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ARTÍCULO

Functional finishes on textile fabrics: The potential of nanostructures of cellulose and lignin

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Abstract:
Nanostructures have gained increasing attention for their ability to impart novel functionalities to materials, yet their application in sustainable textile finishing remains limited. The textile industry continues to face challenges in developing coatings that combine performance, durability, and environmental responsibility. In this context, renewable nanostructures such as nanocellulose and nanolignin represent promisimg alternatives for integrating bio-based materials into textile coatings while reducing dependence on fully synthetic agents. As a functional finishing approach for textile applications, this study coated cotton fabrics with cellulose and lignin nanostructures. To improve the adhesion of these nanostructures, the exhaust method was employed along with binders during the nanocoating process. Glycerol and silicone elastomer were applied as binding agents under different coating conditions. The morphology, surface wettability, color variation, and mechanical properties of the coated fabrics were evaluated. Nanocoating produced a heterogeneous surface layer, especially with silicone, and adhesion improved when a fixing agent was applied. Both glycerol and silicone proved effective as binders: glycerol enhanced flexibility and tensile strength, while silicone increased adhesion and weight gain. Treatments with nanolignin and silicone produced hydrophobic surfaces, whereas those with nanocellulose and glycerol were predominantly hydrophilic. Nanocellulose treatments showed minimal color variations, while lignin-based coatings resulted in darker tones. These findings demonstrate that wood-derived nanostructures can effectively modify cotton fabrics, combining renewable and synthetic components to create functional and more sustainable textile finishes. Overall, this work represents a relevant step toward integration of bio-based materials in advanced textile surface modification.

Keywords: Adhesion, Colorimetry (CIELab), wettability, contact angle, cellulose nanofibrils, lignin nanoparticles, tensile strength, functional textile coatings

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Introduction

Society and industries are increasingly expressing profound environmental and social apprehensions, necessitating a paradigm shift towards the advancement of renewable materials and sustainable technologies to mitigate adverse impacts. This growing emphasis on eco-consciousness underscores the need to minimize the ecological footprint while developing innovative materials and technologies aligned with sustainable practices. By adopting renewable resources and environmentally friendly processes, industries can contribute to a circular economy, reduce dependence on fossil fuels, and foster innovations that balance economic growth with environmental preservation (Colorado *et al.* 2020).

In this context, the utilization of renewable materials, such as those derived from trees, brings significant technical benefits to the development of new and innovative products.

While the potential of nanocellulose and lignin in textiles has been explored, a specific knowledge gap remains regarding their application as renewable, functional coatings on cotton fabrics. Cotton is one of the most widely used natural fibers due to its comfort, breathability, and versatility; however, its inherent hydrophilicity and surface properties limit functional modifications without suitable coatings. By investigating the effects of nanocellulose and nanolignin coatings on cotton, including their mechanical, optical, and surface characteristics, this study aims to address this gap and evaluate how these renewable nanomaterials can enhance cotton fabric performance.

For instance, cellulose nanostructures have limitless applications as a reinforcing agent, being capable of enhancing the properties of composite materials in several aspects, including thermal stability, mechanical properties, increased biological protection, toughness, barrier properties, and electrical conductivity. This versatility makes them highly valuable across multiple industries. Wood-extracted nanocellulose can be employed for the solidification of aqueous paints and filtration aids, coating papers, and as a vehicle for dyeing paper. It finds use as a thickener in the food industry and as excipients in medicine, serving as binders, fillers, and/or disintegrants in the development of solid pharmaceutical forms. In cosmetics, it can be utilized as an additive in skin cleansing cloths, wound healing dressing material, as part of diapers, sanitary pads, adult diapers, and in polymeric composites (Graeff *et al.* 2012, Kontturi 2018, Tan *et al.* 2019, Nadeem *et al.* 2022, Pradeep *et al.* 2022, Tahir *et al.* 2022, Sofiah *et al.* 2023, Tozluoglu *et al.* 2023).

The production of nanocellulose involves various methods, including mechanical, chemical, and enzymatic processes, each tailored to achieve specific structural and functional properties. Mechanical techniques like high-pressure homogenization and ultrasonication often require high energy input. Alternatively, chemical approaches, like acid hydrolysis, yield highly crystalline cellulose nanocrystals, while enzymatic treatments offer a more sustainable and energy-efficient option. Beyond production, chemical modification of nanocellulose is a critical step for tailoring its properties to meet the demands of complex and innovative applications. Surface functionalization, for instance, enables improved compatibility with polymers or enhance performance in specialized fields such as biomedicine, electronics, and environmental remediation (Fernandes *et al.* 2023, Ji *et al.* 2023, Zhang *et al.* 2023, Pitcher *et al.* 2024).

Emerging technologies further expand the potential applications of nanocellulose, particularly in the development of smart textiles. These materials integrate nanocellulose-based components to enhance conductivity, durability, and responsiveness, paving the way for advanced wearable devices and sustainable solutions in the textile industry (Spagnuolo *et al.* 2022).

Simultaneously, it is worth highlighting lignin nanostructures, another nanomaterial originating from a macromolecular component of wood. Kraft lignin from black liquor is often the main raw material available for commercially relevant applications with lignin nanoparticles since it is a major byproduct of the pulp and paper industries and also of biorefineries. Due to its abundance and potential as a biopolymer for sustainable applications, lignin is becoming increasingly recognized as a sustainable resource to produce nanostructures (Qian *et al.* 2016, Hussin *et al.* 2022).

Lignin is an aromatic polymer with three basic phenylpropane monomers: p-coumaryl alcohol, coniferyl alcohol, and sinapyl alcohol, which together form a highly branched and complex macromolecular structure. The composition of these monomers varies by plant species, resulting in differences in lignin properties among hardwoods, softwoods, and grasses. About 70 million tons of lignin are produced each year, making it one of the most abundant natural polymers on Earth, second only to cellulose, and representing approximately about 30 % of the organic carbon in the biosphere. Lignin, which gives the plant cell wall its rigidity, hydrophobicity, and resistance to microbial degradation, is an essential component due to its high aromatic content and structural complexity (Boerjan *et al.* 2003, Figueiredo *et al.* 2018, Salleh *et al.* 2023).

Lignin nanoparticles can be synthesized using various established methods, each offering unique advantages depending on the intended application. Acid precipitation is one of these techniques that is frequently used due to its simplicity and cost-effectiveness, allowing the efficient recovery of lignin nanoparticles from industrial lignin streams (Rahman *et al.* 2018, Sharma *et al.* 2024).

Solvent exchange techniques (Ma *et al.* 2019, Jiang *et al.* 2021) are particularly effective for producing nanoparticles with controlled morphology and size, offering versatility for tailoring properties for specific uses. In contrast, lignin aggregates can be broken down into uniform nanoparticles by ultrasonication (Gilca *et al.* 2015, Edmundson *et al.* 2024) which improves the particles' stability and dispersion in a range of media. Additionally, it has been shown that combining acid precipitation and ultrasonication optimizes particles size distribution and enhances nanoparticle functionality (Agustin *et al.* 2019, Abate Worku *et al.* 2023).

These various production methods provide lignin nanoparticles with a wide range of industrial uses, such as drug delivery system, hydrogels, UV protection, antibacterial agents, biopharmaceutical carriers, and nanofillers. They are important for the development of hybrid nanocomposites that enhance material performance by having better mechanical and thermal characteristics. Their use as biocides and functional surface coatings underscores their potential in antimicrobial and protective applications. In the medical field, lignin nanoparticles serve as promising carriers for drug delivery, enabling targeted therapy while minimizing side effects. Theis intrinsic UV absorption and antioxidant properties make them valuable as UV absorbents in cosmetic formulations and as additives in packaging materials to prolong shelf life. Furthermore, lignin nanoparticles offer a sustainable solution to turn lignin from a high-volume raw material into high-value products. Their role in advancing eco-friendly technologies not only aligns with global sustainability goals but also highlights their transformative potential in sectors ranging from healthcare to advanced materials (Beisl *et al.* 2017, Tang *et al.* 2020, Yadav *et al.* 2022, Zhu *et al.* 2022, Yu *et al.* 2023).

In the industrial sector, one of the pioneering areas to incorporate nanotechnology concepts into its products was the textile industry, a traditional segment that has undergone intensive innovations in recent decades. The two main areas of nanotechnology application in the textile field are the development of nanofibers and the creation of functional finishes. (Raman *et al.* 2022).

Nanotechnology has revolutionized the textile industry, offering numerous opportunities to enhance fabric properties. Key applications include imparting antibacterial, water-repellent, self-cleaning, and wrinkle-free characteristics to textiles. Nanomaterials can be synthesized through physical, chemical, and biological methods and easily incorporated into fabrics (Sarvalkar *et al.* 2022, Prasad *et al.* 2024).

This technology enables the development of multifunctional textiles with improved durability, UV protection, flame retardancy, and antimicrobial properties. Additionally, nanotechnology facilitates biosensing, drug delivery, and energy generation in textiles. However, potential health and environmental concerns associated with nanotechnology in textiles require further investigation despite challenges, nanotechnology continues to drive innovation in high-performance textiles across various industries, including healthcare, military, sports, and wearable electronics (Syduzzaman *et al.* 2023).

Thus, the objective of this research emerges naturally: to explore the application of functional finishes on cotton fabrics using nanocellulose and nanolignin, assessing their potential to improve surface properties, hydrophobicity, mechanical behavior, and aesthetic characteristics, while contributing to the development of sustainable and high-performance textiles.

Material and methods

Fabrics

The finished cotton fabric (100 %) used in this study was a plain weave with a simple 1/1 weave structure, in its natural unbleached color, representative of the inherent color of cotton fibers, without any dyeing chemicals, acquired from a local market in Curitiba, Brazil. The fabric's grammage is 152,7 g/m², and its thickness was measured at 0,11 mm following ASTM D1777-96 (2019) standard procedures. The moisture content (MC) was found to be 4 %.

Preparation of nanocellulose suspension

The nanocellulose fibrils (NFC) used in this study were purchased by The University of Maine, located in Orono, USA. NFC appeared as a white, odorless aqueous suspension, with a solid content of 3 % by weight and a density of 0,001 kg/cm³ according to the manufacturer. The fibers had nominal width dimensions of 50 nm and lengths of up to several hundred micrometers. The material exhibited hydrophilic surface properties with a surface area of 31 m^2/g to 33 m^2/g (BET).

Preparation of lignin nanostructures

The Klason lignin used in this study was produced from *Eucalyptus* sp. sawdust through acid hydrolysis using 72 % sulfuric acid in a 2:1 ratio (w/v). The pH of the soluble fraction was approximately 6 and it was free of extractives. The nanolignin was produced using a mechanical process in the Microprocessor Super Masscolloider Masuko Sangyo mill with a consistency of 2 % using water, resulting in an aqueous material with a fine texture and brown color. The average calculated concentration was 1,98 %.

Morphology of nanostructures

The morphology of NFC and lignin nanoparticles were investigated using a Transmission Electron Microscopy (TEM) in a JEOL JEM-1200EX II microscope at 120 keV. The nanostructures were previously deposited on a carbon coated copper grid and dried at room temperature.

The NFC exhibited diameters ranging from 0,2 to 0,3 µm and various lengths, forming a network-like structure, as observed in the TEM image (Figure 1).

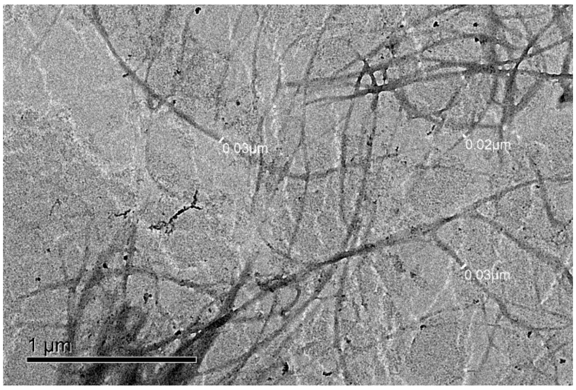


Figure 1: Transmission Electronic Microscopy (TEM) image of NFC.

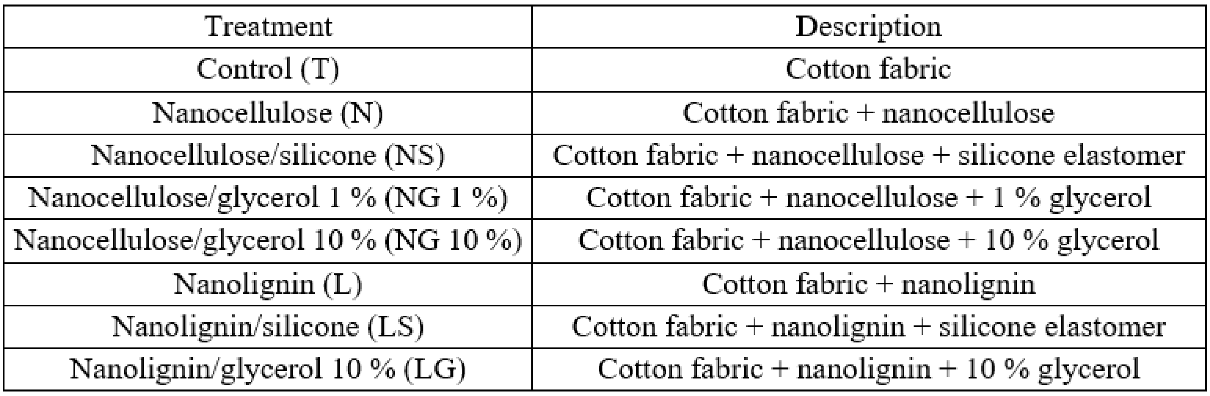
The nanolignin exhibited isolated nanoscale structures, some with diameters of less than 200 nm, and appeared as agglomerates with various shapes. Scanning electron microscopy (SEM) images revealed irregular polymer morphology. The material constituted compact and shapeless aggregates (Lopes *et al.* 2023, Lopes *et al.* 2017).

Preparation of composites for the coating of textile fabrics

The composite coatings were produced from mixtures of NFC, nanolignin, glycerol (0,5 % water content) and silicone elastomer (14,71 % non-volatile content and viscosity of 6769 cst). Glycerol was used as a plasticizer and mixed with NFC to coat the cotton fabric. The silicone elastomer emulsion was used as an auxiliary agent to enhance the adhesion of nanolignin particles to the textile fabric.

The dilutions were prepared following the method described by Nechyporchuk *et al.* (2017), where the initial NFC suspension was diluted to 1 % using deionized water, and the formulation containing glycerol and silicone elastomer was prepared at a 1/10 (v/v) ratio. The NFC and nanolignin formulations were homogenized on a mechanical stirrer for 20 min. After adding the silicone elastomer or glycerol, the solution was further homogenized for another 20 min. Subsequently, cotton fabric samples measuring 50 mm × 150 mm were coated under different conditions (Table 1). The coating process was carried out using the Exhaust method, following the method described by Martins *et al.* (2015). The cotton fabrics were immersed in aqueous dispersions for 20 min with agitation on a shaker (Lab-Line Instruments). After immersion, the cotton fabric samples were oven dried at 103 °C ± 2 °C for 10 min, and then placed in a desiccator. Once dried, the coated samples were weighed using an analytical balance. For the finishing of the coated samples, they were mechanically pressed at a temperature of 120 °C. The weight percent gain (WPG) of the coated cotton fabrics were determined by dry mass ratio.

Table 1: Description of the treatments performed in the cotton fabrics.



Treatment	Description
Control (T)	Cotton fabric
Nanocellulose (N)	Cotton fabric + nanocellulose
Nanocellulose/silicone (NS)	Cotton fabric + nanocellulose + silicone elastomer
Nanocellulose/glycerol 1 % (NG 1 %)	Cotton fabric + nanocellulose + 1 % glycerol
Nanocellulose/glycerol 10 % (NG 10 %)	Cotton fabric + nanocellulose + 10 % glycerol
Nanolignin (L)	Cotton fabric + nanolignin
Nanolignin/silicone (LS)	Cotton fabric + nanolignin + silicone elastomer
Nanolignin/glycerol 10 % (LG)	Cotton fabric + nanolignin + 10 % glycerol

Characterization of the coated cotton fabrics

Morphology and weight percentual gain (WPG)

A visual analysis was done in all evaluated material and weight percentual gain (WPG) in each treatment was calculated based on fabric humidity content and its initial and final weight. Macroscopic characterization of the coated cotton fabrics was conducted using a Zeiss Discovery V.12 stereomicroscope.

Colorimetric characterization

For colorimetric characterization, a Konica Minolta CM-5 spectrophotometer was employed to collect L*, a* and b* color parameters based on the *CIELab** color space. Also, reflectance spectra were obtained in the region from 360 nm to 740 nm. Five samples for each treatment were evaluated. To quantify the total color change, ΔE*, differences between the colorimetric parameters in the *CIELab** color space were calculated using Equation 1. Additionally, the hue angle (h) and chromaticity (C*) were evaluated using Equation 2 and Equation 3, respectively.

$$\Delta E^* = \left(\frac{\Delta L^*2 + \Delta a^*2 + \Delta b^*2}{2} \right) \quad (1)$$

$$h = tan - \frac{1b^*}{a} \quad (2)$$

$$C^* = \left(\frac{a^*2 + b^*2}{2} \right) \quad (3)$$

Where: L* = luminosity; a* = green-red chromatic coordinate; b* = blue-yellow chromatic coordinate.

Wettability

The study of surface wettability of cotton fabrics was conducted using the apparent contact angle (CA) technique on a Krüss DSA25 goniometer (Krüss GmbH, Germany), with the sessile drop method. Deionized water (surface tension of 72,80 mN/m $^{-1}$) with a volume of 5 µL was used to measure the apparent contact angle. Three droplets were deposited on the surface of each cotton fabric sample at different points of the samples. Five samples were used for each treatment. The apparent contact angle (CA) was measured 5 s after the droplet was placed on the surface of the samples, and subsequently, every second until 30 s.

Mechanical analysis

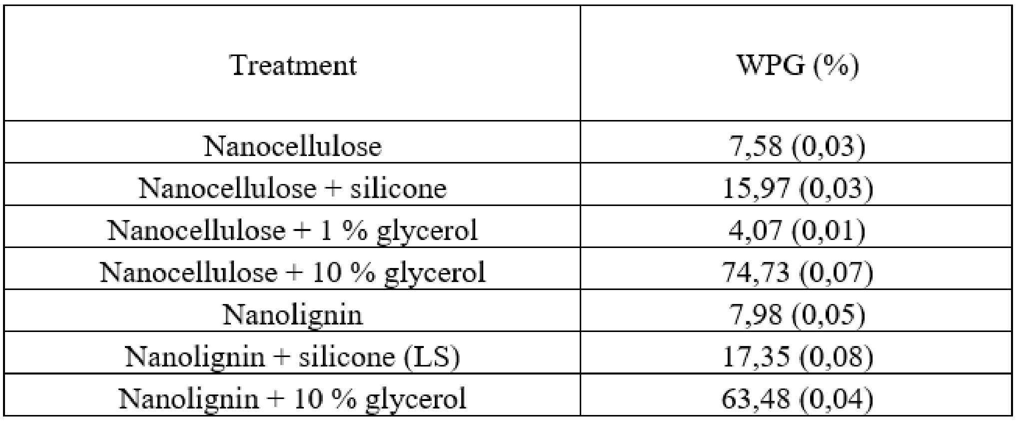
The adapted mechanical analysis was conducted based on ASTM-D5034 (2020) using the EVIC universal testing machine by HSensor. The initial sample length between the grips was 70 mm, with a cross-sectional area of approximately 19 mm^2 and a width of 40 mm. The maximum force applied was 60 N, with a maximum speed of 60 mm/min, leading to rupture. Four repetitions were performed for each sample to ensure the reliability of the results.

Results and discussion

Weight percentual gain

The percentage weight gain (WPG) (Table 2) in the cotton fabric was highest for the treatments involving silicone elastomer and lowest for the 1 % glycerol treatment. In contrast, the 10 % glycerol treatment exhibited notably elevated values, possibly attributed to the oily nature of these samples. The WPG is directly related to the density of binders and impregnated materials within the fabric.

Table 2: Average values of weight percentual gain and for untreated and treated cotton fabrics.



Treatment	WPG (%)
Nanocellulose	7,58 (0,03)
Nanocellulose + silicone	15,97 (0,03)
Nanocellulose + 1 % glycerol	4,07 (0,01)
Nanocellulose + 10 % glycerol	74,73 (0,07)
Nanolignin	7,98 (0,05)
Nanolignin + silicone (LS)	17,35 (0,08)
Nanolignin + 10 % glycerol	63,48 (0,04)

Values between parenthesis corresponding to standard deviation.

Following the exhaustion process, material loss was observed; the samples lost threads at the edges, significantly affecting the WPG determination. In the case of treatments involving nanolignin, the nanostructures impregnated in higher quantities at the edges of the samples, where the threads were cut due to the absence of lubricant residues (such as tallow or mineral oil), which are commonly used in weaving processes (Nechyporchuk *et al.* 2017).

Morphology - Visual aspects

In the context of treatments with nanolignin (Figure 2), achieving uniform dispersion of the solution within the cotton fabric proved to be challenging. Instead, a heterogeneous film appeared on the fabric's surface. This phenomenon can be attributed to the chosen application technique, as well as the specific approach employed for nanolignin preparation and dilution.

It's noteworthy that the treatment with nanolignin + 10 % glycerol exhibited a distinct oily quality. Meanwhile, certain samples treated with nanolignin + silicone displayed marked surface irregularities, characterized by localized patches with distinct features. These observations collectively emphasize the intricate interplay between application methods, formulation of nanolignin solutions, and resulting surface characteristics in treated fabrics.

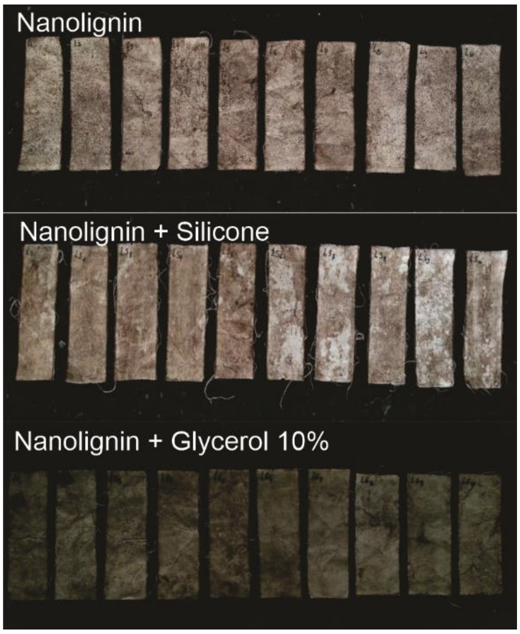


Figure 2: Visual aspects of coated cotton fabrics with nanolignin.

The Nanocellulose and Nanocellulose + silicone treatments also exhibited non-uniform dispersion of the solution, resulting in the formation of a heterogeneous film on the surface of the cotton fabric (Figure 3). This led to variations in material deposition across different areas, primarily attributable to the chosen application technique. The dispersion of the 1 % glycerol treatment was not visually discernible. Notably, the nanocellulose + 10 % glycerol treatment experienced the least material loss (threads) during the exhaustion process. This treatment resulted in a higher level of oiliness than the nanolignin + 10 % glycerol treatment, prompting further exploration of the nanocellulose + 1 % glycerol treatment.

These observations collectively emphasize the complexities associated with solution application techniques, particularly concerning dispersion uniformity, and resulting surface characteristics. While the reduced material loss observed in the nanocellulose + 10 % glycerol treatment suggests its potential for practical applications, the heightened oiliness warrants further investigation, as evident in the nanocellulose + 1 % glycerol treatment.

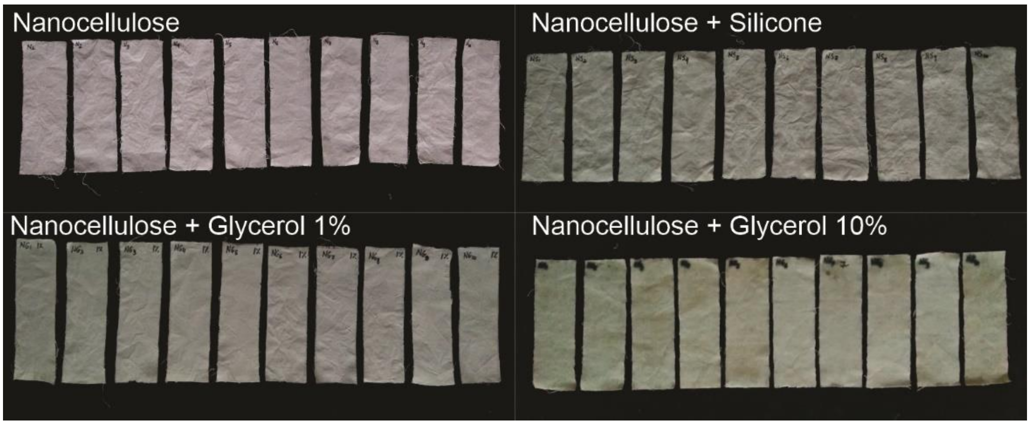


Figure 3: Visual aspects of coated cotton fabrics with nanocellulose.

Morphology - Macroscopic characterization

This analysis was conducted to characterize the structure of the textile samples, serving to illustrate the appearance that the cotton fabric has acquired following each treatment. Through this procedure, it becomes possible to observe the interweaving of threads, the arrangement of fibers within these threads, as well as attributes like pilosity and porosity. These properties are inherently linked to the structural elements of the material (Figure 4).

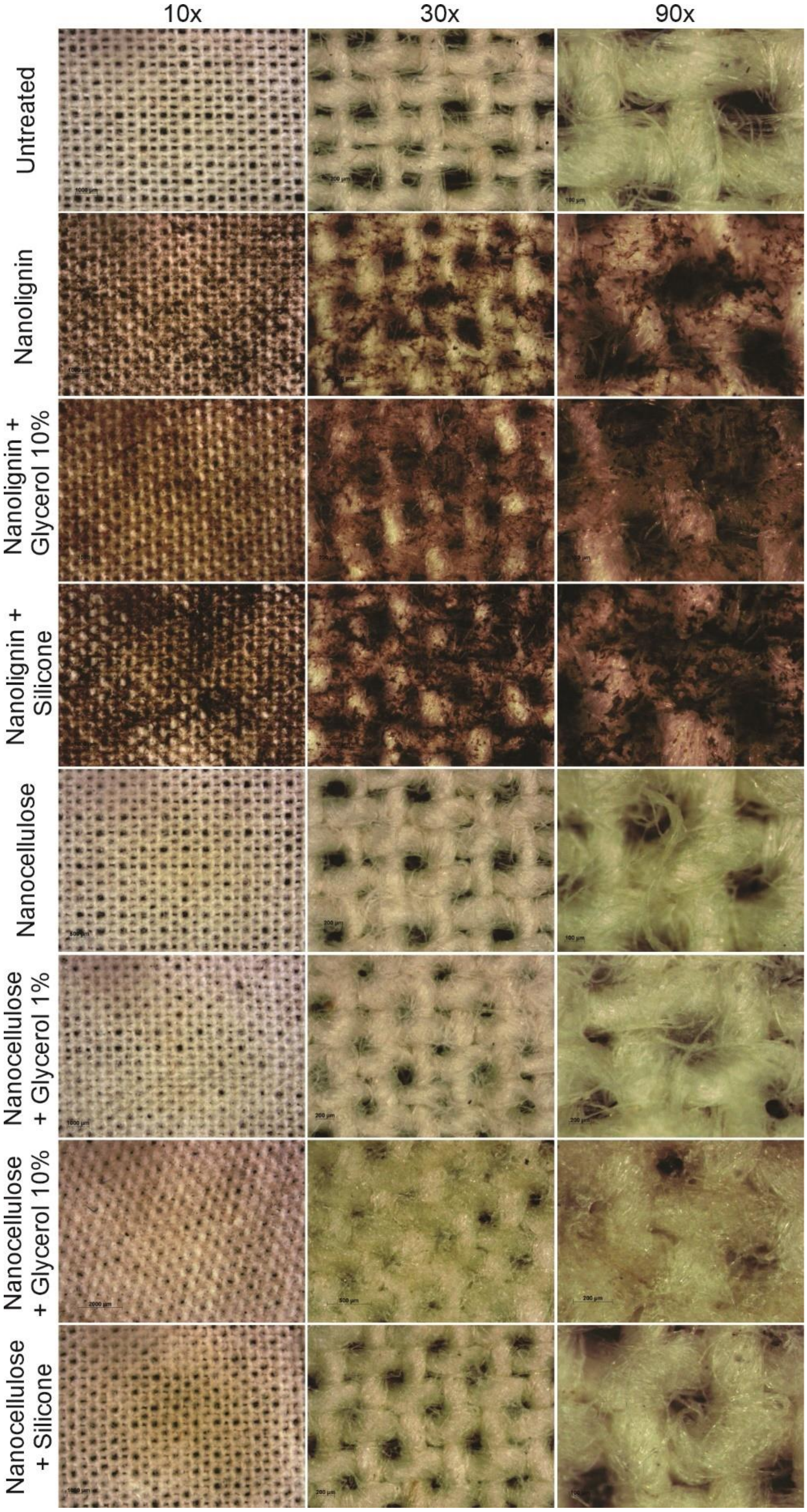


Figure 4: Macroscopic images of cotton fabrics before and after different treatments.

In the untreated cotton fabrics, we observed the simple 1/1 weave pattern, and the finishing of the fibers (tallow or mineral oil) carried out during spinning before weaving, resulting in a glossy appearance. This finishing reduces the fiber's porosity, rendering it less hygroscopic.

For the nanolignin treatments, the amount of product superficially impregnated into the fabric's thread structure is evident. Greater impregnation is visible within the fabric's pores, forming a surface film on the cotton fabric. The lowest impregnation was seen in the nanolignin treatment with a 7,98 % weight gain, while the highest was in the nanolignin + silicone treatment with a 17,35 % gain (Table 2). This can be explained by the preparation of lignin, which was carried out in water. When silicone, a less polar solvent, was added, better dilution occurred, as nonpolar substances tend to dissolve in nonpolar liquids. Upon adding glycerol, considered polar, there was less dilution of the substance, resulting in a lower impregnation of lignin nanostructures, evident in the calculated weight gain (Table 2), which could be linked to the oily outcome.

Comparing the nanocellulose treatment with the untreated cotton fabrics, an increased pilosity is observed – protruding threads on the fabric's surface - forming a surface coating with nanofibrillated cellulose due to its entangled structure and size.

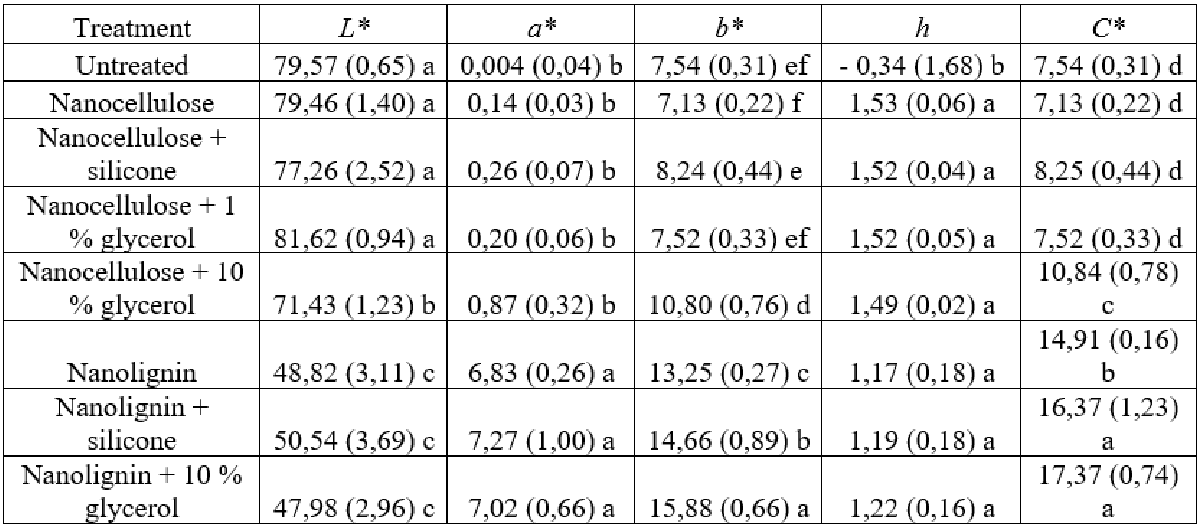
The nanocellulose + 1 % glycerol and nanocellulose + silicone treatments exhibited even greater thread pilosity compared to the Nanocellulose treatment, indicating further entanglement of nanofibrillated cellulose. The 1 % glycerol treatment displayed better coverage of fabric pores with cellulose nanostructures compared to the silicone elastomer treatment. However, it yielded a weight gain value of 4,07 % (Table 2), which can be attributed to the flexibility it imparts. Cellulose nanostructures in the silicone elastomer treatment appear shinier, adhering to the fabric's threads rather than the pores, with a weight gain value of 15,97 %.

The nanocellulose + 10 % glycerol treatment coated almost all fabric pores, forming an oily film on the cotton fabric. As indicated in Table 2, it displayed the highest weight gain value of 74,73 %.

Colorimetric characterization

The colorimetric parameters (Table 3) indicate that treatments with a base deposition of nanolignin exhibit lower luminosity, as expected, since lignin was used in its natural color state without bleaching. Additionally, higher values are observed for the chromatic coordinates a* and b*, indicating a tendency towards reddish-yellowish coloration, respectively.

Table 3: Average value of colorimetric parameters for untreated and treated cotton fabrics.



Treatment	*L**	*a**	*b**	*h*	*C**
Untreated	79,57 (0,65) a	0,004 (0,04) b	7,54 (0,31) ef	- 0,34 (1,68) b	7,54 (0,31) d
Nanocellulose	79,46 (1,40) a	0,14 (0,03) b	7,13 (0,22) f	1,53 (0,06) a	7,13 (0,22) d
Nanocellulose + silicone	77,26 (2,52) a	0,26 (0,07) b	8,24 (0,44) e	1,52 (0,04) a	8,25 (0,44) d
Nanocellulose + 1 % glycerol	81,62 (0,94) a	0,20 (0,06) b	7,52 (0,33) ef	1,52 (0,05) a	7,52 (0,33) d
Nanocellulose + 10 % glycerol	71,43 (1,23) b	0,87 (0,32) b	10,80 (0,76) d	1,49 (0,02) a	10,84 (0,78) c
Nanolignin	48,82 (3,11) c	6,83 (0,26) a	13,25 (0,27) c	1,17 (0,18) a	14,91 (0,16) b
Nanolignin + silicone	50,54 (3,69) c	7,27 (1,00) a	14,66 (0,89) b	1,19 (0,18) a	16,37 (1,23) a
Nanolignin + 10 % glycerol	47,98 (2,96) c	7,02 (0,66) a	15,88 (0,66) a	1,22 (0,16) a	17,37 (0,74) a

Values between parenthesis corresponding to standard deviation. Different letters within the same column mean statistically significant differences.

The hue angles (*h*) (Table 3) for each treatment were low, ranging between - 0,34° and 1,53°. The angle 0° is considered the color red. The results of all hue angles (*h*) displayed reddish hues. Neutral colors have low saturation, while pure colors have high saturation. Here, higher chroma values indicate greater color saturation, with higher chroma values observed for the nanolignin treatments and lower values for the nanocellulose treatments.

The variation of each parameter concerning the untreated cotton fabric, and the total color difference (Table 4) reveals that the treatments with nanolignin exhibited the highest values for total color difference (ΔE^*), with noticeable color changes observed in all treatments. The largest result was observed in the nanolignin + 10 % glycerol treatment. In contrast, the nanocellulose treatments displayed lower values, with the Nanocellulose treatment showing the smallest total color change. The ΔE* values quantify the total color difference but do not provide a qualitative assessment. This implies that relying solely on ΔE* values does not allow us to determine which color axis (brightness, red/green, yellow/blue) experienced the color variation or in which direction it occurred.

Table 4: Color variation (ΔE) of the treated fabrics in relation to untreated one.

Treatment	ΔL^*	Δa^*	Δb^*	ΔE^*
Nanocellulose	- 0,11	0,14	- 0,41	0,45
Nanocellulose + silicone	- 2,31	0,26	0,71	2,43
Nanocellulose + 1 % glycerol	2,05	0,20	- 0,02	2,06
Nanocellulose + 10 % glycerol	- 8,14	0,87	3,26	8,81
Nanolignin	- 30,75	6,83	5,72	32,01
Nanolignin + silicone	- 29,03	7,27	7,12	30,76
Nanolignin + 10 % glycerol	- 31,59	7,01	8,34	33,42

However, it is noteworthy that the treatment with nanocellulose + 1 % glycerol was the only one that exhibited a brighter luminosity, while the other treatments displayed a darker luminosity. Furthermore, all treatments displayed a more reddish hue in terms of Δa^*. Regarding Δb^*, the Nanocellulose and nanocellulose + 1 % glycerol treatments showed bluish tones, whereas the other treatments exhibited more yellowish tones. These findings underscore the complexity of color variations, highlighting the importance of considering multiple color parameters beyond ΔE^* to understand the specific color shifts and their directions.

Then, to assess the behavior of all treatments regarding light incidence, spectral curves were evaluated within the wavelength range of 360 nm to 740 nm (Figure 5).

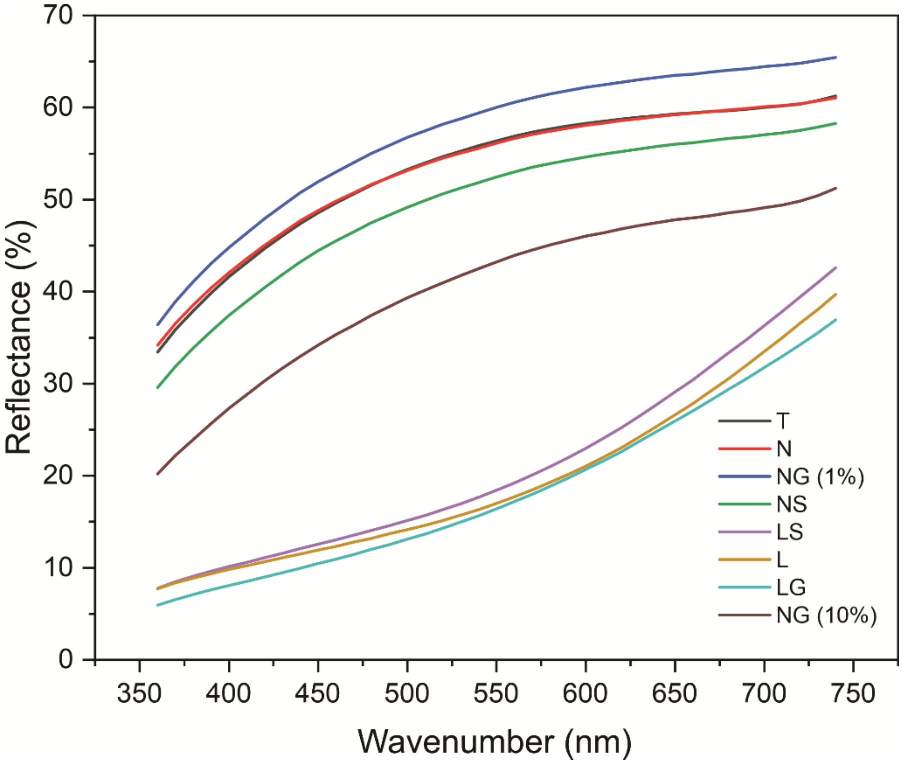


Figure 5: Color reflectance curves of the untreated and treated cotton fabrics.
T = untreated fabric; N = nanocellulose; NG (1 %) = nanocellulose + 1 % glycerol; NS = nanocellulose + silicone; L = nanolignin; LS = nanolignin + silicone; LG = nanolignin + 1 % glycerol; NG = nanolignin + 10 % glycerol.

By analyzing the spectrum (360 nm to 740 nm), it was observed that the untreated and nanocellulose treatments exhibited a similar behavior, as their curves are overlapping. When a ray of light strikes a textile fabric, a portion of this radiation can be absorbed by the sample, another part is reflected, and the remainder is transmitted. Several factors come into play in this interaction, including the type of fiber, thickness and structure of the fabric, porosity, and other factors (Alam *et al.* 2021).

Nanocellulose-treated textiles exhibited higher reflectance. In the context of UV radiation protection, textile fabrics typically offer varying degrees of protection, depending on factors such as fiber type, fabric structure, color, density, weight, among others. The determination of the ultraviolet protection factor (UPF) of a fabric occurs within the 280 nm to 400 nm range. Upon examining the spectra, the samples treated with nanocellulose show higher percentages of reflection. In general, untreated cotton fibers absorb small amounts of UV radiation (Grifoni *et al.* 2009, Bashari *et al.* 2018).

Wettability

Apparent contact angle (WCA) for all treatments varies according to the coating composition. The untreated cotton fabric exhibited values between 110,98° and 112,48°, while the nanolignin-treated samples ranged from 112,97° to 117,05°. The highest contact angle values were obtained for the nanolignin + silicone treatment (117,88° to 119,90°), confirming the formation of a hydrophobic surface layer. In contrast, the nanocellulose + silicone treatment showed WCA values between 87,83° and 95,45°, indicating a less hydrophobic and more moderately wettable surface (Figure 6).

Importantly, the contact angle values for the nanolignin + silicone samples remained stable over time, reinforcing their hydrophobic character. This hydrophobic behavior is consistent with the higher weight gain measured for the nanolignin + silicone samples (17,35 %), suggesting that a more continuous and compact coating film was formed.

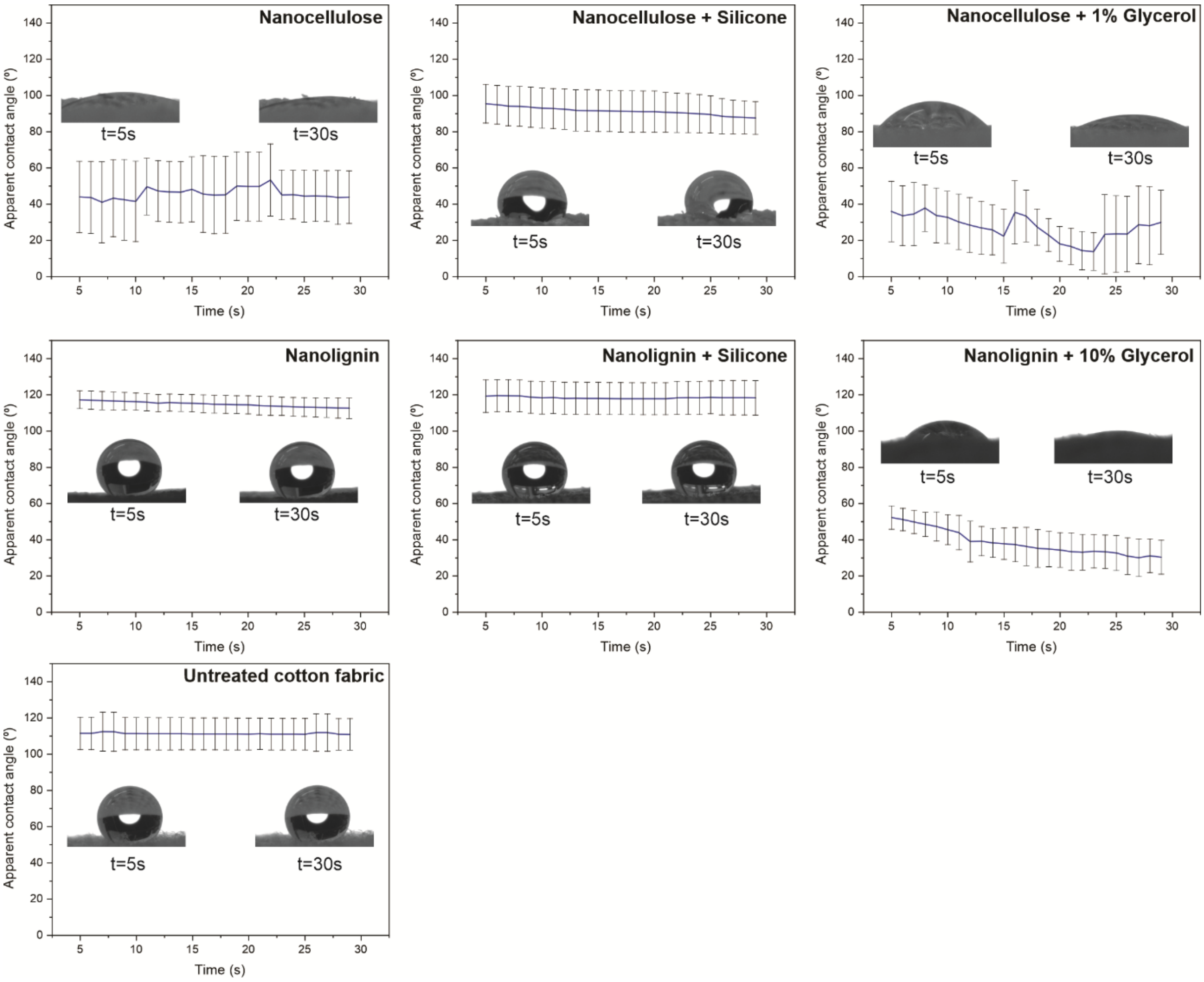


Figure 6: Time-dependent contact angle of untreated and treated cotton fabrics and visual illustration of the droplets at 5 s and 30 s.

The high contact angle value for the untreated cotton fabric, without the coating of nanostructures and fixatives/plasticizers, can be attributed to the presence of residues from lubricants (such as tallow or mineral oil) commonly used in weaving processes. These residues impart hydrophobic properties to the cellulose (Nechyporchuk *et al.* 2017).

On the other hand, the WCA contact angle of the nanolignin + 10 % glycerol, Nanocellulose and nanocellulose + 1 % glycerol treatments decreased compared to the untreated cotton fabric. These treatments exhibited WCA values ranging from 30,10° to 52,33°, 41,06° to 52,33° and 14,38° to 35,98° respectively. These values indicate instability over time, resulting in a hydrophilic surface. As expected, the contact angle in these treatments was reduced compared to the untreated cotton fabric, indicating faster absorption rates, since nanocellulose and glycerol are hydrophilic materials. Nanocellulose is naturally hydrophilic due to the abundance of hydroxyl (-OH) groups on its surface (Panchal *et al.* 2019). Specifically, for the nanocellulose + 10 % glycerol treatment, the droplet was rapidly absorbed, and its WCA was close to 0°, indicating a superhydrophilic surface. Hence, it was not possible to measure the contact angle over time.

The high standard deviation in the nanocellulose and nanocellulose + 1 % glycerol treatments is related to the heterogeneity of the film on the cotton fabric's surface, resulting in some areas with more deposited material, consequently altering the wettability degree.

Lignin contains hydrophobic domains ($-CH_3$), and previous studies have reported that lignin exhibits greater hydrophobic characteristics than cellulose due to its larger contact angle (Hodgson and Berg 1988). The increase in the contact angle for the nanolignin treatment was subtle when compared to the untreated cotton fabric, making them similar.

In summary, the incorporation of silicone elastomer enhanced hydrophobicity of the treated cotton fabrics, particularly in the nanolignin + silicone nanocoating, while glycerol led to increased hydrophilicity compared to treatments with only nanolignin or nanocellulose. This can be explained by their chemical structures. Glycerol, with its three hydroxyl groups, forms hydrogen bonds responsible for its high boiling point (290 °C) and infinite solubility in water and ethanol, making it highly hygroscopic (Castelló *et al.* 2009). On the other hand, silicone possesses hydrophobic characteristics due to its chemical composition, featuring typically nonpolar and water-repellent chains (Amin *et al.* 2007). Its wetting resistance arises from the presence of methyl groups ($-CH_3$), which are hydrophobic (Zhao *et al.* 2009).

While the results are promising, further in-depth studies are needed. Hydrophobic samples show potential for use in disposable clothing and aprons for medical and healthcare applications where washability is not a limiting factor. Hydrophilic samples, when using the correct proportion of glycerol, demonstrate potential for applications requiring high absorption, such as sanitary napkins and diapers. Since glycerol and silicone elastomer are already used in cosmetics, the use of the nanocellulose + glycerol treatments with adequate proportions could be explored for application in facial masks.

Mechanical properties

In the tensile tests, the Taylor modulus and the rupture force (Table 5) of the samples were calculated based on the data obtained during the experiments. Each graph displays the four repetitions of the analysis and their respective average curve (Figure 7).

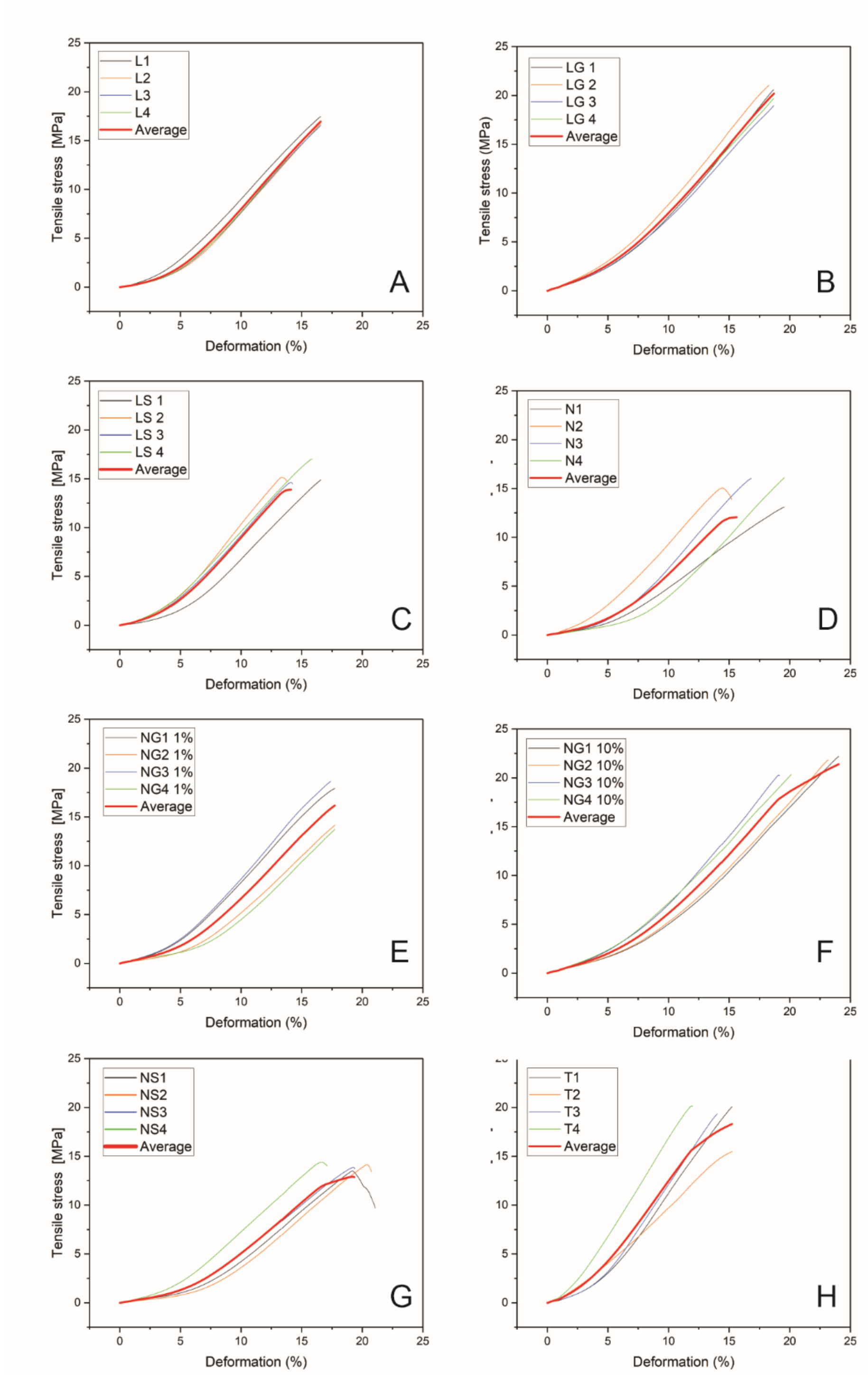


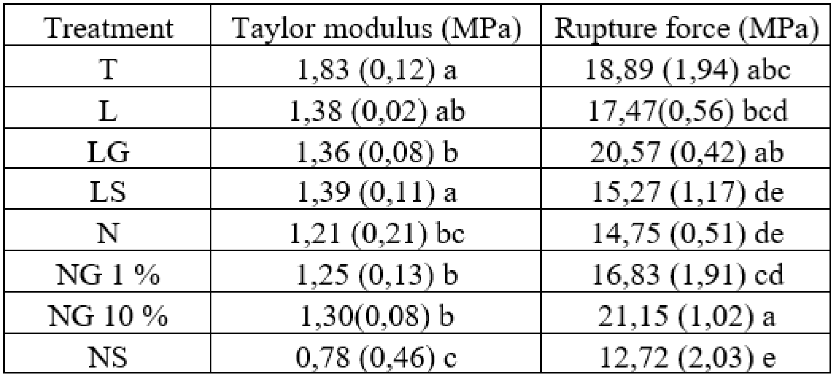
Figure 7: Graphs of the tensile analysis of untreated and treated cotton fabrics with the four repetitions and their respective average curves.

All treatments with glycerol and untreated cotton fabrics exhibited high tensile strength values. This occurs because glycerol enhances the resistance of the fabric coating due to its properties as a plasticizer, which improves flexibility and reduces material brittleness. It also promotes molecular interactions by forming hydrogen bonds with the coating matrix (NFC and nanolignin), improving cohesion and adhesion to the substrate. Furthermore, glycerol helps distribute stresses uniformly and acts as a damping agent, preventing premature rupture and increasing tensile strength (Roy *et al.* 2022, Bartolome *et al.* 2020). As mentioned earlier, untreated cotton fabrics contain residues from lubricants (such as tallow or mineral oil). Tallow can have a more direct effect on flexibility and resistance (Dąbrowski 2024).

Coating treatment with silicon elastomer showed a decrease in tensile strength when combined with nanolignin, but it made the coating more homogeneous in terms of flexibility with NFC. Silicone elastomer, acting as a plasticizer and binder, makes the coating more uniform. This can decrease the tensile strength, as it allows greater deformation without failure, but compromises the rigidity needed to resist rupture. When combined with nanolignin and NFC, silicone elastomer helps form a more cohesive and uniform network, although it may reduce the material's ability to withstand high tensile forces (Eduok *et al.* 2017).

The Taylor modulus indicated that the treatment with lignin and silicone elastomer, as well as the untreated cotton fabrics, showed the lowest flexibility among the treatments (Table 5).

Table 5: Average values of the Taylor modulus and rupture. Values between parenthesis corresponding to standard deviation. Different letters within the same column mean statistically significant differences.



Treatment	Taylor modulus (MPa)	Rupture force (MPa)
T	1,83 (0,12) a	18,89 (1,94) abc
L	1,38 (0,02) ab	17,47(0,56) bcd
LG	1,36 (0,08) b	20,57 (0,42) ab
LS	1,39 (0,11) a	15,27 (1,17) de
N	1,21 (0,21) bc	14,75 (0,51) de
NG 1 %	1,25 (0,13) b	16,83 (1,91) cd
NG 10 %	1,30(0,08) b	21,15 (1,02) a
NS	0,78 (0,46) c	12,72 (2,03) e

The most flexible treatment, with the lowest rupture force, was nanocellulose with silicone elastomer, confirming what was previously mentioned. The maximum force the fabric could withstand before breaking was 21,15 MPa in the treatment with nanocellulose and 10 % glycerol, demonstrating that glycerol enhances the strength of the cotton fabric.

Conclusions

The study explored the use of nanostructured coatings based on nanocellulose and nanolignin, both individually and combined with additives such as glycerol and silicone, on cotton fabrics. The findings show that the different treatments led to variations in surface wettability, optical properties, and weight gain, with nanolignin + silicone generating a surface with reduced wettability and higher weight gain, and nanocellulose improving reflectance. Glycerol-treated fabrics remained hydrophilic and showed minimal visual changes.

These observations indicate that renewable nanomaterials can be used to modify surface and optical properties of cotton fabrics under the conditions tested. The outcome of the nanolignin + silicone treatment, in particular, points to its potential use in water-repellent or stain-resistant textiles. However, aspects such as long-term performance and real-use behavior were not explored in this study and could be addressed in future work.

Although this was an exploratory study, it contributes to addressing the initial knowledge gap related to the use of biobased nanomaterials as functional textile coatings. Future research could expand the range of characterization methods or examine the performance of these materials in more varied textile applications.

Authorship contributions

I. C. B. Z.: Conceptualization, methodology, formal analysis, investigation, writing - original draft. G. G. L.: Investigation, data curation. M. E. B. C.: Resources, supervision. G. I. B. M.: Resources, supervision, funding acquisition, project administration, funding acquisition. P. H. G. C.: Visualization, supervision, writing - review & editing.

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