectra of the nanocomposite with its peak absorption at 425 nm corresponding to the silver nanoparticles' surface plasmon resonance, which confirms the synthesis of the nanoparticles using carboxymethyl chitosan (CMC) as a reducing agent. It has been reported that nanoparticles with maximum absorption at 410–450 nm are spherical [124]. Furthermore, it has been reported that the absence of broad peaks or secondary peaks in the UV-vis absorption spectra correlated with the absence of dispersed nanoparticles, resulting in stable nanoparticles [125,126]. Figure 1b shows the SEM/STEM image of the AgNPs-CMC nanocomposite exhibiting the spherical nature of the silver nanoparticles uniformly distributed within the CMC polymeric matrix, with an average size between 5 and 20 nm. In the nanocomposite synthesis process, CMC was utilized as a reducing and stabilizing agent for the silver nanoparticles, which caused CMC to surround the surface of the nanoparticles and avoid its aggregation. Other research groups have utilized other reducing agents to synthesize AgNPs-CMC nanocomposites. For instance, Xu et al. [127] used sodium borohydride as a reducing agent at high concentrations (940 mM), which is harmful to the environment and poses different human health hazards [128]. The same research group [129] also used high silver nitrate (AgNO₃) concentrations at 470 mM and CMC at 1%. Our research group used green chemistry

synthesis to produce AgNPs-CMC with CMC at 0.025% as a reducing and stabilizer agent without using sodium borohydride, making our method eco-friendly. Similarly, we utilized less silver nitrate and CMC than those used by other research groups [129], indicating that our method is more efficient and cost-effective.

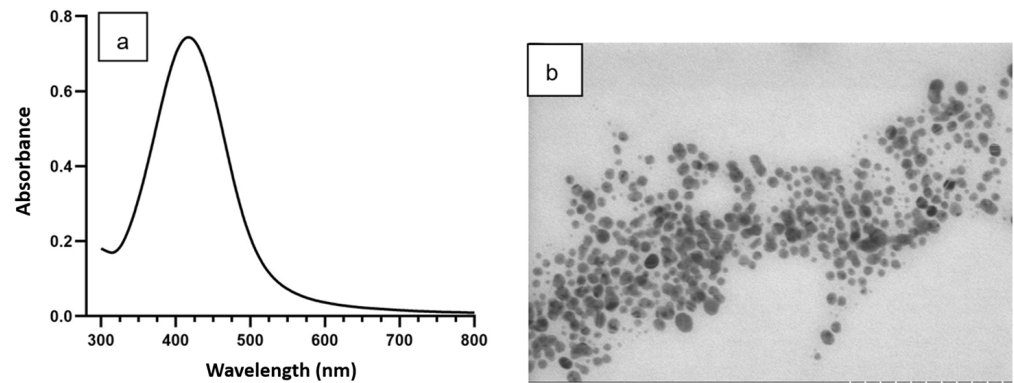


Figure 1. UV-visible spectrum (a) and SEM/STEM) image (b) of the AgNPs-CMC nanocomposite.

3.2. Physical Aspect of the Functionalized Fabric

Figure 2 shows the color of the control fabric (Figure 2a) and the functionalized fabric (Figure 2b). It can be observed that the functionalized fabric compared to the control has developed a light cream coloration; however, this difference is not statistically significant ($p > 0.05$) based on the whiteness index as previously assessed [130]. Other research groups functionalized cotton fabric with AgNPs and CMC but produced a significant change in color from white to beige and brown [131]. A plausible explanation for this difference in coloration change could be attributed because the other research groups utilized up to 2000 ppm silver nitrate concentrations, while in our study, we utilized 169.88 ppm (equivalent to 1 mM) of silver nitrate. Image 2 is a photographic image.

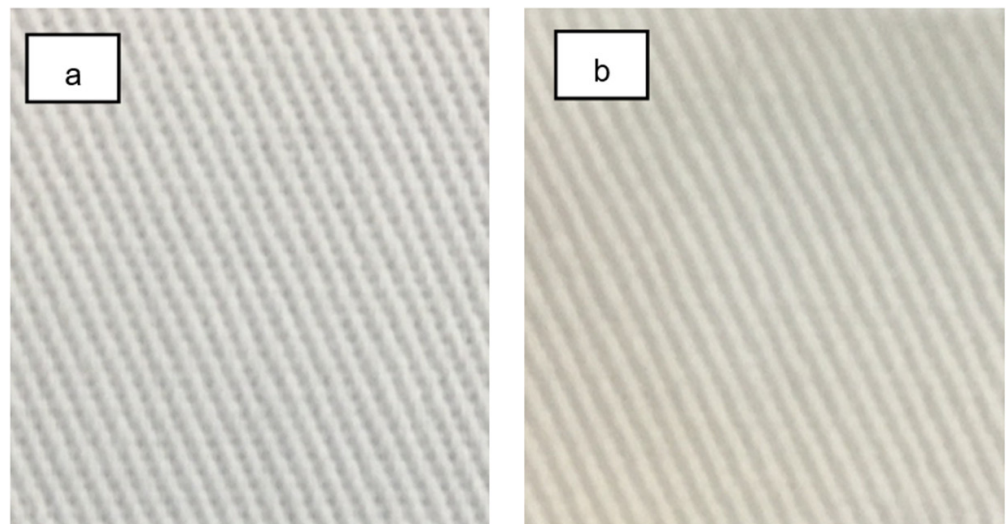


Figure 2. Image of the control (a) and functionalized (b) fabric with AgNPs-CMC nanocomposite.

3.3. X-ray Diffraction (XRD) Analysis

The phase and the purity of the control and functionalized fabric were confirmed from XRD analysis (Figure 3). The diffractograms show the typical peaks of cellulose at 15° , 16.8° , and 22.7° , which correspond to the (101), (101), and (002) crystal planes of cellulose (I) in cotton fabric, as previously reported [132,133]. The functionalized fabric contains three peaks at 38.3° , 44.1° , and 64.6° corresponding to (111), (200), and (220) planes of silver nanoparticles, as previously reported [133].

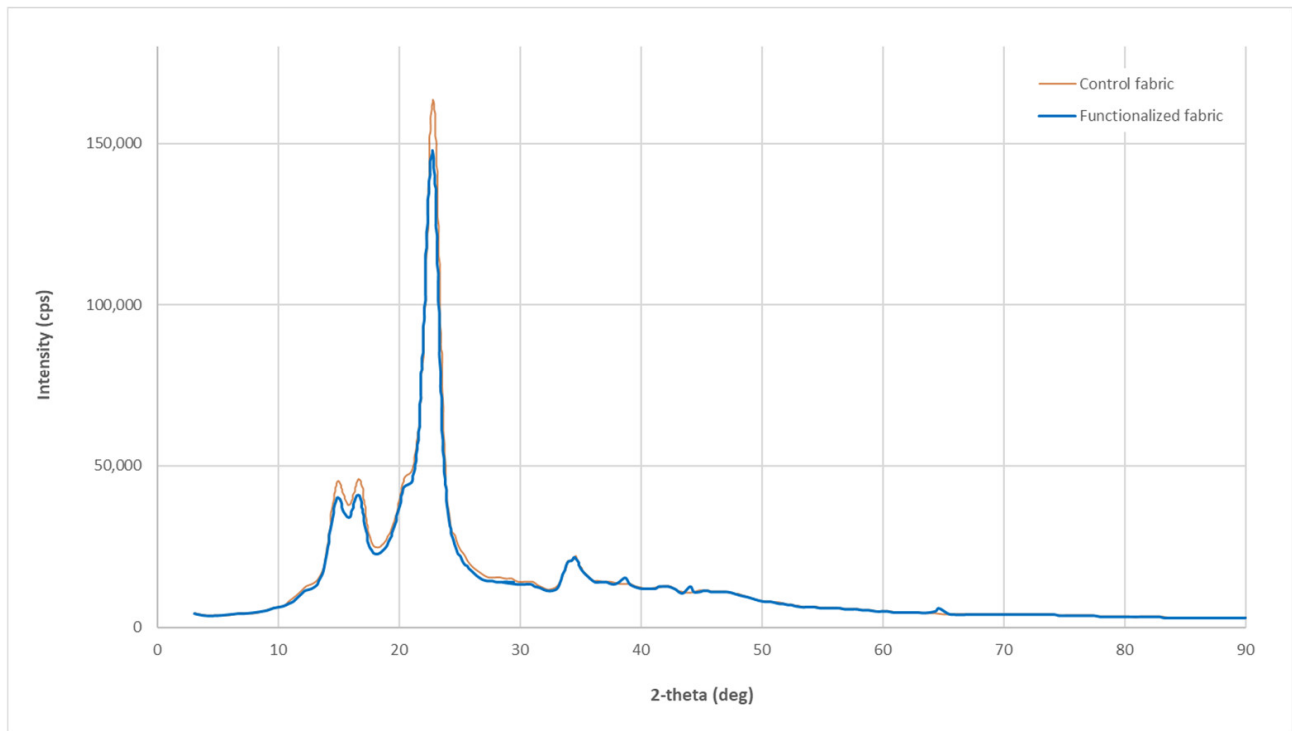


Figure 3. XRD pattern of control fabric and functionalized fabric with AgNPs-CMC nanocomposite.

3.4. Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)

Table 1 shows the concentration of silver in the control and functionalized fabric with AgNPs-CMC nanocomposite. It can be observed that the concentration of silver found in the control fabric was lower than the detection limit (0.2 mg/kg) of the utilized equipment, while the concentration in the functionalized fabric was on average 13.5 mg of silver per kilogram of fabric.

Table 1. Silver concentration (mg/kg) in cotton fabrics. Results showed as Mean \pm Standard Deviation (n = 3). LOQ = 0.2 mg/kg.

Fabric	Control	Functionalized
Silver concentration (mg/kg)	LLOQ	13.5 \pm 0.1

LLOQ = Lower than Limit of Quantitation.

3.5. Scanning Electron Microscope (SEM)

Figure 4 shows the surface morphology of the control and functionalized fabric with AgNPs-CMC nanocomposite. It can be observed that the surface of the control fabric is clean and smooth (Figure 4a), while the surface of the functionalized fabric shows roughness and numerous brilliant particles uniformly distributed, indicating the presence of the silver nanoparticles in the fabric. These results show that the cotton fabric was functionalized with nanocomposite composed of silver nanoparticles and coated with carboxymethyl chitosan. Similar results were previously observed in functionalized cotton fabric with nanocomposite composed of silver nanoparticles and coated with alginate [130].

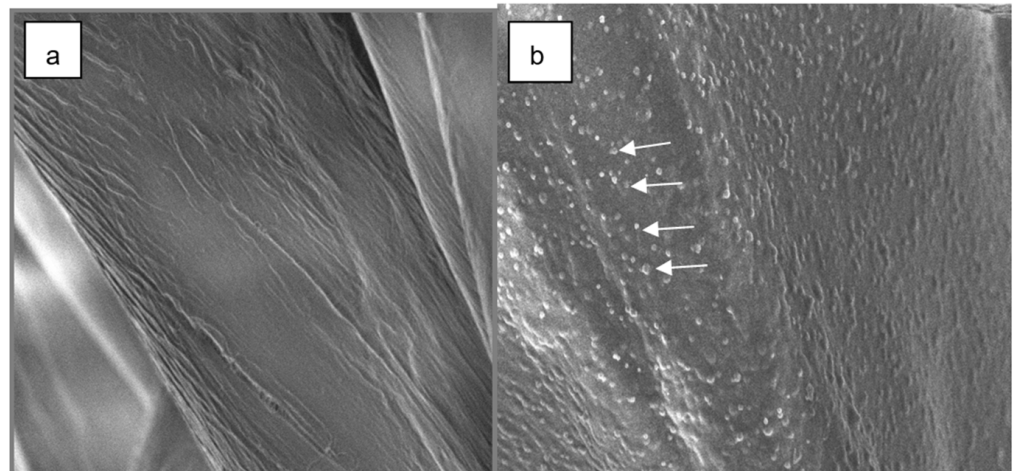


Figure 4. Image under scanning electron microscope (SEM) of the control (a) and functionalized (b) fabric with AgNPs-CMC nanocomposite. The arrows indicate brilliant particles uniformly distributed, representing the silver nanoparticles in the fabric.

3.6. Fourier-Transform Infrared Spectroscopy (FTIR)

Figure 5 shows the FTIR spectrum of the control (Figure 5A) and functionalized (Figure 5B) fabric. The position of the bands in the functionalized fabric shows an apparent change compared to the control. The band at 3328.06 cm^{-1} , corresponding to the hydroxyl group in the control, changed to a lower region at 3324.29 cm^{-1} after the fabric functionalization with the nanocomposite. The previous variation could be by reducing hydrogen bonds in the crystalline structure of the cellulose, which could be interpreted as the polarization of the cellulose OH^- group and the carboxymethyl ($-\text{CH}_2-\text{COOH}$) groups [131]. It was also observed that the position of the band at 1636.18 cm^{-1} in the control fabric got displaced to a lower region at 1594.32 cm^{-1} , indicating the possibility of an asymmetrical stretching of carboxylic (COO^-) anions and the stretching of amino groups, which are capable of attracting silver ions and providing an electron source for the process of reduction [134,135].

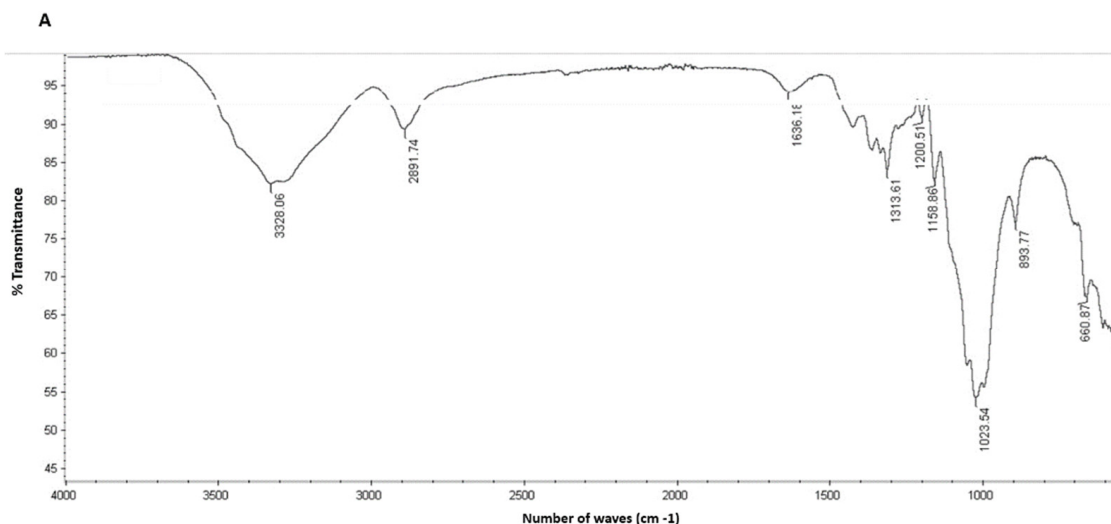


Figure 5. Cont.

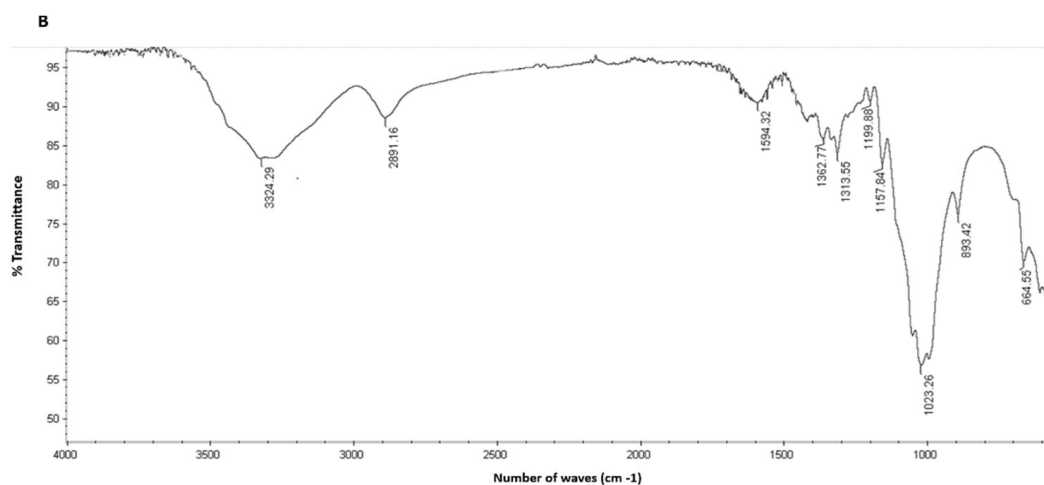


Figure 5. FTIR spectrum of the control (A) and functionalized (B) fabric with AgNPs-CMC nanocomposite.

3.7. Raman Spectroscopy

Figure 6 shows that the functionalized fabric has a very intense peak at 1592.84 cm^{-1} that corresponds to the primary amino group of the carboxymethyl chitosan and a peak at 1371.5 cm^{-1} that corresponds to the carboxylic anions. The latter can interact with the hydroxyl groups in the fabric to form an ester bond in the nanocomposite, which is critical for the functionalization of the fabric with the AgNPs-CMC nanocomposite.

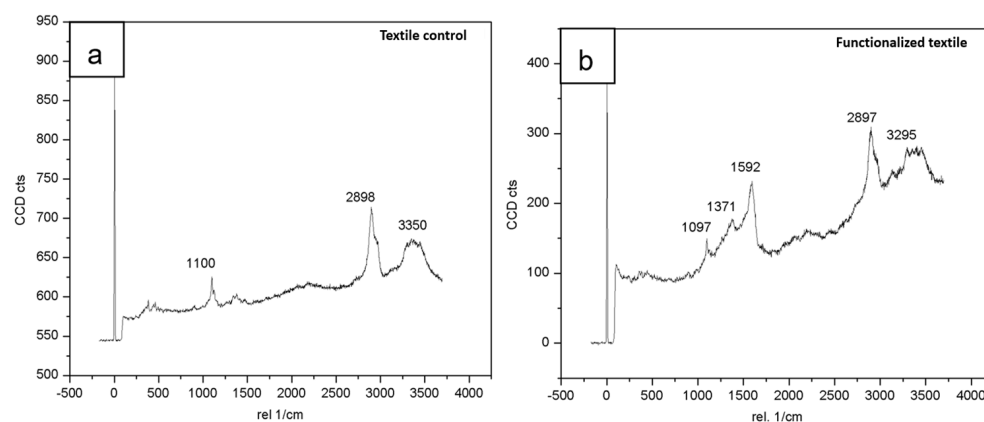


Figure 6. Raman spectrum of the control (a) and functionalized (b) fabric with AgNPs-CMC nanocomposite.

3.8. Physical and Mechanical Characterization

The statistical analysis between control and functionalized fabric reported that the difference between the tensile strength for textile warp and weft is not statistically significant ($p > 0.05$). Figure 7a shows the tensile strength results for textile warp and textile weft. For the textile warp, the control fabric reported 72.400 kgf, while the functionalized fabric was 70.213 kgf. For the textile weft, the control fabric reported 59.250 kgf, while the functionalized fabric was 60.823 kgf. These results correlate to similar cotton fabric functionalized with carboxymethyl chitosan and silver nanoparticles [136]. Figure 7b shows the results for the whiteness index. It can be observed that the control fabric has a 68.92 CIE whiteness index, while the functionalized fabric 64.32 CIE. This difference was not statistically significant ($p > 0.05$). Our results show that the functionalization of the fabric with nanocomposite did not alter the natural textile properties of the fabric, which is a highly desirable characteristic and solves a practical problem with other research groups

where they produced a functionalized fabric with a significant change in color from white to beige and brown color [134].

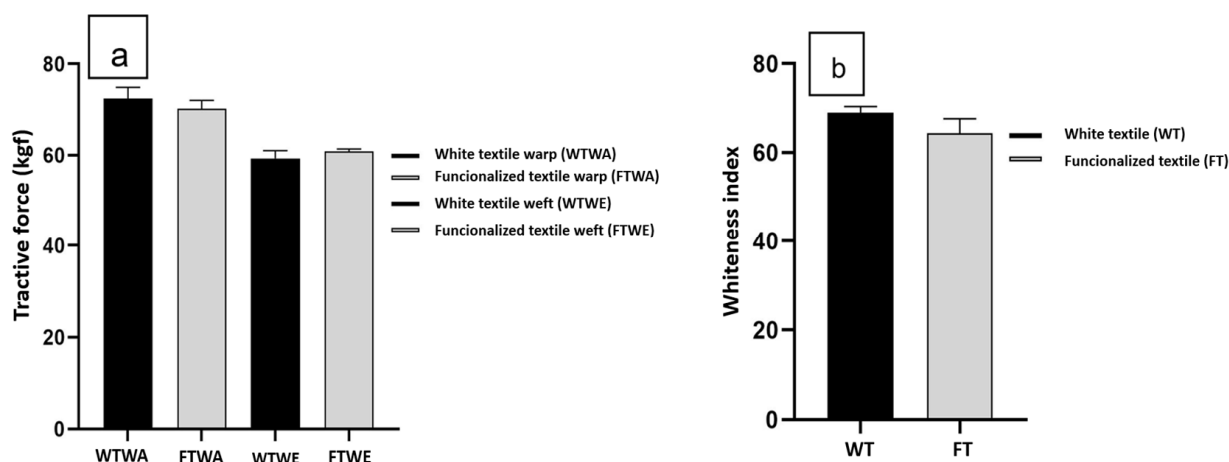


Figure 7. Tensile strength for textile warp and weft (a) and whiteness index (b) results for the control and functionalized fabric with AgNPs-CMC nanocomposite.

4. Conclusions

Cotton is the most widely used natural fiber for textiles, with the recent incorporation of silver nanoparticles due to its broad-spectrum antibacterial activity and low toxicity towards mammalian cells. A functionalized cotton textile was obtained with the synthesized nanocomposite using a green and eco-friendly chemical method, which showed excellent antimicrobial properties without altering the intrinsic properties of the textile, mainly the whiteness index. This functionalized fabric showed that our fabric could be used in garments for hospital use to reduce nosocomial infections, which grants further investigation and assessment of other applications.

Author Contributions: Conceptualization, L.G.Q.-Q., P.L.-R., C.V.-G. and M.M.G.; methodology, L.G.Q.-Q., P.L.-R., C.V.-G. and M.M.G.; software, C.A.A.-C. and C.V.-G.; validation, C.A.A.-C. and C.V.-G.; formal analysis, L.G.Q.-Q., C.A.A.-C., P.L.-R., C.V.-G. and M.M.G.; investigation, L.G.Q.-Q., C.A.A.-C., P.L.-R., C.V.-G. and M.M.G.; resources, L.G.Q.-Q., C.A.A.-C., M.M.G. and C.V.-G.; data curation, L.G.Q.-Q., C.A.A.-C., M.M.G. and C.V.-G.; writing—original draft preparation, L.G.Q.-Q., C.A.A.-C., M.M.G., C.V.-G., C.R.M., A.A.-R., S.D.-A.-A. and J.A.Y.; writing—review and editing, L.G.Q.-Q., C.A.A.-C., M.M.G., C.V.-G., C.R.M., A.A.-R., S.D.-A.-A. and J.A.Y.; visualization, L.G.Q.-Q., C.A.A.-C., M.M.G., C.V.-G., C.R.M., A.A.-R., S.D.-A.-A., and J.A.Y. All authors have read and agreed to the published version of the manuscript.

Funding: We would like to acknowledge the Programa Nacional de Innovación para la Competitividad y Productividad (National Innovation Program for Competitiveness and Productivity) by Innóvate Perú for the nonreimbursable funding granted to the project: “Functionalization of cotton fabrics with nanocomposites NPsAg-carboxymethyl chitosan to improve the antibacterial and antifungal properties with potential application in the manufacturing of garments for hospital use”. AGREEMENT 396-PNICP-PIAP-2014.

Institutional Review Board Statement: Not applicable.

Data Availability Statement: The data presented in this study are available on request from the corresponding author.

Acknowledgments: We would like to thank Clemente Luyo and Jose Carlos Diaz Rosado, Centro para el Desarrollo de Materiales Avanzados y Nanotecnología of Universidad Nacional de Ingenieras (UNI). Similarly, we would like to extend our thanks to Carmen Uribe Valenzuela, Faculty of Textile Chemistry of the Universidad Nacional de Ingenieras (UNI) for providing the cotton fabric.

Conflicts of Interest: The authors declare no conflict of interest.

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