EFFECT OF TEMPERATURE ON THE MODIFICATION OF COTTON FABRIC WITH CROSSLINKED CHITOSAN

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ABSTRACT

This study aimed to compare the properties of the cotton fabric coated with a layer of chitosan crosslinked with citric acid at room temperature or after thermal treatment (at 80°C for 180 min). The applied conditions assume the formation of ionic bonds between the coating components in the first case and their covalent interactions in the second case. Various analyses have been used to confirm this statement. The quantity of the obtained coating on the fabric and the colour characteristics of the materials was characterised with gravimetric and colorimetric analysis. The changes in the bands of functional groups in FT-IR analysis and thermal behaviour of modified fabrics compared to pristine cotton fabric were studied. The surface morphology was investigated using the contact angle measurement. It was found that after thermal treatment, the fabric surface hydrophobicity increases, and the antimicrobial activity against both Gram-negative Pseudomonas aeruginosa and Gram-positive Bacillus cereus strains also increases.

Keywords: cotton fabric, chitosan, surface modification, antibacterial properties, hydrophobicity.

INTRODUCTION

Cotton fabrics are the most common textile materials used for medical and biomedical applications [1]. These fabrics show excellent mechanical properties, large surface area, high porosity and biodegradability. However, some drawbacks are also observed, such as its porous hydrophilic structure that retains water, oxygen, and nutrients, which favour the growth of microorganisms (fungi and bacteria) and significantly limits its use in more advanced medical applications [2]. Microorganism contamination has adverse effects on textile materials by imparting unpleasant odours, mould and mildew strains, discolouration, and loss of functional properties (e.g., tensile strength and elasticity). Microbes can disrupt manufacturing processes such as dyeing, printing and finishing of textile material by reducing viscosity, degradation and mould formation [3].

Biopolymers have been the subject of intensive research due to their numerous advantages, such as biodegradability, biocompatibility, good permeability, widespread distribution in nature, low cost and others [4, 5]. Hydrogels based on natural polymers such as chitosan, alginate, gelatine, etc., are most used in the final processing of textile materials to produce materials with biomedical applications [2].

Chitosan is a deacetylate derivative of chitin, which is the second most abundant polysaccharide on Earth after cellulose [6]. It is one of the main components in the structure of crustaceans such as lobsters, crabs and shrimps; it is also found in protozoa, yeasts, algae, etc. [7]. Due to its specific properties, such as biocompatibility, biodegradability, hydrophobicity, non-toxicity, high biological and antimicrobial activity, economic availability, and high sorption properties, chitosan is used in various biomedical processes [8]. Its functional amino and hydroxyl groups in its structure can enhance its characteristics, which are susceptible to

modification. The polycationic nature it possesses is due to the protonated amino groups (NH_3^+), which results in its solubility in weak acids [9]. When negatively charged residues on the bacterial cell surface interact with positively charged chitosan, cell surface modifications are observed that alter the permeability of the cell wall [10]. However, the small surface area, low porosity and poor mechanical properties of chitosan in the presence of water limit its use in practical applications [11]. It can be used to modify textile materials, which improve the mechanical strength of the former and give the latter new properties. To protect materials from attack by microorganisms, their appropriate modification by chemical or physical treatments can be achieved, resulting in textiles with antimicrobial properties [12]. Since no strong bonds are formed between the cotton fabric and the chitosan, it is necessary to use a crosslinking agent. Glutaraldehyde and genipin are the most used crosslinking agents for chitosan but adversely affect the biocompatibility of the polymer [13, 14]. Citric acid is an organic acid, cheap and readily available, which shows good biocompatibility compared to other crosslinking agents [15, 16]. This acid can form electrostatic bonds between chitosan macromolecules, or covalent bonds in the presence of a catalyst and thermal treatment.

This work aims to investigate the influence of thermal treatment on the modification of cotton material with citric acid crosslinked chitosan, and to examine the effect of this modification on the resulting properties with a view to its application as an antimicrobial textile.

EXPERIMENTAL

Materials

A plain-woven 100 % cotton fabric with a surface weight of 135 ± 5 g m^{-2} was used throughout the work. Chitosan with a molecular weight ranging from 600.000 - 800.000 g mol^{-1} was purchased from Acros Organics (Geel, Belgium). Glacial acetic acid was purchased from Merck (Darmstadt, Germany), and citric acid was purchased from Sigma Aldrich (Darmstadt, Germany).

Preparation of composite materials

Cotton fabrics were soaked in a solution of citric acid (1 % w/w chitosan) and then dried at room temperature. The samples were then soaked in a chitosan solution (2.7 % w/v), which was prepared according to the procedure described previously [17, 18]. The volume of solution to tissue was in a ratio of 2:1. Two composites were obtained and treated as follows: one sample was dried at room temperature, designated as CHRT, and the second sample was heat-treated for 3 h at 80°C, designated as CHHT.

A KERN DBS-BA-def-1714 electronic moisture analyser with an accuracy of 0.01 % at 105°C was used to investigate moisture content. The cotton fabric absorbs moisture, which increases its weight by 7 - 8 %. For this purpose, the moisture content of the starting cotton fabric and the resulting CHRT and CHHT composites were investigated. The pre-prepared samples were weighed, then dried at 105°C in a moisture analyser to constant weight and weighed again. The moisture contents of CHRT and CHHT were 9.55 % and 8.59 %, respectively. The moisture recovery in the CHRT material was higher because the water molecules were retained inside the hydrogel layer formed on the fiber surface.

The chitosan hydrogel textile sample was pre-weighed after the cotton fabric was modified and dried. The samples were immersed in distilled water for 18 h at room temperature to separate unreacted chitosan and citric acid and then dried to constant weight and weighed. The determination of the gel fraction was carried out three times for each composite, and the average of the measurements was recorded. The gel fraction G is calculated according to Eq. (1):

$$G = \frac{w_1}{w} \cdot 100,\% \quad (1)$$

where w_1 is the weight of the textile sample after gel removal by water extraction and w is the weight of the dry sample after modification.

The obtained composites were characterized using gravimetric, infrared (IR), thermogravimetric and colorimetric analysis. The change in weight of the fabric after processing was determined using Eq. (2):

$$Weight\ gain = \frac{w - w_0}{w_0} \cdot 100,\% \quad (2)$$

where w_0 and w represent the weight of the tissue before treatment and after modification and immersion in distilled water and subsequent drying, respectively.

FT-IR analysis was conducted using a Fourier Transform Infrared spectrometer (IRAffinity-1, Shimadzu,

Kyoto, Japan) equipped with a diffuse-reflectance attachment (MIRacle Attenuated Total Reflectance Attachment). Measurements were performed within the spectral range of 600 - 4500 cm^{-1}. Tissue colour characteristics were determined using a Konica Minolta optoelectronic system with a CM-36dG spectrophotometer, D_{65} light source, and D/10° illumination and viewing geometry. The colour coordinates (L*a*b*) and colour difference (ΔE*) were used for characterization. The colour characteristics of the fabrics after their modification were measured. Simultaneous thermogravimetric analysis (TGA) and its first derivative-differential thermogravimetric (DTG) curve were performed on an STA PT1600 TG-DTG/DSC analyser (LINSEIS Messgeräte GmbH, Selb, Germany) with a heating rate of 10°C min^{-1} covering the temperature range from room temperature to 600°C in an air atmosphere. The contact angles of distilled water on pure cotton fabric (CO) and CHRT and CHHT composites were measured with a THETA Flow Auto 1 optical tensiometer and a Basler acA-2500-60 μm digital camera (Biolin Scientific AB, Gothenburg, Sweden). The reported results represent the average of five measurements.

Antibacterial activity of the treated cotton fabrics

The antibacterial activity of the cotton samples was investigated in meat-peptone broth (MPB). Test tubes with sterile MPB and square cotton specimens (10 mm x 10 mm) were inoculated with the cell suspension of each bacterial culture. Tubes with untreated cotton samples and without specimens were also prepared as controls. After 18 h of incubation under shaking, the specimens were removed, and the turbidity of the medium at 600 nm (OD_{600}) was determined. The antimicrobial activity of the treated cotton samples was evaluated by reduction of OD_{600} after incubation compared to the control CO (untreated cotton sample). All assays were performed in triplicate, and the average was taken (standard deviations less than 5 %).

RESULTS AND DISCUSSION

Two composite materials were prepared by coating the surface of cotton fabric with chitosan crosslinked with citric acid at room temperature (CHRT) and with thermal treatment (CHHT). The citric acid solution was applied first, and the fabric was dried at room temperature before the chitosan solution was used. This sequence provides a uniform distribution of the crosslinking agent, facilitating the physical interaction of citric acid with the hydroxyl groups of cellulose fibres through hydrogen bonding. This interplay leaves the carboxyl groups of citric acid free to react with the amino groups of chitosan, thereby firmly fastening the polymer to the fabric surface. Room temperature processing enables ionic crosslinking, as shown in Fig. 1a. In contrast, moderate heat treatment at 80°C for 180 min creates conditions for a low yield amidation reaction between chitosan and citric acid (Fig. 1b), as observed in the studies of Zhuang et al. [19]. In this way, the cellulose fibers are also protected from thermal degradation.

Table 1. presents the results of the fraction of the obtained gel and the weight change of the cotton fabric after modification, calculated using equations (1) and (2), respectively. The data show that with an increase in the processing temperature of the sample, the proportion of gel obtained increases to 96.47 % for CHHT, compared to 92.30 % for CHRT, and the fabric weight rises to 19.27 % for CHHT and to 17.04 % for CHRT.

Colour coordinates L*, a*, b* of the cotton fabric

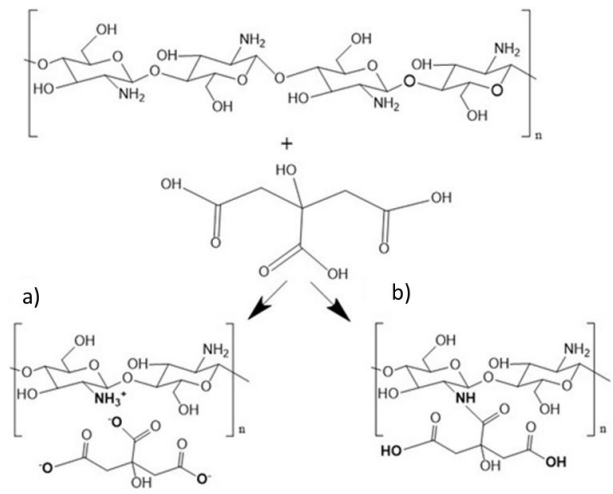

Fig. 1. Interaction of chitosan with the citric acid: (a) by ionic bonds at room temperature until completely drying; (b) by covalent bonds at 80°C for 180 min.

Table 1. Name of the studied samples and their processing.

Sample	Gel fraction, %	Fabric weight Gain, %
CHRT	92.30	17.04
CHHT	96.47	19.27

Table 2. Colour coordinates of the unmodified cotton fabric (CO) and the materials CHRT and CHHT.

Sample	L*	a*	b*	ΔL*	Δa*	Δb*	ΔE*
CO	84.54	-0.09	0.36	-	-	-	-
CHRT	83.90	-1.30	5.73	-0.64	-1.21	5.37	5.54
CHHT	83.36	-1.49	7.02	-1.19	-1.40	6.66	6.90

(CO) and the materials CHRT and CHHT are presented in Table 2. The lightness L* of the fabrics after the coating with chitosan decreases insignificantly, as the values of a* (the red/green) coordinate show an appearance of a weaker green tint. Changes that are more significant are observed in the values of b* (the yellow/blue) coordinate, which is related to the increase of the yellow colour. The colour differences of the two samples compared to the pristine cotton fabric are approximately the same, which means that the heat treatment had a negligible effect on the colour of the sample CHHT.

Fig. 2 shows the FT-IR spectra of chitosan, citric acid (CA) and the materials CHRT and CHHT. In Fig. 2a, chitosan is seen to have a broad band with two maxima at 3358 cm^{-1} and 3300 cm^{-1}, indicating the presence of OH vibrations and NH_2 vibrations of free amino groups in its structure. The two small bands at 2905 cm^{-1} and 2870 cm^{-1} correspond to the symmetric and asymmetric valence vibrations of CH_3 and CH_2. The peak at 1654 cm^{-1} and 1590 cm^{-1} corresponds to the amide-I (C=O stretching) and amide-II (N-H bending of primary -NH_2) vibration bands in chitosan, respectively. The existing peak at 1324 cm^{-1} confirms acetyl groups in the structure of chitosan, while the peak at 1151 cm^{-1} and 1090 cm^{-1} indicates the presence of C-H bending and asymmetric stretching of the C-O-C bridge in its structure, respectively [20]. Fig. 2b presents the FT-IR spectrum of citric acid, which is characterised by the peaks at 3498 cm^{-1} and 3284 cm^{-1} for the O-H stretching vibrations; the peaks at 1741 cm^{-1} and 1702 cm^{-1} characterise the C=O elongation in the carboxyl groups, and the band at 781 cm^{-1} is related to the stretching vibrations of the CH_2 groups [21]. The differences between the spectra of chitosan (Fig. 2b), CHRT (Fig. 2c), and CHHT (Fig. 2d) were observed only in peak intensities. The situation is similar when comparing the spectra of the two composite materials, CHRT and CHHT, with that of the untreated cotton fabric, as shown in Fig. 3.

The similarity in the structure of chitosan and cellulose, combined with the minimal amount of citric acid used to crosslink chitosan, was the reason for

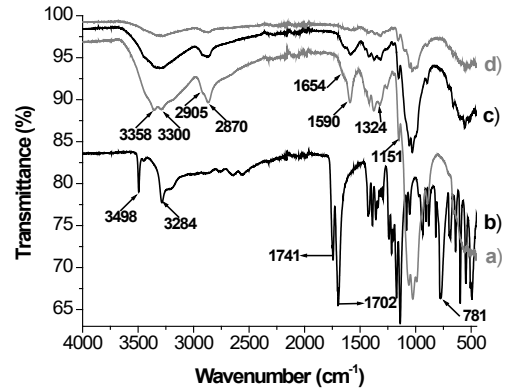

Fig. 2. FT-IR spectra of: (a) chitosan; (b) citric acid and materials; (c) CHRT and (d) CHHT.

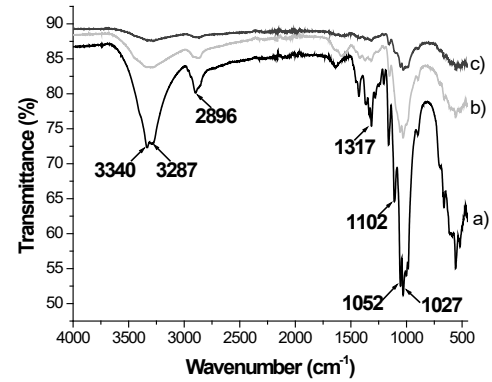

Fig. 3. FT-IR spectra of: (a) untreated cotton fabric; (b) CHRT and (c) CHHT.

subtracting the spectrum of untreated cotton fabric from the spectra of CHRT and CHHT materials, as shown in Fig. 4. In the mid-infrared region from 1200 cm^{-1} to 1800 cm^{-1}, the main differences are observed, which are due to the characteristic bands of the carbonyl (C=O), amino (-NH_2) and ammonium (-NH_3^+) functional groups.

The peak nonappearance at 1700 cm^{-1} in the spectra of CHRT and CHHT indicates the absence of free carboxyl groups from CA. From Fig. 2b, it can be noticed that the peak at 1590 cm^{-1} in the spectrum of chitosan powder shifts to 1580 cm^{-1} in the spectrum of

CHRT as -NH_2 has been protonated to -NH_3^+ (Fig. 4a). The appearance of the shoulder of this peak at 1640 cm^{-1} and the band at 1385 cm^{-1} are characteristic of the carboxylate ion -COO^-.

These peaks that correspond to -NH_3^+ and -COO^- are intense in the spectrum of the CHRT sample (Fig. 4a), because of the formation of an ionic bond between chitosan and citric acid, as shown in Fig. 1a [23]. The intensity of these bands decreases in the spectrum of the CHHT and indicate the weakening of the interaction between the carboxyl group and the ammonium group, since the amino groups are involved in the formation of amide groups with the carboxyl group of citric acid [24 - 26]. This interaction is presented schematically in Fig. 1 b.

The other characteristic bands for the polysaccharide structure appear at 1141 cm^{-1}, due to the C-O-C bond of the glycosidic linkage, and at 1080 cm^{-1} and 1041 cm^{-1} for C-O stretching skeletal vibrations. The vibration in the 893 cm^{-1} region is the C-H deformation vibration in β-sugars.

The thermogravimetric analysis (TGA) and the differential thermogravimetric curve (DTG) are shown in Fig. 5a for cotton fabric, in Fig. 5b for CHRT and Fig. 5c for CHHT. From the obtained results, a three-stage weight loss was observed for all samples. The first weight loss starts at about 109°C and is due to moisture loss (4 % for the CO sample and 6 % for the CHRT and CHHT samples). The most significant thermal degradation occurs in the second stage. For the CO material, this degradation starts at about 271°C (Fig. 5a). For the composite materials CHRT and CHHT, this second stage begins at a lower temperature of 232°C (Fig. 5b and c), due to the presence of acetic and citric acid, which induce thermal degradation of chitosan and cotton. [27]. At this stage, the weight loss is due to processes such as dehydration of the glucoside units, depolymerisation, and degradation [28]. From Fig. 5, it can also be seen that this substantial thermal degradation was completed earliest for the samples of CHHT, CHRT at about 342°C and a higher temperature of 351°C for the untreated cotton fabric. The loss in mass at 370°C was 72.6 % for CO and 63.0 % for CHRT and CHHT. The first derivative also shows that the inflexion point for the starting material CO is at 335°C for CHRT is at 319°C and for CHHT is at 326°C. The slight difference of 9°C between cotton fabric and CHHT may be indicative of stronger interactions between the components obtained because of heat treatment [29]. In contrast, for CHRT, this temperature difference is 16°C. At 600°C, the cotton fabric burns completely without any dry residue, while for the CHRT and CHHT composites, the dry residue for both materials is 14 %. The modification of the cotton fabric with a chitosan cross-linked with citric acid slows down the degradation process and protects the fibres

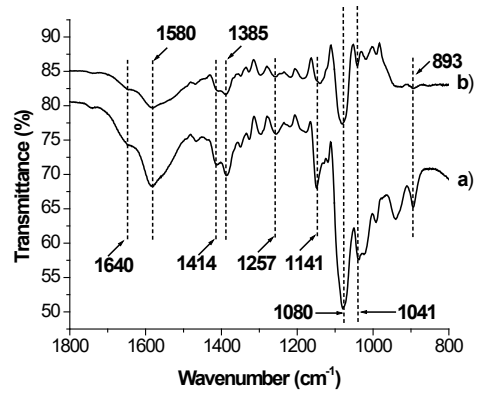

Fig. 4. Substracting FT-IR spectrum of cotton fabric from the spectra of composite materials (a) CHRT and (b) CHHT.

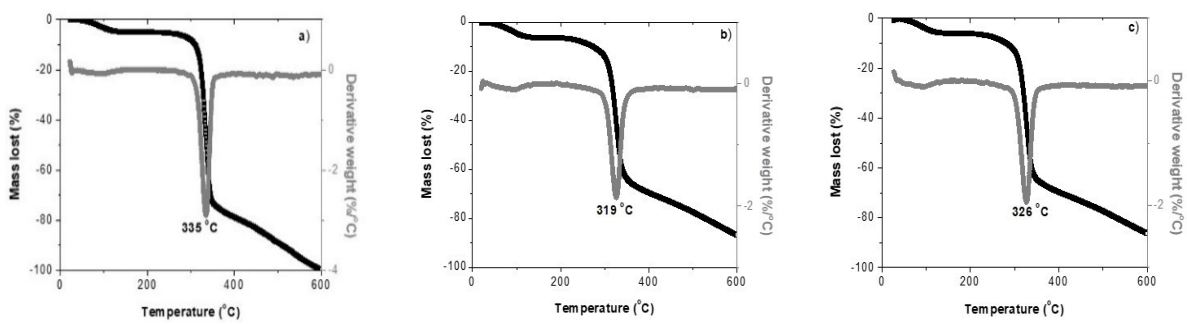

Fig. 5. TG and DTG of (a) cotton fabric and materials (b) CHRT and (c) CHHT.

from further weight loss.

Contact angle is an important metric that provides information on the hydrophilic/hydrophobic property of a material and is critical for future applications. Fig. 6 shows the surface wettability of cotton samples modified with chitosan crosslinked with citric acid obtained at room temperature (CHRT) and high temperature (CHHT). The original cotton fabric has good hydrophilicity as the water droplet immediately soaks into the interior of the material [30]. The large number of hydroxyl groups, pores and capillaries of the yarn are responsible for this [31, 32].

It can be seen in Fig. 6 that once the cotton fabric is coated with chitosan, it is no longer hydrophilic, and the water contact angle increases to 99.03° and 106.76° for the CHRT and CHHT samples, respectively. These results indicate that the composites exhibit a hydrophobic character, with a larger contact angle observed for the CHHT specimen, which may be due to its high-temperature processing that resulted in the chitosan film being homogeneously distributed on the cotton fabric surface, resulting in a decrease in hydrophilic functional groups.

The antimicrobial activity of pristine cotton fabric was used as a control and compared with the ability of CHRT and CHHT fabrics to reduce bacterial growth in MPB. From Fig. 7, both new materials exhibited antimicrobial activity, unlike the control. The CHHT composite material prepared at high temperature has more pronounced hydrophobicity in contrast to the CHRT sample treated at room temperature, suggesting that the layer of chitosan hydrogel formed on the surface of the cotton fabric is responsible for its antibacterial properties. The Gram-negative bacterial strain *P. aeruginosa* is more resistant to the samples than the Gram-positive *B. cereus*, and the CHHT cotton tissue was slightly more active than the CHRT sample due to the sample processing and its hydrophobicity. The method used for assessment of antibacterial activity assumes that the active substance diffuses in liquid media and kills the tested bacteria. In study materials, CHRT and CHHT, the leaching of components with antibacterial properties is prevented by their ionic or covalent interaction. The antimicrobial effect in this study is due to the enhanced hydrophobicity of the surface, which prevents bacteria from depositing on the surface and forming a biofilm, and possibly disrupts their cell membrane [33 - 35].

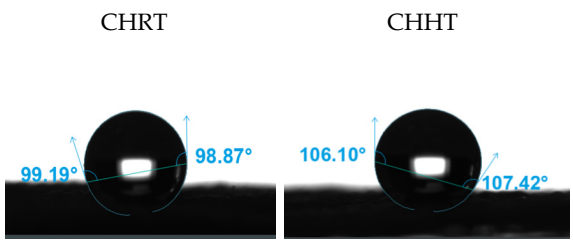

Fig. 6. Measurement of the contact angle of composite materials CHRT and CHHT with distilled water.

CONCLUSIONS

The study finds that the surface modification of cotton fabric with chitosan crosslinked by citric acid enhances the fabric's properties, particularly when thermal treatment is applied. The thermally treated sample (CHHT) exhibited improved gel fraction, greater weight gain, enhanced hydrophobicity, and thermal stability compared to the room temperature-

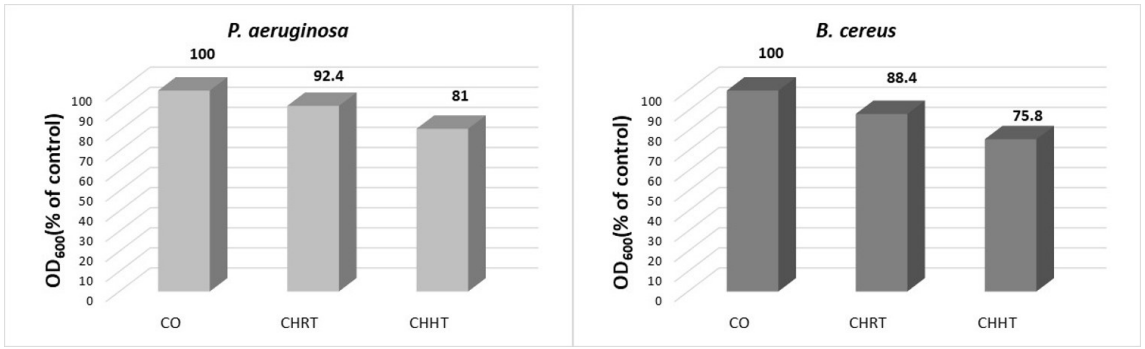

Fig. 7. Growth of model strains *P. aeruginosa* and *B. cereus* in the presence of cotton fabric CO (control) and materials CHRT and CHHT.

treated sample (CHRT). FT-IR analysis showed that in the coating of cotton fabric, interactions between chitosan and citric acid are ionic at room temperature and covalent bonding with heat. Both CHRT and CHHT samples displayed antimicrobial activity, with CHHT being more effective, likely due to its increased hydrophobicity. These results suggest that the proposed treatment method, especially under thermal conditions, is a promising approach for developing functional cotton-based materials for biomedical and hygienic applications.

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Authors' contributions: *D.A.: research execution, data analysis, draft manuscript preparation; D.S.: conceptualization, discussions, proofreading, supervision.*

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