

Article

Biomass Waste-Derived Chitosan/Sacred Lotus Leaf Wax Composite Biocoating for Water-Resistant Cotton Fabrics

Walaikorn Nitayaphat ^{1,*}, Kageeporn Wongpreedee ² and Thanut Jintakosol ^{2,*}

¹ Department of Chemistry, Faculty of Science, Srinakharinwirot University, Bangkok 10110, Thailand

² College of Creative Industry, Srinakharinwirot University, Bangkok 10110, Thailand; kageeporn@g.swu.ac.th

* Correspondence: walaikorn@g.swu.ac.th (W.N.); thanut@g.swu.ac.th (T.J.)

Abstract

A sustainable water-resistant coating based on a chitosan/sacred lotus leaf wax composite was developed and deposited onto cotton fabrics using a simple dip–dry–cure process. The effect of the chitosan-to-sacred lotus leaf wax weight ratio on the surface wettability, air permeability, water vapor permeability, mechanical properties, and laundering durability of the coated fabrics was systematically investigated. The incorporation of sacred lotus leaf wax significantly enhanced the hydrophobicity of the cotton fabrics, with the highest water contact angle (WCA) of 167.12° achieved at a chitosan-to-wax weight ratio of 7:3. Although increasing the wax content improved water repellency, it also resulted in slight reductions in tensile strength, air permeability, and water vapor permeability. After 20 home-machine washings, the coated fabric retained a WCA of 137.11°, demonstrating good laundering durability. These results demonstrate that the chitosan/sacred lotus leaf wax composite is a biodegradable, non-toxic, and environmentally friendly alternative to conventional water-repellent finishes, offering considerable potential for the development of sustainable functional textile coatings.

Keywords: chitosan; bio-based wax; lotus leaves; water-resistant cotton; surface coating; sustainable textiles; sustainable development

1. Introduction

In recent years, functional textiles have attracted considerable interest in both research and industrial areas due to their ability to provide a variety of advanced properties, such as antimicrobial activity, flame retardancy, self-cleaning performance, superhydrophobicity, ultraviolet (UV) protection, and wrinkle resistance [1,2]. This increasing attention is largely attributed to the development of diverse surface modification techniques, ranging from conventional chemical treatments to environmentally sustainable biological processes [3]. Among these methods, chemical finishing remains one of the most effective and widely adopted approaches for imparting new functionalities and enhancing the performance of both natural and synthetic textile materials [4].

Cotton fabric is widely used in daily life because of its excellent properties, including softness, thermal comfort, affordability, breathability, flexibility, mechanical durability, renewability, and biodegradability [5–7]. Despite these advantages, the naturally hydrophilic nature of cotton limits its performance in applications requiring water resistance [8]. To address this limitation, various strategies have been developed, including surface modification of the fabric and the application of hydrophobic coatings. Common water-repellent



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coating techniques include dip-coating [9], impregnation [10], padding [11], plasma treatment [12], sol-gel processing [13], and spray coating [14]. Among the available coating techniques, the dip-coating method is widely employed for fabric hydrophobic modification because of its simplicity, cost-effectiveness, and scalability [15]. With increasing demand for sustainable textile finishing technologies, the development of environmentally benign and efficient water-repellent agents has emerged as a promising strategy for achieving durable hydrophobic modification of fabrics. Various hydrophobic agents, such as fluorochemicals [16], polyurethanes [17], silanes [11], silicones [18], stearic acid [19], waxes [20], and acrylate-based materials [21], have been extensively investigated for improving the water-repellent properties of cotton fabrics. Although these substances exhibit excellent water-repellent performance, their persistence, bioaccumulation, and toxicity raise significant environmental and health concerns [22]. Consequently, increasing regulatory restrictions and growing environmental awareness have driven the development of more sustainable alternatives [23,24].

Sacred lotus (*Nelumbo nucifera* Gaertn.), a member of the Nelumbonaceae family, is widely cultivated in Australia, India, Japan, China, South Korea, and Thailand. Owing to its nutritional and therapeutic value, all parts of the plant, including the rhizomes, seeds, pods, and leaves, are extensively utilized as food and in traditional medicine [25]. The lotus leaf surface is not perfectly smooth but features numerous micrometer-sized papillae. Studies have shown that these papillae play a crucial role in the superhydrophobicity of the leaf surface [26]. In addition to its unique surface topography, the lotus leaf possesses a hydrophobic epicuticular wax layer composed of tubular wax crystals distributed across the epidermis [27]. These wax crystals consist predominantly of aliphatic compounds, including nonanol, nonanediol, long-chain n-alkanes (C21, C23, C25, C27, C29, and C31), fatty alcohols (C16, C20, C22, C24, C26, C28, and C29), fatty acids (C16, C17, C20, C22, C24, and C28), lipids, and several unidentified constituents. Among these, C27 n-alkanes and C29 fatty alcohols are the dominant components. These compounds are biosynthesized in the epidermal cells and subsequently secreted onto the leaf surface, where they spontaneously self-assemble into crystalline wax structures that further enhance the leaf's water-repellent properties [28,29]. Importantly, recent studies have confirmed that wax extracted from sacred lotus leaves retains water-repellent characteristics, indicating that lotus leaf biomass can serve as a promising natural source of hydrophobic wax for surface modification [30]. Thus, the utilization of discarded lotus leaves for wax extraction provides an opportunity to convert an underutilized plant residue into a value-added, bio-based water-repellent material.

Chitosan is a linear cationic polysaccharide composed of randomly distributed β -(1 $\rightarrow$ 4)-linked D-glucosamine and N-acetyl-D-glucosamine units, which are generated through the partial deacetylation of chitin [31]. It is commercially produced primarily by the deacetylation of chitin extracted from shellfish processing waste, particularly the shells of shrimp, crabs, and lobsters, although alternative sources such as fungi and insects have also been explored [32]. As the second most abundant natural biopolymer after cellulose, chitosan is an abundant, renewable, biodegradable, and biocompatible material [33]. Its film-forming capability, together with abundant amino and hydroxyl groups, promotes strong interactions with cellulose through hydrogen bonding and other intermolecular interactions, making chitosan a suitable bio-based material for textile coating [34]. Chitosan can therefore serve not only as a coating-forming material but also as an effective binder for immobilizing hydrophobic substances on cellulose fibers, potentially improving the adhesion and durability of hydrophobic coatings [35,36]. In particular, combining chitosan with natural waxes derived from agricultural biomass has emerged as a promising fluorine-free strategy for imparting water resistance to cotton fabrics [37,38]. The combination is attractive because chitosan and plant-derived waxes offer complementary properties: chi-

tosan can interact with the hydroxyl-rich cellulose surface and serve as a polymeric matrix, while the hydrophobic wax contributes water-repellent functionality by reducing surface wettability. Such synergistic effects provide an opportunity to develop biomass-based coating systems with enhanced water resistance while reducing reliance on conventional fluorinated water-repellent agents.

In this study, a sustainable water-resistant coating was developed by utilizing wax extracted from waste sacred lotus leaves in combination with chitosan as a bio-based binder. Stable coating suspensions were prepared by dispersing different amounts of the substance to obtain different concentrations of sacred lotus leaf wax in chitosan solutions and subsequently applied to cotton fabrics using a simple coating process. The coated fabrics were comprehensively characterized by FTIR spectroscopy to identify characteristic functional groups and assess possible coating–fiber interactions based on changes in characteristic absorption bands, including band shifts and variations in band intensity. In addition, the surface morphology, color characteristics, wettability, air permeability, water vapor transmission rate, and mechanical properties of the coated fabrics were evaluated. The findings provide insights into the combined effects of chitosan and sacred lotus leaf wax on the performance of the coated fabrics and demonstrate the potential of this fluorine-free, biomass-derived coating system as an environmentally friendly approach for producing durable water-resistant cotton textiles.

2. Materials and Methods

2.1. Materials

Fresh sacred lotus leaves were collected from submerged fields in Bang Phli District, Samut Prakan Province, Thailand. The specimens underwent a washing process utilizing clean water to eliminate any extraneous physical matter, were gently blotted dry with a fine lint-free cloth, and were then cut into small pieces (approximately $1 \times 1 \text{ cm}^2$). The high-molecular-weight chitosan (average molecular weight $\sim 2100 \text{ kDa}$; degree of deacetylation $\sim 90\%$) was supplied by Marine Bio Resource Co., Ltd. (Samut Sakhon, Thailand). Plain weave cotton fabrics were obtained from a local fabric supplier. N-Hexane and ethanol of analytical reagent (AR) grade were purchased from Chemical Co., Ltd. (Bangkok, Thailand).

Prior to coating, the cotton fabrics were scoured using sodium carbonate to remove impurities and improve fabric wettability. Sodium carbonate was applied at a concentration of 4% on the weight of fabric (owf) using a liquor-to-fabric ratio of 1:100. The fabrics were treated at 80°C for 30 min, followed by thorough rinsing with cold water to remove residual scouring agent. The scoured fabrics were then dried at room temperature before further treatment.

2.2. Wax Extraction

Sacred lotus leaf wax was extracted using a solvent maceration method. Briefly, 100 g of sacred lotus leaves were soaked in 500 mL of n-hexane in a volumetric flask and kept at room temperature for 24 h with the lotus leaf crumbs completely submerged in the solvents. The solvent was subsequently evaporated using a rotary evaporator (Buchi, Flawil, Switzerland), and the residue primarily contained wax mixtures with some impurities. The crude residue was subsequently dispersed in 500 mL of ethanol and stirred at 1500 rpm at room temperature for 1 h to remove undesirable pigments and other colored compounds. The ethanol-insoluble fraction was recovered by filtration and centrifugation and subsequently collected as the crude wax fraction. Finally, the decolorized wax was dried in an oven at 60°C overnight and stored in a desiccator until further use.

2.3. Preparation of Chitosan/Sacred Lotus Leaf Wax Composites Dispersions

Chitosan/sacred lotus leaf wax composites were prepared using a solution-mixing method. Briefly, chitosan powder was dissolved in a 1% (v/v) aqueous acetic acid solution under continuous high-speed mixing at 2500 rpm, followed by stirring at 65 °C for 4 h. The use of high-speed mixing facilitated polymer dispersion, minimized aggregation, and promoted uniform dissolution. Sacred lotus leaf wax was subsequently added to the chitosan solution under vigorous stirring to form the chitosan/sacred lotus leaf wax composite. The total solid content of the resulting composite suspensions was maintained at 1% (w/v), with chitosan and sacred lotus leaf wax mixed at weight ratios of 10:0, 9:1, 7:3, 5:5, and 3:7. The corresponding concentrations of chitosan/sacred lotus leaf wax were 1.0/0, 0.9/0.1, 0.7/0.3, 0.5/0.5, and 0.3/0.7% (w/v), respectively. The mixtures were subsequently stirred continuously at room temperature for 4 h to obtain homogeneous suspensions. The resulting chitosan/sacred lotus leaf wax composite suspensions were designated as “XW,” where X represents the weight ratio of chitosan to sacred lotus leaf wax in the composite.

2.4. Coating Chitosan/Sacred Lotus Leaf Wax Composite on the Cotton Fabric

Chitosan/sacred lotus leaf wax composite solutions were applied on cotton fabrics using a dip–dry–cure technique. The cotton fabrics were soaked in composite solution and ultrasonicated at room temperature for 10 min using a fabric-to-liquor ratio of 1:20. The treated fabrics were then dried at 80 °C for 10 min, followed by curing at 120 °C for 3 min. Subsequently, the fabric samples were stored in a desiccator at 30 ± 3 °C prior to further characterization.

2.5. Measurement

2.5.1. Fourier Transform Infrared Analysis

Fourier Transform Infrared Spectroscopy (FTIR) was used to characterize the functional groups present in the uncoated and coated cotton fabrics and to assess possible chemical interactions between the cotton substrate and the deposited chitosan/sacred lotus leaf wax coating. FTIR measurements were performed using a BRUKER ALPHA II FTIR spectrometer (Bruker Optik GmbH, Ettlingen, Germany) equipped with an attenuated total reflectance (ATR) accessory. Fabric specimens were cut into small pieces (~1 mm) and placed directly on the ATR crystal. The spectra were recorded over a wavenumber range of 4000 to 600 cm^{-1} with a resolution of 2 cm^{-1} .

2.5.2. Color Measurements

The color parameters (CIELAB coordinates) of the cotton fabric samples were measured using an UltraScan VIS spectrophotometer (Hunter Lab, Reston, VA, USA) under a D65 illuminant and a 10° standard observer. Each sample was evaluated at five different positions, and the average values were recorded. Untreated cotton fabric was used as the reference sample. The measured color parameters included lightness (L^*), redness-greenness (a^*), blueness-yellowness (b^*), color difference (ΔE), whiteness index (WI), and yellowness index (YI).

Whiteness assessment is important not only for products intended to appear white, but also for non-white products, as it reflects the overall visual appearance of the material. A higher whiteness index value indicates a greater degree of whiteness. The whiteness index (WI) of the fabric samples was calculated using the Hunter whiteness index equation, as shown in Equation (1):

$$\text{whiteness index(WI)} = L^* - 3b^* \quad (1)$$

The yellowness index (YI) was determined directly using the instrument in accordance with ASTM E313-1973 [39].

The total color difference (ΔE) between the untreated and treated fabrics was calculated using Equation (2):

$$\Delta E = \sqrt{(L_0^* - L^*)^2 + (a_0^* - a^*)^2 + (b_0^* - b^*)^2} \quad (2)$$

where L_0^* , a_0^* , and b_0^* represent the color coordinates of the untreated fabric, and L^* , a^* , and b^* correspond to those of the treated fabric.

2.5.3. Mechanical Characterization

The mechanical properties of the cotton fabrics, including tensile strength, elongation at break, and stiffness, were evaluated in both the warp and weft directions. Tensile properties were measured using a Instron 5965 universal testing machine (Instron, Norwood, MA, USA) in accordance with ASTM D5035-11 (2024) (strip method) [40]. Fabric stiffness was determined according to ASTM D1388-14 [41] using a Shirley Stiffness Tester (Shirley Development Ltd., Didsbury, Manchester, UK). Stiffness was characterized in terms of bending length and flexural rigidity. All reported values represent the average of at least five independent measurements.

2.5.4. Water Contact Angle

Static water contact angles were measured using a contact-angle goniometer (KINO SL150E, KINO Scientific Instrument Inc., Boston, MA, USA). A 0.5 μ L water droplet was carefully deposited onto the treated surface of the fabric, and the droplet profile was monitored for 60 s. Images were acquired at predetermined time intervals during the observation period to monitor changes in droplet shape and contact angle associated with spreading or penetration into the porous fabric structure. The contact angle measured at 5 s after droplet deposition was used for comparison among the samples. Measurements were conducted on the treated surface only, with the fabric specimens positioned consistently with respect to the warp and weft directions. Measurements were performed at multiple randomly selected locations on each specimen, and the mean value was reported.

2.5.5. Morphological Characterization

The surface morphology of cotton fabric samples was observed using JSM-IT210 scanning electron microscope (JEOL Ltd., Akishima, Tokyo, Japan). Fabric samples were securely mounted on a specimen holder using carbon tape to prevent movement during handling. Prior to observation, the specimens were sputter-coated with a thin conductive gold layer to minimize charge accumulation. Micrographs were obtained at magnifications of 300 $\times$ to characterize the surface features of the fabrics.

2.5.6. Physical Characterization

The physical properties of the cotton fabrics, including air permeability and water vapor transmission rate (WVTR), were evaluated. Air permeability was determined using an air permeability tester (Model M021A, SDL Atlas, Rock Hill, SC, USA) in accordance with ISO 9237 [42]. Fabric specimens (6 cm²) were conditioned by drying at 60 °C for 24 h before testing. Measurements were performed using a circular test head with an effective test area of approximately 5 cm² under a pressure differential of 100 Pa. Water vapor transmission properties of the cotton fabrics were evaluated using a Water Permeability Tester (Model M261, SDL Atlas, Rock Hill, SC, USA) according to BS 7209 [43]. The WVTR was calculated according to Equation (3) and expressed as g/m²/day.

$$WVP = \frac{24M}{At} \quad (3)$$

where M_r is the mass loss of the test assembly during the measurement period (g), A is the exposed area of the fabric or the internal area of the test dish (m^2), and t is the time interval between successive weighings (h).

2.5.7. Coating Durability

The washing durability of the coated cotton fabrics was evaluated according to AATCC 61-1994 test method [44] using a Launder-O-meter under Test No. 2A conditions. Each fabric specimen was placed in a stainless-steel canister containing 150 mL of detergent solution and 50 stainless-steel balls. The specimens were laundered at $49 \pm 2^\circ\text{C}$ for 45 min per cycle. The accelerated laundering procedure was conducted for four cycles, with each cycle corresponding to approximately five home-machine washings, resulting in an equivalent of 20 home-machine washings. After each laundering cycle, the specimens were removed, rinsed thoroughly with distilled water, and dried under ambient conditions. The weight loss and water contact angle (WCA) were measured before and after the four accelerated laundering cycles to evaluate coating retention and water-repellent performance, respectively.

3. Results and Discussion

3.1. FTIR Analysis

Figure 1 presents the FTIR spectra of chitosan, sacred lotus leaf wax, and the chitosan/sacred lotus leaf wax composite, presented with vertical offsets for clarity.

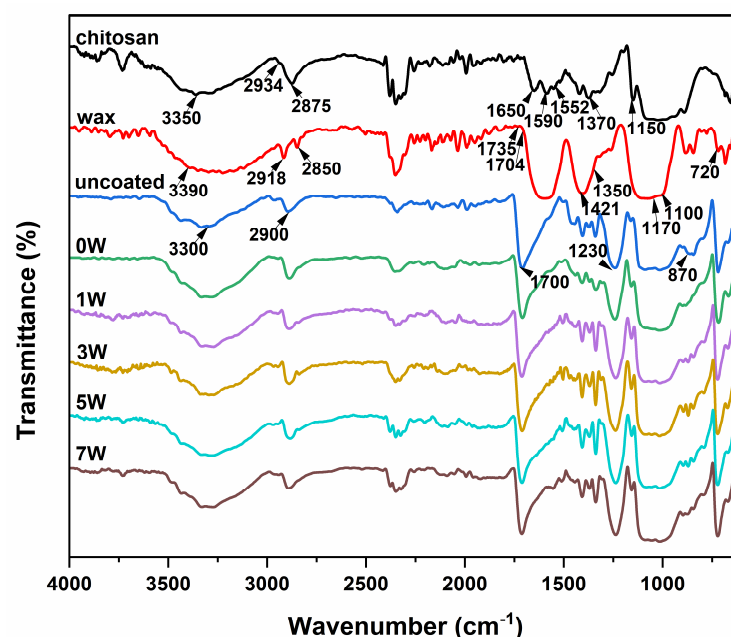


Figure 1. FTIR transmission spectra of chitosan, extracted sacred lotus leaf wax, uncoated cotton fabric, and cotton fabrics coated with a chitosan/sacred lotus leaf wax composite.

For chitosan, the band at 3350 cm^{-1} corresponds to O-H stretching vibration and the N-H extension vibration of the polysaccharide moieties of chitosan [38,45]. The band at 2934 and 2875 cm^{-1} is associated with symmetric or asymmetric CH_2 stretching vibration [45]. The band at 1650 cm^{-1} is attributed to C=O in the amide group (amide I band), and that at 1590 cm^{-1} is attributed to the N-H bending vibration in the amide group [46]. The band at 1552 cm^{-1} is attributed to N-H amide II bending and C-N stretching vibra-

tions [47]. The band at 1370 cm^{-1} is likely due to the stretching vibrations of carbodiimides and CH_3 in the amide group of chitosan [48]. The band at 1150 cm^{-1} corresponds to C-O stretching [46,47].

The FTIR spectrum of extracted sacred lotus leaf wax confirmed the presence of characteristic functional groups commonly found in plant epicuticular waxes. The absorption bands at 2918 and 2850 cm^{-1} were assigned to the asymmetric and symmetric stretching vibrations of aliphatic C-H bonds ($-\text{CH}_2$ and $-\text{CH}_3$), indicating the presence of long-chain hydrocarbons. The bands at 1421 and 1350 cm^{-1} corresponded to the bending vibrations of $-\text{CH}_2$ and $-\text{CH}_3$ groups, respectively. A broad absorption band centered at approximately 3390 cm^{-1} was attributed to O-H stretching vibrations, while the bands at 1170 and 1100 cm^{-1} were assigned to C-O stretching vibrations, suggesting the presence of alcohol-containing compounds in the wax. The detection and intensity of the O-H band may depend on the chemical composition of the wax as well as the extraction solvent, as reported in previous studies [49]. In addition, the absorption bands at 1735 and 1704 cm^{-1} were assigned to C=O stretching vibrations of ester, aldehyde, and carboxylic acid functional groups, which are commonly found in plant waxes [50]. The band observed at approximately 720 cm^{-1} was attributed to the rocking vibration of long-chain methylene ($-\text{CH}_2-$) groups, further confirming the presence of crystalline long-chain aliphatic hydrocarbons in the wax. The dominance of these long hydrocarbon chains contributes to the intrinsically low surface energy of the wax. Consequently, the hydrophobicity of sacred lotus leaf wax is primarily attributed to its high content of nonpolar aliphatic compounds, which minimize favorable interactions with water and reduce surface wettability. In general, increasing the proportion of long-chain alkyl groups decreases the surface free energy of the wax, thereby enhancing its water-repellent properties [51].

For the uncoated cotton fabric, the FTIR spectrum exhibited the characteristic absorption bands of cellulose. A broad band centered at approximately 3300 cm^{-1} was attributed to the stretching vibrations of hydroxyl (O-H) groups, reflecting the extensive hydrogen bonding present in cellulose. The absorption bands around 2900 cm^{-1} corresponded to the symmetric and asymmetric stretching vibrations of $-\text{CH}_2$ associated with the aliphatic structure of cellulose. A band near 1700 cm^{-1} was assigned to carbonyl (C=O) stretching vibrations, which may originate from oxidized cellulose species or residual impurities from the scouring process [52]. In addition, the fingerprint region between 1230 and 870 cm^{-1} contained several characteristic cellulose bands related to the stretching and bending vibrations of the polysaccharide backbone, including C-O-C glycosidic linkages and C-O-H bonds [53].

Compared with the uncoated cotton fabric, the FTIR spectra of the coated fabrics exhibited noticeable changes, confirming the successful deposition of the chitosan/sacred lotus leaf wax coating. Characteristic absorption bands originating from both cellulose and the coating components were observed, and their intensities varied with the chitosan-to-wax ratio. In particular, the wax-related bands at 2918 , 2850 , 1735 , 1421 , and 720 cm^{-1} became progressively more intense in the 1W, 3W, 5W, and 7W samples, indicating an increase in the amount of sacred lotus leaf wax deposited on the cotton fiber surfaces with increasing wax concentration.

These results indicate the successful deposition of the chitosan/sacred lotus leaf wax composite coating onto the cotton fabric. The FTIR spectra exhibited the characteristic absorption bands of chitosan, sacred lotus leaf wax, and cotton cellulose. However, no distinct new absorption bands or clear, systematic shifts in the characteristic bands were observed after coating. Therefore, the FTIR results do not provide direct evidence for the formation of new chemical bonds or allow the proposed intermolecular interactions to be conclusively established. Nevertheless, based on the functional groups present in the constituent materials, hydrogen bonding may contribute to the adhesion of the composite

coating to the cotton substrate. The hydroxyl (-OH) groups of cellulose and chitosan, together with the amino (-NH₂) and hydroxyl (-OH) groups of chitosan and oxygen-containing functional groups in the wax, may participate in intermolecular hydrogen bonding. The structural similarity between cellulose and chitosan, both of which are polysaccharides containing abundant hydroxyl groups, may further facilitate interfacial compatibility. In addition, van der Waals forces and physical interactions may contribute to the adhesion and retention of the composite coating on the cotton surface. These interactions are therefore proposed as possible mechanisms contributing to coating adhesion rather than as interactions directly confirmed by FTIR analysis. In addition to acting as a film-forming binder, chitosan may facilitate the dispersion and stabilization of the hydrophobic wax phase within the coating formulation. Under acidic conditions, the amino groups of chitosan become protonated, which may promote interactions with oxygen-containing functional groups at the wax-chitosan interface. Accordingly, the sacred lotus leaf wax was dispersed as a hydrophobic phase within the chitosan solution through vigorous stirring and ultrasonication rather than being molecularly dissolved. Hydrogen bonding, van der Waals forces, and possible electrostatic interactions may collectively contribute to reducing wax aggregation and promoting its distribution within the chitosan matrix [54,55]. These proposed interactions should be regarded as plausible explanations for the observed coating morphology and stability rather than as mechanisms directly demonstrated by FTIR spectroscopy.

3.2. Colorimetric Data and Visual Appearance

Figure 2 and Table 1 present the colorimetric parameters and visual appearance of the uncoated and coated cotton fabrics. The uncoated cotton fabric exhibited a characteristic white appearance. Upon application of the chitosan/sacred lotus leaf wax composite coatings, the color characteristics of the fabrics changed, producing coated samples with distinct visual appearances. Nevertheless, all coated fabrics exhibited a light brownish coloration. The deposition of the chitosan/sacred lotus leaf wax composite coatings altered the color properties of the cotton fabrics compared with the uncoated substrate. For the cotton fabric coated only with chitosan (0W), the lightness (L*) decreased, while the a* (redness) and b* (yellowness) values increased. These changes are attributed to the intrinsic yellowish color of chitosan. Consequently, the 0W sample exhibited a lower whiteness index (WI) and a higher yellowness index (YI) than the uncoated fabric. These changes are primarily attributed to the intrinsic yellowish color of chitosan and the formation of a continuous chitosan layer on the fiber surface. In addition, the increased yellowing observed after coating may be associated with the chemical characteristics of chitosan and the thermal treatment applied during the curing process. Possible thermally induced changes in the coating components or cellulose substrate may contribute to the development of chromophoric structures, resulting in the increased yellowness of the coated fabric [56,57]. The incorporation of sacred lotus leaf wax into the chitosan coating further influenced the color characteristics of the coated fabrics. As the wax content increased, gradual changes in the L*, a*, and b* values were observed, indicating that the wax affected both the lightness and color characteristics of the coated fabrics. Compared with the uncoated fabrics, the chitosan/sacred lotus leaf wax composite-coated fabrics exhibited significantly greater redness and yellowness, together with a darker appearance. These changes can be attributed to the inherent color of the sacred lotus leaf wax and the formation of a thicker coating layer on the fiber surface (Figure 3). The effect of the coating on fabric color was further confirmed by the color difference (ΔE) values between the coated and uncoated samples. Notably, the application of chitosan and sacred lotus leaf wax coatings resulted in noticeable changes in the visual appearance of the cotton fabrics, as indicated by the ΔE

values greater than 2.0 [58]. Overall, the results demonstrate that the coating composition influenced the color of the treated fabrics, with the resulting color varying according to the relative proportions of chitosan and sacred lotus leaf wax in the coating formulation.

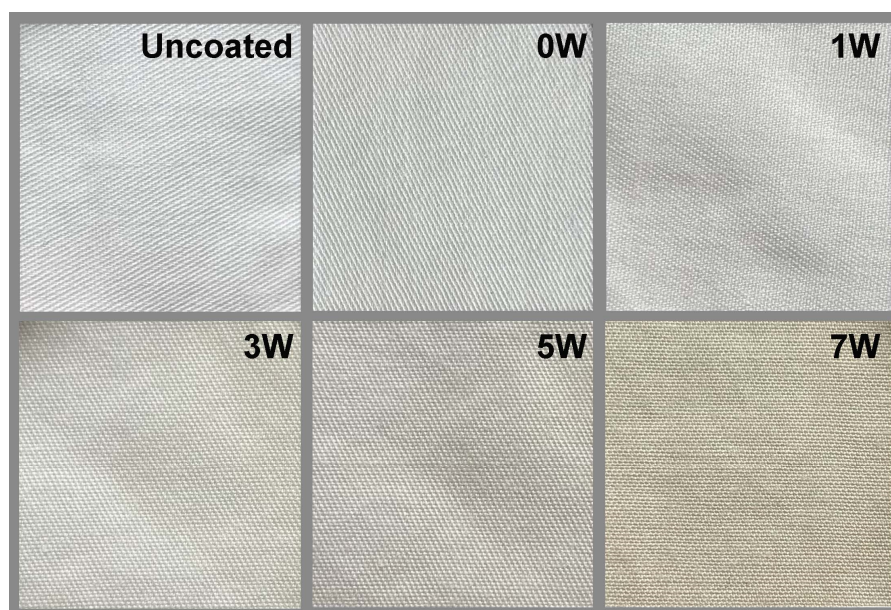


Figure 2. Photographs of cotton fabrics coated with chitosan/sacred lotus leaf wax composite.

Table 1. Colorimetric data of cotton fabrics coated with chitosan/sacred lotus leaf wax composite.

Samples	WI	YI	L*	a*	b*	ΔE
Uncoated	168.24 ± 1.1	-26.96 ± 0.3	102.00 ± 0.2	1.95 ± 0.1	-14.83 ± 0.1	-
0W	152.07 ± 1.2	-26.19 ± 0.6	98.06 ± 0.7	2.25 ± 0.1	-13.71 ± 0.2	4.11 ± 0.8
1W	130.98 ± 2.4	-15.80 ± 1.2	96.83 ± 0.3	2.47 ± 0.9	-8.80 ± 0.6	7.96 ± 0.4
3W	100.53 ± 2.5	-6.22 ± 0.3	92.90 ± 1.1	2.63 ± 0.1	-3.86 ± 0.2	14.27 ± 0.7
5W	88.81 ± 1.1	-0.06 ± 1.0	92.42 ± 0.3	2.79 ± 0.2	-0.52 ± 0.9	17.24 ± 0.7
7W	58.66 ± 0.7	11.73 ± 1.9	89.46 ± 1.2	4.07 ± 0.2	4.21 ± 0.8	22.90 ± 1.3

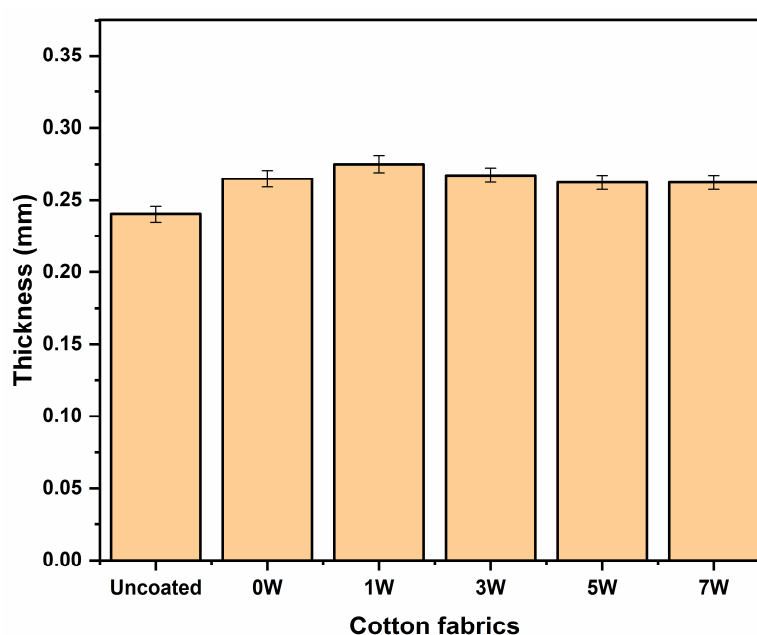


Figure 3. Thickness of cotton fabrics coated with chitosan/sacred lotus leaf wax composite.

3.3. Mechanical Properties

Breaking force is defined as the maximum force required to rupture a fabric specimen under uniaxial tensile loading [59]. It is an important mechanical property for evaluating the strength, durability, and structural integrity of textile fabrics. In the present study, breaking force was used to assess the effects of chitosan/sacred lotus leaf wax coating on the mechanical performance of cotton fabrics. The breaking force and elongation at break of the coated cotton fabrics in both the warp and weft directions are presented in Figures 4 and 5.

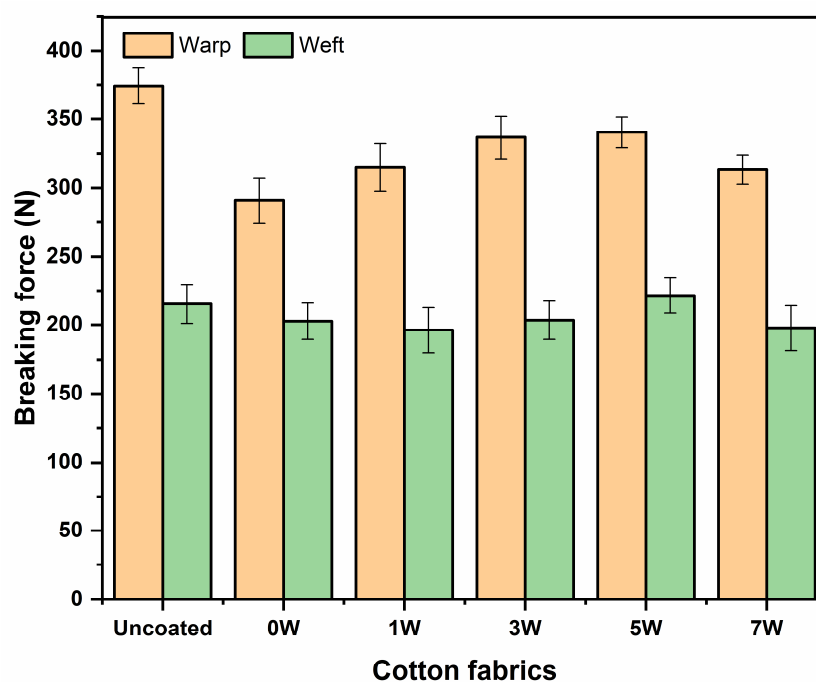


Figure 4. Breaking force of cotton fabrics coated with chitosan/sacred lotus leaf wax composite.

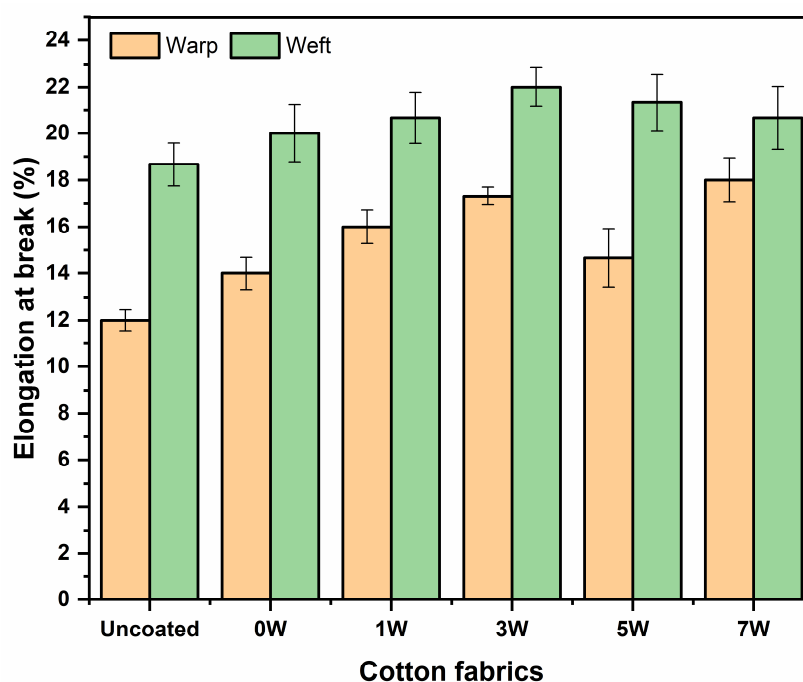


Figure 5. Elongation at break of cotton fabrics coated with chitosan/sacred lotus leaf wax composite.

Compared with the uncoated cotton fabric, the chitosan-coated fabric (0W) exhibited slightly lower breaking force and elongation at break in both the warp and weft directions. The reduction was more pronounced in the weft direction, whereas the warp direction retained relatively higher mechanical performance. The decrease in tensile properties is likely attributed to the acidic conditions used during the chitosan coating process. Specifically, the combined effects of acetic acid and the elevated curing temperature may induce partial hydrolysis of cellulose, resulting in degradation of the cotton fibers and consequently reduced breaking force and elongation [60]. In addition, the chitosan coating was predominantly observed on the fiber surfaces, with limited apparent deposition within the inter-fiber regions. This surface-localized coating may be associated with the observed changes in the mechanical properties of the coated fabric. However, the specific relationship between the coating morphology and stress transfer or load distribution could not be directly confirmed by the present analyses [61]. The tensile properties of the chitosan/sacred lotus leaf wax composite-coated fabrics showed similar mean values to those of the chitosan-coated fabric. The incorporation of sacred lotus leaf wax resulted in relatively small changes in the tensile properties under the conditions investigated. However, because no inferential statistical analysis was performed, statistical significance among the samples cannot be established. The observed similarity in tensile performance may be related to the predominantly surface-localized deposition of the wax phase.

All coated cotton fabrics exhibited a slight increase in elongation at break after the coating process. The minor changes in the mechanical properties are attributed to the deposition of sacred lotus leaf wax on the fiber surfaces. The wax layer likely reduces the frictional forces between adjacent fibers, promoting fiber slippage during tensile loading and consequently resulting in a slight decrease in breaking force accompanied by an increase in elongation at break. Similar changes in tensile behavior have been reported for cotton fabrics coated with chitosan-based functional layers, where the deposited coating modifies fiber-fiber interactions while largely preserving the structural integrity of the fabric. The slight reduction in breaking force and the corresponding increase in elongation are attributed to surface film formation and altered load transfer between adjacent fibers during tensile deformation [62–64].

Fabric stiffness, defined as the resistance of a fabric to bending deformation under its own weight or an applied force, is commonly characterized by the bending length and flexural rigidity. These parameters are among the most important mechanical properties for evaluating the handle, drape, and end-use performance of textile materials. Therefore, their determination provides valuable information for optimizing fabric comfort, functionality, durability, and processing performance. Among these parameters, bending length is a direct measure of fabric stiffness, with higher bending length values indicating greater resistance to bending and, consequently, a stiffer fabric structure [65]. Likewise, flexural rigidity represents the bending resistance of the fabric by considering both its bending length and mass per unit area, providing a comprehensive assessment of fabric stiffness [66]. As shown in Figures 6 and 7, the uncoated cotton fabric exhibited relatively low bending length and flexural rigidity, indicating good flexibility. This behavior is attributed to the porous woven structure of the fabric, which permits greater yarn mobility and facilitates bending under an applied load.

The bending length and flexural rigidity of the chitosan-coated cotton fabric were higher than those of the uncoated fabric, indicating that the chitosan coating increased fabric stiffness. This increase is attributed to the excellent film-forming ability of chitosan, which forms a continuous coating on the fiber surfaces and partially fills the inter-fiber spaces, thereby restricting fiber and yarn mobility during bending [56]. A further, although slight, increase in flexural rigidity was observed after coating with the chitosan/sacred

lotus leaf wax composite. Moreover, fabric stiffness increased progressively with increasing sacred lotus leaf wax content. This behavior is attributed to the formation of a continuous chitosan/wax composite film on the fabric surface, which limits fiber slippage and bending deformation, resulting in greater resistance to bending. Similar increases in bending length and flexural rigidity have been reported for chitosan-coated cotton fabrics, where the deposited polymer film reduced fabric flexibility by restricting the movement of fibers and yarns [67].

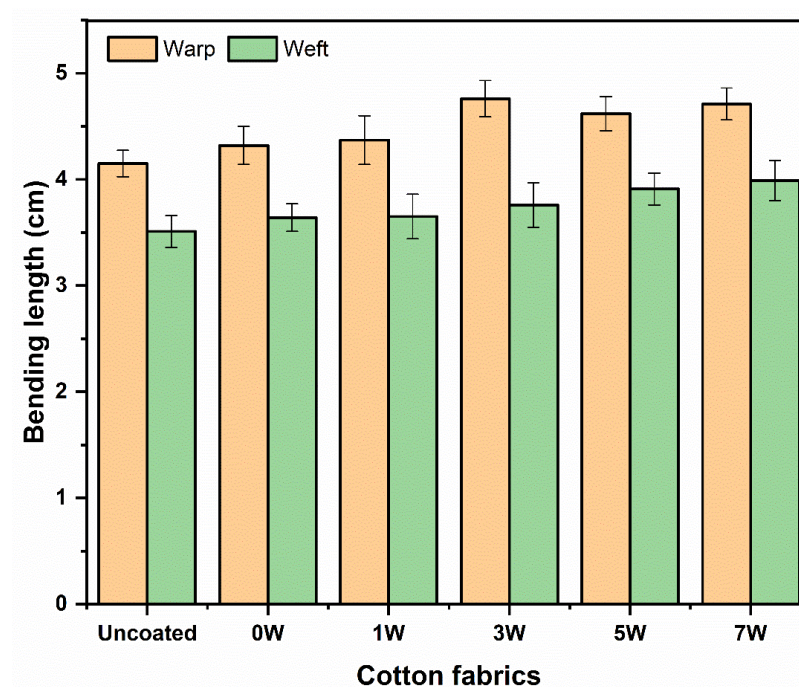


Figure 6. Bending length of cotton fabrics coated with chitosan/sacred lotus leaf wax composite.

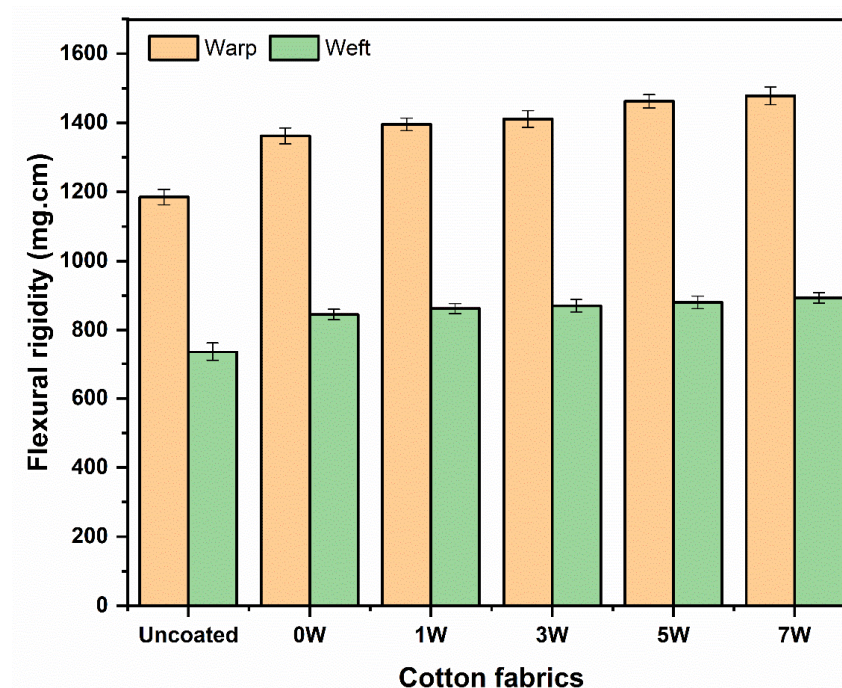


Figure 7. Flexural rigidity of cotton fabrics coated with chitosan/sacred lotus leaf wax composite.

3.4. Hydrophobic Property

The hydrophilic–hydrophobic character of the cotton fabrics was first evaluated by measuring the water contact angle (WCA). WCA is a widely used parameter for assessing the wettability and surface characteristics of textile materials because it reflects the interaction between a water droplet and the solid surface. The WCA is governed by both the surface chemistry (surface free energy) and the surface topography of the material. In general, a surface exhibiting a WCA greater than 90° is considered hydrophobic, indicating reduced wettability and a lower tendency for water penetration, whereas a WCA below 90° corresponds to a hydrophilic surface with high wettability and rapid water spreading. Therefore, WCA measurements provide a simple and effective means of assessing the effectiveness of surface modification treatments designed to impart water-repellent properties to textile substrates [68,69]. For hydrophobic coatings based on natural waxes, the water repellency is primarily attributed to the presence of long-chain aliphatic hydrocarbons, alcohols, fatty acids, esters, and other low-surface-energy constituents. Increasing the proportion and chain length of these aliphatic components generally lowers the surface free energy, thereby enhancing hydrophobicity and increasing the WCA. Furthermore, the formation of a continuous polymer-wax coating layer through intermolecular interactions between chitosan, wax, and cellulose improves surface coverage and reduces pathways for water penetration. When combined with an appropriate micro-/nano-scale surface roughness, these factors synergistically enhance the water-repellent performance of the coated fabrics [70,71]. The water-repellent performance of the coated fabrics was evaluated by measuring the static WCA (Figure 8), while the corresponding droplet morphologies were observed using optical images of water droplets deposited on the fabric surfaces (Figure 9).

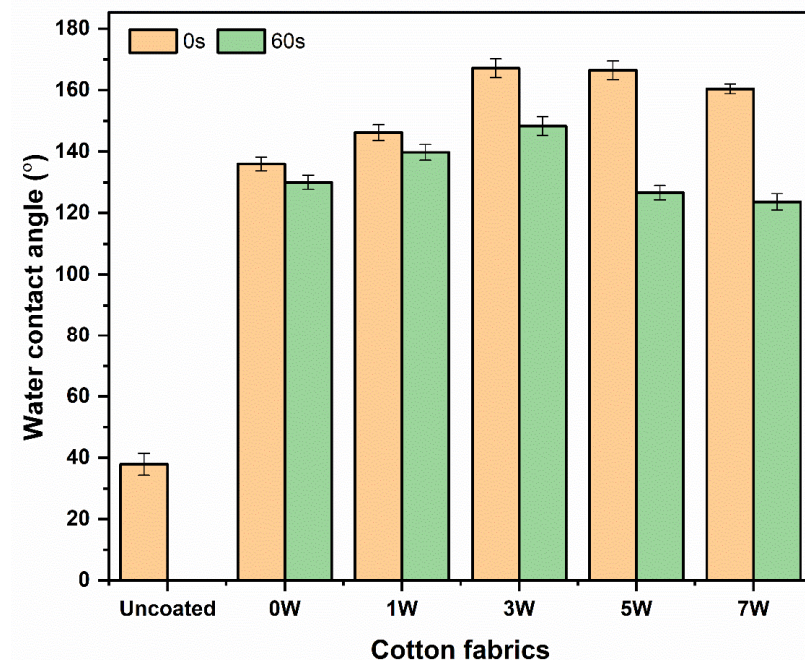


Figure 8. Water contact angle of cotton fabrics coated with chitosan/sacred lotus leaf wax composite.

The uncoated cotton fabric exhibited a WCA of 37.88° , confirming its inherently hydrophilic nature. This behavior is associated not only with the hydrophilic nature of cellulose, whose hydroxyl groups have a strong affinity for water, but also with the porous and fibrous structure of the cotton substrate. The interconnected spaces between fibers and yarns can facilitate rapid spreading and transport of water into the fabric structure. Conse-

quently, the deposited water can rapidly penetrate and distribute throughout the textile substrate, making a distinct droplet difficult to observe on the fabric surface. Therefore, the rapid disappearance of the water droplet should be considered a combined effect of the chemical hydrophilicity of cellulose and the capillary transport characteristics associated with the porous textile structure [72,73]. Following chitosan coating, the WCA increased markedly to 135.98° , indicating a transition from a hydrophilic to a hydrophobic surface. Although chitosan is intrinsically hydrophilic due to its hydroxyl and amino groups, the chitosan-coated cotton exhibited a higher WCA than the uncoated fabric. This apparent increase in hydrophobicity may be attributed to the formation of a dense, well-adhered chitosan film during drying, which, through intermolecular hydrogen bonding and molecular rearrangement, can reduce the accessibility of polar groups and limit direct water interaction with the underlying cellulose surface [74].

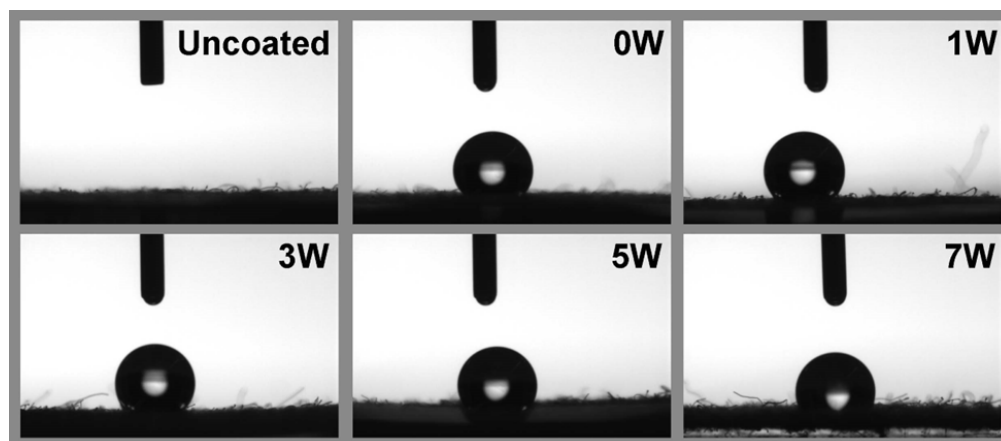


Figure 9. Microscopic images of water droplets on the surface of cotton fabrics coated with chitosan/sacred lotus leaf wax composite, measured at a holding time of 60 s.

The incorporation of sacred lotus leaf wax into the chitosan matrix further enhanced the water-resistance performance of the coated fabrics. The WCA increased progressively with increasing wax content and reached a maximum value of 167.12° for the 5W sample. This enhancement is attributed to the synergistic combination of the film-forming capability of chitosan and the low-surface-energy characteristics of the natural wax. Sacred lotus leaf wax consists predominantly of long-chain aliphatic hydrocarbons, fatty acids, fatty alcohols, esters, and other lipophilic constituents that effectively reduce the surface free energy of the coating. Moreover, chitosan functions as a bio-based emulsifier and stabilizing matrix, facilitating the homogeneous dispersion and immobilization of wax particles through electrostatic interactions and hydrogen bonding. The combination of reduced surface free energy and the increased micro-scale surface roughness generated by the deposited wax particles contributes to the remarkable enhancement in water repellency [75,76].

When the wax content exceeded the optimum level (5W and 7W samples), the WCA decreased slightly despite the higher wax loading. The formation of wax agglomerates on the fiber surfaces at elevated wax concentrations. Excessive wax loading likely exceeded the stabilizing capacity of the chitosan matrix, resulting in particle coalescence and heterogeneous coating coverage. Such non-uniform surface morphology can disrupt the continuity of the low-surface-energy layer and reduce the ability of the coating to effectively entrap air beneath water droplets, thereby decreasing the apparent contact angle.

3.5. SEM Analysis

Scanning electron microscopy (SEM) was employed to examine the surface morphology and fiber structure of cotton fabrics before and after coating with the chitosan/sacred

lotus leaf wax composite. The three-dimensional SEM images enabled detailed visualization of the morphological changes induced by the coating treatment. The corresponding SEM images are presented in Figure 10.

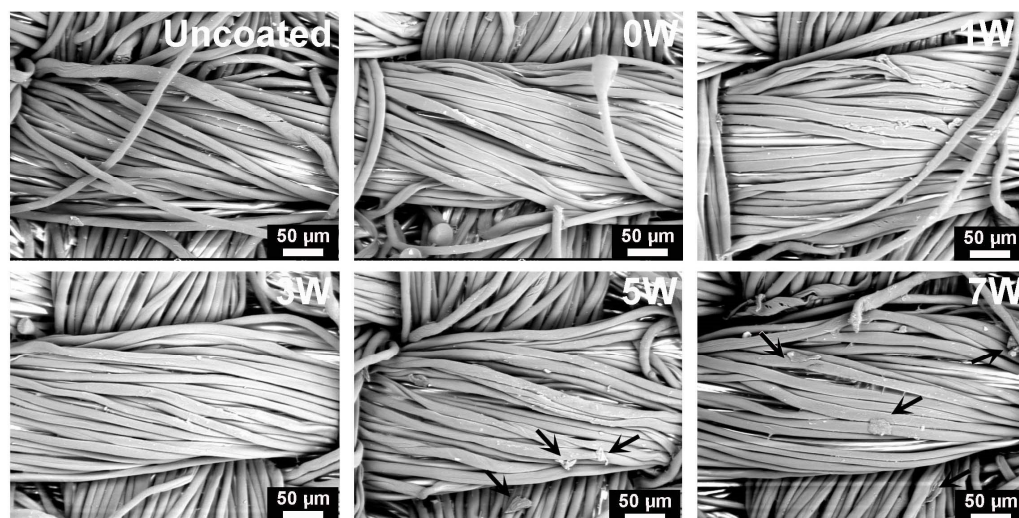


Figure 10. SEM micrographs of cotton fabrics coated with chitosan/sacred lotus leaf wax composite at a magnification of 300 $\times$.

The characteristic ribbon-like morphology and natural convolutions of the cotton fibers were preserved after the coating treatment. In the uncoated fabric, the fiber surfaces appeared relatively smooth and free of surface deposits, reflecting the inherent morphology of the scoured cotton substrate. This smooth appearance is consistent with the effective removal of most non-cellulosic impurities, including natural waxes, pectin, hemicellulose, and residual surface contaminants during the pretreatment process. In addition, distinct inter-fiber cavities were observed, corresponding to the porous structure of the woven cotton fabric, which facilitates air and moisture transport. In contrast, the chitosan-coated fabric (0W sample) exhibited a slightly rougher fiber surface than the uncoated fabric, which can be attributed to the deposition of a thin, continuous chitosan layer on the cellulose fibers. The coating is likely formed through hydrogen-bonding interactions and physical adhesion between chitosan and the cotton fiber surface, which may contribute to the adhesion of the biopolymer to the substrate. The hydroxyl groups of cellulose can interact with the hydroxyl and amino groups of chitosan through hydrogen bonding. In addition, chitosan may penetrate or conform to surface irregularities and pores of the cotton fibers before forming a continuous coating layer, thereby contributing to the physical retention of the coating on the fiber surface [77]. Following deposition of the chitosan/sacred lotus leaf wax composite, the wax particle domain became evident on the fiber surfaces, and their surface coverage increased progressively with increasing wax content. In the 5W sample, inter-fiber cavities remained visible, suggesting that the reduced chitosan content was insufficient to form a continuous matrix capable of effectively filling and bridging the fabric interstices. At higher wax loadings, pronounced wax agglomerates were observed, indicating reduced dispersion stability of the wax phase. This behavior is likely attributable to the limited amount of chitosan available to stabilize the molten wax droplets and maintain their homogeneous distribution during coating. Furthermore, the curing process was conducted at 120 $^{\circ}\text{C}$, which exceeds the reported melting temperature of natural plant waxes (approximately 68–86 $^{\circ}\text{C}$) [78,79]. Under these conditions, the wax particles would have melted and subsequently redistributed during drying before solidifying upon cooling, thereby promoting coalescence and the formation of localized wax agglomerates on the fiber surface.

3.6. Physical Properties

Air permeability and water vapor transmission (WVTR) of cotton fabrics coated with the chitosan/sacred lotus leaf wax composite were evaluated.

As shown in Figure 11, the uncoated cotton fabric exhibited an air permeability of $11.32 \text{ cm}^3/\text{cm}^2/\text{s}$ and a WVTR of $897.34 \text{ g}/\text{m}^2/\text{day}$. Following coating, both air permeability and WVTR decreased compared with the uncoated fabric. The air permeability decreased from approximately 11.32 to $8.16 \text{ cm}^3/\text{cm}^2/\text{s}$, corresponding to approximately 72.08% retention of the original air permeability. Similarly, the WVTR decreased from 897.34 to approximately $613.57 \text{ g}/\text{m}^2/\text{day}$, corresponding to approximately 68.38% retention. This reduction is attributed to the deposition of the chitosan/sacred lotus leaf wax composite on the fiber surface, which partially filled and blocked the inter-fiber pores, thereby restricting the passage of air and water vapor through the fabric. This interpretation is consistent with the SEM observations, which revealed progressive coverage of the inter-fiber voids and the formation of a more compact surface morphology after coating. Despite these reductions, the coated fabrics retained approximately 72% of the original air permeability and 68% of the original WVTR, indicating that the coating did not completely compromise the fabric's air and moisture-vapor transport properties. The WVTR of approximately $613.57 \text{ g}/\text{m}^2/\text{day}$ is also within the range reported for waterproof-breathable textile materials [80]. Notably, coating the cotton fabric with the natural chitosan/sacred lotus leaf wax composite using the dip-dry-cure process at a relatively low curing temperature resulted in only minor changes to the fabric properties. The coated fabrics largely retained their original physical characteristics, indicating that the finishing treatment caused minimal alteration to the cotton substrate. These findings demonstrate that the chitosan/sacred lotus leaf wax composite can serve as an environmentally friendly biopolymer coating without adversely affecting the physical performance of the fabric. Furthermore, the mild dip-dry-cure process provides a safe and effective textile finishing approach for producing water-resistant cotton fabrics while preserving their desirable properties [81].

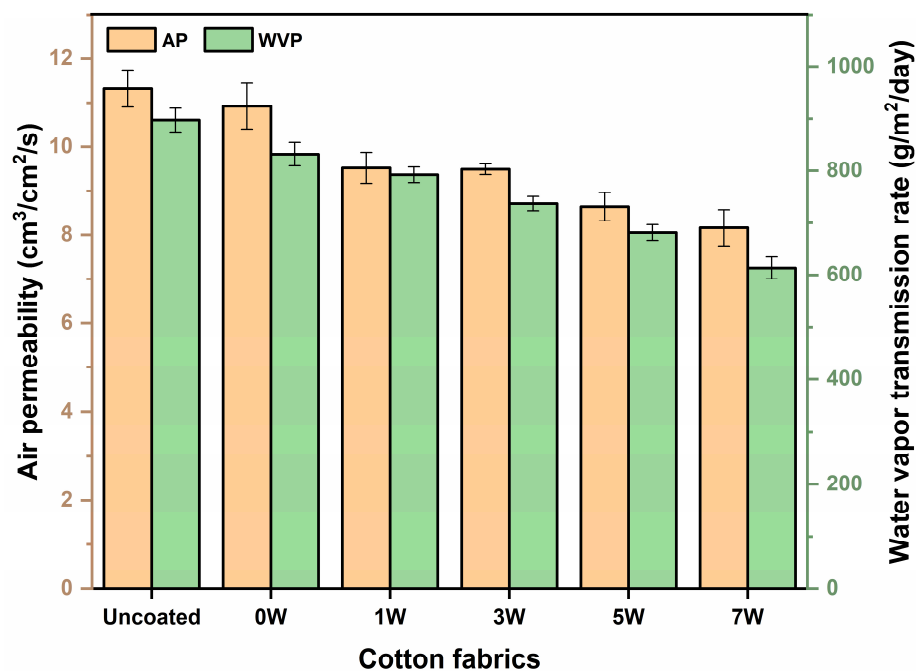


Figure 11. Air permeability and water vapor transmission of cotton fabrics coated with chitosan/sacred lotus leaf wax composite.

3.7. Durability Test

For practical applications, functional fabrics must retain their performance after repeated laundering, making washing durability a key indicator of their long-term serviceability. Therefore, the laundering durability of cotton fabrics coated with the chitosan/sacred lotus leaf wax composite was evaluated by monitoring changes in the WCA following successive washing cycles. The weight loss and water contact angle of cotton fabrics coated with chitosan/sacred lotus leaf wax composite after four accelerated laundering cycles, corresponding to approximately 20 home-machine washings, are presented in Figures 12 and 13, respectively.

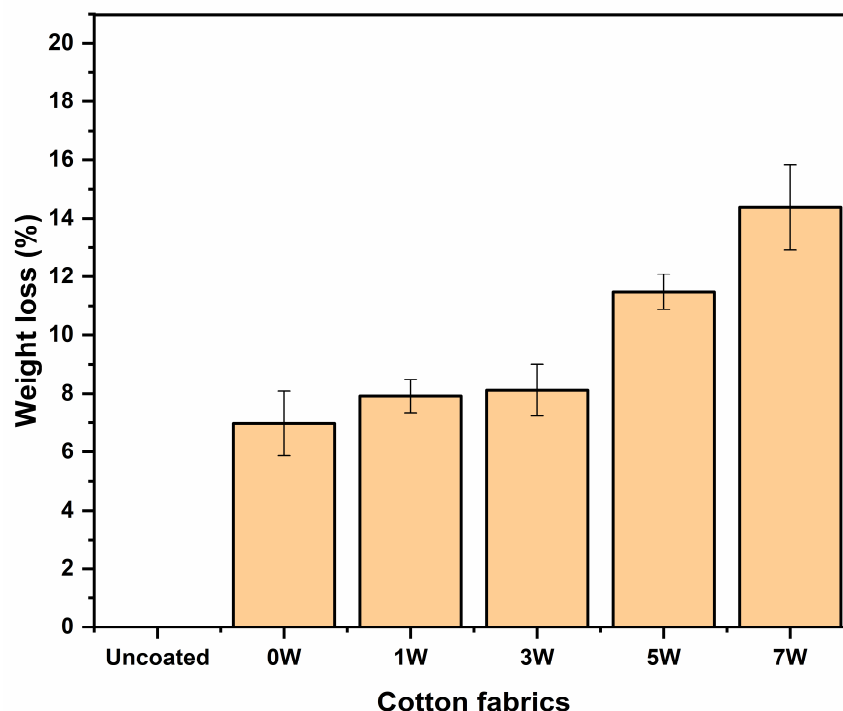


Figure 12. Weight loss of cotton fabrics coated with chitosan/sacred lotus leaf wax composite after four accelerated laundering cycles, corresponding to approximately 20 home-machine washings.

Figure 12 compares the weight loss of the coated cotton fabrics after four accelerated laundering cycles. The weight loss was determined from the difference in specimen weight before and after washing and was used to assess the washing durability and retention of the coatings. Among the tested samples, the chitosan-coated fabric exhibited the lowest weight loss, indicating relatively good retention of the chitosan coating on the cotton fiber surface. For the chitosan/sacred lotus leaf wax composite-coated fabrics, the weight loss increased with increasing wax content, suggesting that a greater amount of the wax phase was removed during laundering.

Figure 13 presents the WCA of the coated fabrics before and after four accelerated laundering cycles. After laundering, the WCA decreased for all coated fabrics, indicating a reduction in surface hydrophobicity. This decrease in water repellency can be attributed to partial disruption and removal of the coating during laundering. Mechanical agitation and repeated wetting–drying cycles may weaken the continuity and adhesion of the chitosan/wax coating on the cotton fiber surface. In addition, the alkaline components and surfactants in the detergent may promote swelling or partial degradation of the chitosan matrix and facilitate the removal of loosely bound wax from the fabric surface, thereby reducing its hydrophobicity [70]. Nevertheless, the coated fabrics retained relatively high WCA values after 20 home-machine washings, indicating that the chitosan/sacred lotus

leaf wax composite provided good resistance to laundering and maintained appreciable water-repellent performance.

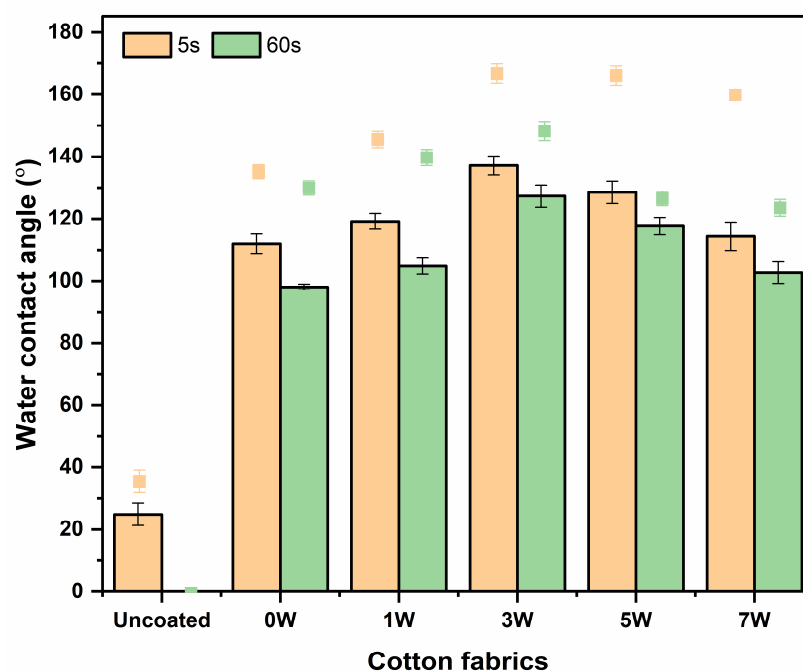


Figure 13. Water contact angles of cotton fabrics coated with chitosan/sacred lotus leaf wax composites after four accelerated laundering cycles, corresponding to approximately 20 home-machine washings; reference points indicate the WCA before laundering.

Compared with the previously reported chitosan/outer cabbage leaf wax coating [82], the chitosan/sacred lotus leaf wax composite exhibited higher water repellency, with a maximum WCA of 167.12°, compared with 157.87° for the chitosan/OCL wax system. Moreover, the chitosan/sacred lotus leaf wax composite-coated fabric retained a WCA of 137.11° after 20 home-machine washings, indicating good washing durability. To further evaluate its potential, the water-repellent performance of the chitosan/sacred lotus leaf wax composite coating was compared with previously reported bio-based wax coatings (Table 2). The WCA obtained in the present study is therefore competitive with, and in some cases higher than, those reported for analogous beeswax- and carnauba-wax-based coatings. Importantly, sacred lotus leaf wax is recovered from discarded lotus leaves, providing a renewable, waste-derived alternative to commercially available waxes. Thus, the chitosan/sacred lotus leaf wax composite system combines high water repellency, good laundering durability, and biomass waste valorization, highlighting its potential as a sustainable and fluorine-free wax-based coating for water-resistant textile applications.

Table 2. Comparison of the water-resistant performance of different bio-based wax coatings.

Wax/Coating System	Substrate	Method	WCA (°)	Durability	Reference
Beeswax	Polyester/cotton/viscose	Pad-dry	129.40	Not reported	[82]
Biosilica/chitosan/carnauba wax	Cotton	Dip-coating	154.70	Not reported	[83]
Zein nanoparticles/beeswax	Cotton	Layer-by-layer	140.20	Not reported	[84]
Chitosan/outer cabbage leaf wax	Cotton	Dip-coating	157.87	125.44 after 20 washings	[20]
Chitosan/sacred lotus leaf wax	Cotton	Dip-dry-cure	167.12	137.11 after 20 washings	This study

4. Conclusions

In summary, a sustainable water-repellent cotton fabric was successfully fabricated by depositing a chitosan/sacred lotus leaf wax composite onto the fabric surface using a simple dip–dry–cure process. The coated fabric exhibited excellent hydrophobicity, achieving a maximum water contact angle (WCA) of 167.12° at a chitosan-to-sacred lotus leaf wax weight ratio of 7:3. SEM observations confirmed the successful deposition of the composite coating on the cotton fiber surface while preserving the intrinsic fiber morphology. In addition, the coated fabric retained a WCA of 137.11° after 20 home-machine washings, demonstrating good laundering durability and the stability of the coating under repeated washing. Overall, the chitosan/sacred lotus leaf wax composite provides a biodegradable, non-toxic, and sustainable alternative to conventional synthetic water-repellent finishes, offering considerable potential for the development of durable water-repellent textiles and other environmentally friendly functional textile applications.

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Abbreviations

The following abbreviations are used in this manuscript:

WI	Whiteness index
YI	Yellowness index
WVP	Water vapor permeability
WCA	Water contact angle
FTIR	Fourier transform infrared spectroscopy
SEM	Scanning electron microscopy

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