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UTILISING CHITOSAN TO FACILITATE THE FUNCTIONALIZATION OF METHODOLOGY MATERIALS

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Abstract

Textiles need a range of qualities because they are utilised in so many diverse contexts. Due to their wide range of applications, textiles require a variety of characteristics. Better properties, such as hydrophilicity and antimicrobial activity, are present in textiles with wet functionalization. Because of this, the textile industry has looked at chitosan, a bio-based polymer, in great detail. In an effort to determine the most accurate method for detecting chitosan in textiles composed of cotton and polyester, two different kinds of weavings were functionalized with different amounts of chitosan: one composed wholly of polyester, and the other of cotton and polyester combined.

Chitosan solutions containing either 3-aminopropyltriethoxy silane (AMEO) or 3-glycidyloxypropyltriethoxy silane (GLYEO) were applied in one or two stages to the same fabrics. By dying the textile surfaces with Remazol Brilliant Red F3B and utilising energy-dispersive X-ray spectroscopy (EDS) and scanning electron microscopy (SEM), it was possible to demonstrate the presence of silanes and chitosan on the surfaces. Since the dyeing depths were dependent on the chitosan concentrations, we were able to ascertain the efficacy of a very short processing time and a mild dyeing temperature, even if non-functionalized fabrics were not discoloured. The fabric that had both AMEO and chitosan applied simultaneously had the highest concentration of silicon and the deepest colour intensity.

While chitosan-containing solutions boosted the material's hydrophobicity, GLYEO functionalization shortened the polyester's water sink-in time. Regardless of the kind of functionalization, washing tests showed that the cotton/polyester samples became more hydrophilic. These studies demonstrate that the type of functionalization can be utilised to modify the hydrophilic characteristics of polyester and cotton/polyester textiles, and that chitosan-containing recipes can be part of a practical approach. Future developments of novel blends of bio-based polymers and inorganic binder systems may be made through this initial stage, which could ultimately result in the creation of sustainable antibacterial materials with altered hydrophilic characteristics.

Keywords:

chitosan; polyester; cotton; 3-glycidyloxypropyltriethoxy silane (GLYEO); 3-aminopropyltriethoxy silane (AMEO); technical textiles; functionalization

1. Introduction

The term "functionalization of textiles" refers to any techniques utilised to give a textile substrate new, practical properties [1].These functional properties might be linked to whole other domains, including flame retardancy, UV protection, antibacterial activity, or hydrophilic or hydrophobic properties [2,3,4,5].Functionalization is a typical method that includes adding a chemical component or additive with a specific functional attribute to a textile fabric that does not have it. This process is known as wet chemical processing. Using bio-based and sustainable materials is a modern trend [6,7,8].

It is possible to functionalize textile processes with bio-based polymers to make them more ecologically friendly. One bio-based polymer that has been investigated for textile treatments is chitosan [9,10,11, 12]. The biopolymer chitin is created by deacetylating it. Chitin, which is derived from insects or crustaceans, is one popular raw material [13, 14]. The deacetylation of chitin results in the formation of primary amino groups in the polymer structure of chitosan. The molecular weight and the "degree of deacetylation" (DD), which is the ratio of acetyl groups to amino groups, greatly influence the chemical and physical properties of chitosan, such as its viscosity, solubility, and biological activity [15].

Chitosan has been studied for many years as a possible agent for textile treatments [16, 17]. Chitosan has received special attention due to its antibacterial properties [10,18]. In this method, bio-based antibacterial activities can be obtained.However, the use of chitosan can also cause modifications in textile materials that are hydrophilic, hydrophobic, or antistatic [19, 20]. In pure water, chitosan is not soluble.However, it is soluble in acidic solutions due to the protonation of the amino groups NH2 producing charged NH3+ units [21, 22].Because of this, chitosan is frequently dissolved in acidic solutions and employed in textile applications.

However, chitosan affects not only the previously mentioned properties but also the dyeing ability of cotton [12,23] and polyester [24,25] materials. While cotton may be effectively coloured with direct or reactive dyes in neutral or slightly alkaline solutions, polyester is usually dyed at high temperatures with dispersion dyes [26, 27]. Here, a reliable method for determining the presence of chitosan in cotton/polyester blends and pure polyester textiles has been developed by exploiting the effects of chitosan on textile dyeing behaviour. Additionally, spectroscopic techniques such as energy dispersive spectroscopy (EDS) and scanning electron Apart from this simple approach to chitosan functionalization of textiles, it's fascinating to modify chitosan applications by adding elements of hydrolyzed alkoxysilane [19].

Alkoxysilane components can be used as precursors in sol–gel processes and in combination with chitosan, as mentioned before [18]. However, in this study, chitosan is mixed with the only trialkoxy silanes that have been functionalized with amino and epoxy to treat textiles. PES and CO/PES woven fabrics have been functionalized for use in technical textiles using the adhesive properties of a silica-based binder system.

Ultimately, a proof of concept was accomplished by giving the alkoxy silane constituents chitosan's characteristics and washing stability, enabling the modification of textile substrates. It is possible to design novel inorganic binder systems and bio-based polymers to produce sustainable antibacterial materials with altered hydrophilic characteristics.

2. Materials and Methods

2.1. Textiles

Weaved materials supplied by an industrial partner (Wenzel & Hoos GmbH, Lauterbach, Germany) were utilised exactly as received. The materials were polyester (PES) and cotton/polyester blend (CO/PES). About 80% cotton and 20% polyester were combined together to create the cotton/polyester blended cloth.

2.2. Textile Finishing

Textiles were completed in one or two steps using silane/chitosan mixes and chitosan solutions in varying concentrations.

2.2.1. Chitosan Dispersion

A warm (60 °C) acetic acid solution containing chitosan (ChD; 90% DD; BioLog Heppe GmbH) (1 or 2%, prepared from 100% acetic acid, VWR International GmbH, Darmstadt, Germany) is used to functionalize textiles using chitosan solutions. , Landsberg, Germany) was stirred intensively for approximately 30 min using a magnetic stirrer (Carl Roth GmbH and Co. KG, Karlsruhe, Germany). The homogeneous chitosan solution was dispersed twice with a pad machine (Ernst Benz, Rümlang-Zurich, Switzerland or Wichelhaus GmbH and Co. KG, Solingen, Germany). The textiles were then oven dried at 130°C for five minutes and condensed at 170°C for one minute. Weighing the dry, unoperated sample and the wet operated sample for each sample allowed us to calculate the amount of alcohol collected during operation..

2.2.2. Silane Hydrolyzation

Silanes (3-glycidyloxypropyltriethoxysilane (GLYEO) or 3-aminopropyltriethoxysilane (AMEO)) were purchased from ABCR GmbH (Karlsruhe, Germany) and hydrolyzed before use. For this, one part of ethanol (99.7% + MEK, AnalytiChem GmbH, Duisburg, Germany) was placed in a beaker and then one part of AMEO or GLYEO was added. In addition, one part of acetic acid (50%) was added with vigorous stirring (ethanol:silane:acetic acid = 1:1:1 v/v/v). The mixture was stirred for 30 minutes at room temperature until a single-phase homogeneous solution (hydrolyzate) was obtained. From this mixture, a 2% diluted silane solution in water was prepared for later use..

2.2.3. One-Step Procedure for Application of Chitosan and Silane

46.5 mL of 2% dilute acetic acid, 3 mL of silane hydrolyzate, and 0.5 g of chitosan powder were combined in a beaker and stirred for 30 min at 60 °C as part of a one-step finishing technique. The treated textiles were then dried using the above method after padding with this solution.

2.2.4. Two-Step Procedure for Application of Chitosan and Silane

Initially, we applied the silane hydrolyzate using a horizontal padding machine in the two-step finishing procedure. Afterwards, the textile was placed in a bowl containing a 1% chitosan dispersion in acetic acid (2%) (see Section 2.2.1), manually squeezed, and then passed through a padding machine. Subsequently, the textile was dried according to the instructions provided.

2.3. Dyeing

The fabrics were stained with a reactive dye in order to determine the presence of chitosan. The samples were coloured using an aqueous solution containing 0.1% (w/v) Triton X-100 (Carl Roth GmbH & Co KG, Karlsruhe, Germany) and 2% Remazol Brilliant Red F3B (DyStar Colours Distribution GmbH, Raunheim, Germany) (see [29]). First, a 1:100 stock solution was made in order to create the dye solution. The samples were dyed in an Ahiba IR pro dyeing equipment (Datacolor GmbH, Marl, Germany) for five minutes at fifty degrees Celsius. Three degrees Celsius were heated every minute. One of the staining bombs has a temperature sensor that was used to detect the temperature. The samples were cold-rinsed twice for three minutes in one litre of soft water each right after dyeing.The samples were left to dry at room temperature while lying down.

2.4. Washing

Using an industrial washing machine (Electrolux Professional, WH6-11CV, Stockholm, Sweden), washing tests were conducted on a subset of samples. To wash the fabrics, 30 mL of liquid laundry detergent (Persil Power Gel, Henkel AG & Co. KGaA, Düsseldorf, Germany) was used, and a regular coloured washing programme was used at 40 °C. To guarantee even washing, a cotton lab coat was added to the load. After up to five rounds of washing, the samples were line-dried. The sample name for these was "B." When required, the unwashed samples were referred to as "A."

2.5. Analytics

2.5.1. Viscosity

Using a rotational rheometer (Haake viscotester iQ, thermos scientific, Karlsruhe, Germany) at 30 °C, the viscosity of the chitosan dispersions was determined. A measuring exemplar was carried out for both 0.5% and 3% ChD.

2.5.2. Thickness Measurement

The textiles' thickness was measured using a Micrometre universal S16502, Frank-PTI GmbH, Birkenau, Germany, in compliance with DIN EN ISO 5084_1996 standard. For every sample, five measurements were made. We computed the standard deviation and the average.

2.5.3. Air Permeability

Using an FX3300 Lab Air from Textest AG, Zurich, Switzerland, the air permeability of the textiles was assessed in accordance with DIN EN ISO 9237_1995 (DIN German Institute for Standardisation, Berlin, Germany). A test area of 20 cm2 and a differential pressure of 100 Pa were used for five measurements per cloth. We computed the standard deviation and the average.

2.5.4. Microscopy

Light microscopy (Digital Microscope VXH and VH-Z2OR, Keyence Deutschland, Neu-Isenberg, Germany) was used to acquire an overview of the fabric structure. Photos were obtained at a magnification of 30×. A scanning electron microscope (SEM) (Tabletop TM4000 Plus, Hitachi High-Technologies GmbH Europe, Krefeld, Germany) was used to examine the surfaces of the textiles, and magnification measurements were made at 100×, 300×, and 1000×. Energy-dispersive X-ray spectroscopy (EDS) was used to map the surfaces of the samples and identify the chemical components present (Bruker SCU, Brucker Nano GmbH, Berlin, Germany). The EDS mapping was done at a 300× magnification. The atom distribution in atom percent was assessed three times for each sample, and the mean value and standard deviation were computed.

2.5.5. UV-Vis Spectroscopy

After dyeing with Remazol Brilliant Red F3B, the textile samples were clamped without tension in a special sample holder within the UV-Vis spectrometer UV2600 from Shimadzu (Kyōto, Japan) in order to measure the samples' absorption. Every sample was tested three times within the 400–700 nm wavelength range. The average value throughout the three measurement curves was ascertained, and the standard deviations for the average absorptions at 550 nm were computed.

2.5.6. TEGEWA Drop-Test

Using an aqueous solution of Patent Blue V (2%), the TEGEWA drop-test was used to evaluate the completed textiles' water absorption qualities, as previously mentioned [30]. The test comprised timing the droplets' sink-in period and gauging how far they dispersed in the weft and warp directions. For every sample, the test was run three times, and the mean values were computed. Samples that took longer than five minutes to sink were only examined once.

3. Results and Discussion

3.1. Different Concentrations of Chitosan

The initial trials were conducted in order to develop a reliable technique for detecting chitosan. As a result, various concentrations of chitosan were applied to the textiles, and various analytical techniques were assessed. For this, 0.5–4% quantities of ChD were dissolved in 1% acetic acid. After that, the solution was padded onto polyester and cotton/polyester textiles. REM/EDS analysis was performed on greater chitosan concentrations in addition to the findings reported in [28]. Additionally, samples functionalized with chitosan were dyed with Remazol Red, a reactive dye.

The liquor uptake during functionalization for every sample was calculated using a technique that has been previously published [28]. The liquid pickups at 1% and 3% ChD concentrations were measured twice. These measurements' standard deviations were computed. These were measured independently of the chitosan content, and they were 2% for PES and 6.5% for CO/PES fabrics. Consequently, all spirits pickups for the various materials were subject to these values. The values of both materials are shown in Figure 1.

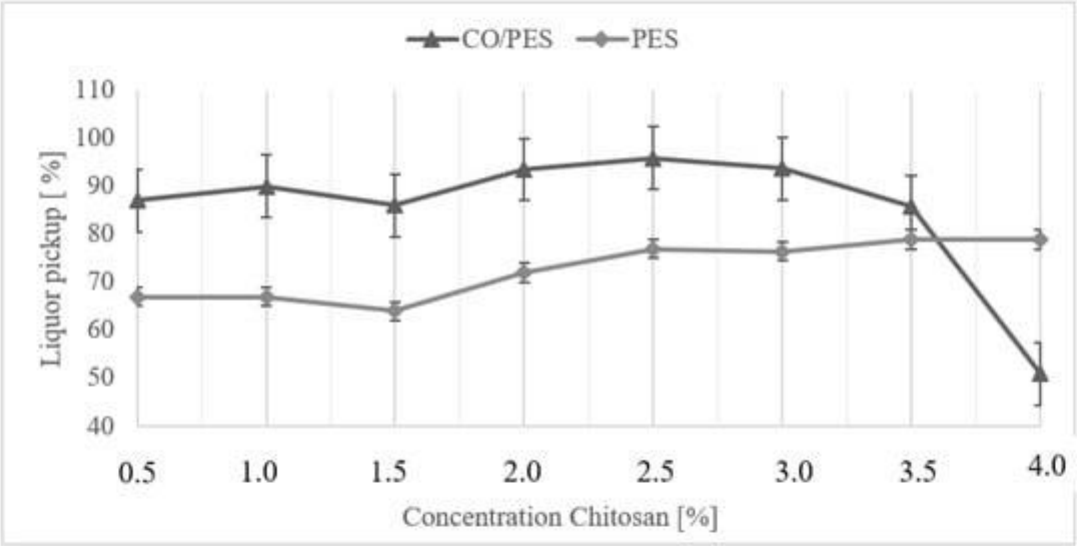


Figure 1. Pickup of polyester and cotton/polyester textiles with varying concentrations of chitosan during functionalization. For PES, the standard deviation was 2%, while for CO/PES, it was 6.5%. This picture was altered based on findings from [31].

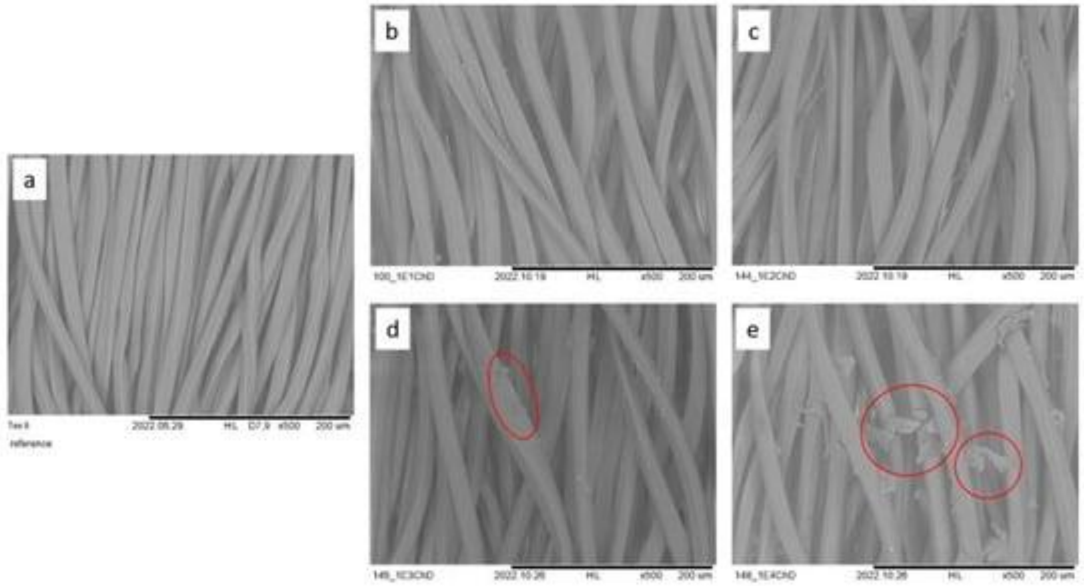
Figure 1 demonstrates the functionalized PES and CO/PES samples' spirits uptake. Because the cellulose monomer has a higher hydrophilicity than the terephthalate monomer in polyester, the CO/PES samples had a liquor pickup that was up to 20% higher. Samples with up to 3% of chitosan in both materials tended to exhibit an increase in spirits uptake as concentration increased. The CO/PES and PES have a very similar trend. The chitosan solution's viscosity grew dramatically when the concentration was raised (for example, from 91 mPa·s for 0.5% ChD to 862 mPa·s for 3% ChD).

This made it exceedingly challenging to apply a high-concentration solution by padding onto the textile, particularly for the CO/PES textile.

As a result, the samples containing 3.5% and 4% chitosan had lower liquor pickups and an uneven and unfavourable liquor application. It is noteworthy, nonetheless, that the 1.5% chitosan's degree of alcohol uptake differed for the two fabrics.

3.1.1. SEM/EDS Analysis

SEM pictures of polyester and cotton/polyester samples functionalized with varying chitosan concentrations are displayed in Figures 2 and 3.



SEM pictures of CO/PES textiles functionalized with chitosan are shown in Figure 3; the scale bar is 200 µm. (a) reference, 0% ChD, (b) 1% ChD, (c) 2% ChD, (d) 3% ChD, and (e) 4% ChD. Undissolved chitosan is indicated by red circles. Pictures are sourced from [31] with permission.

The functionalized textiles have been shown to contain chitosan by the use of SEM images. As shown in Figures 2 and 3, the initial stage involved observing the chitosan as a film on and between the fibres, including undissolved particles on the fibres and in the gaps between them. Chitosan appears as a streak-like coating on the fibres that varies in thickness and density when it dissolves. Undissolved chitosan particles are also visible in the SEM images, especially at 3% and 4% chitosan concentrations (red circles in Figures 2d,e and 3d,e indicate these concentrations).The pictures verify that chitosan is present on the PES and CO/PES textiles. Although it cannot be measured further, there is also a discernible shift in the chitosan concentration. Furthermore, it has been established that, beginning at around 3% concentrations, the applied chitosan was not entirely dissolved.

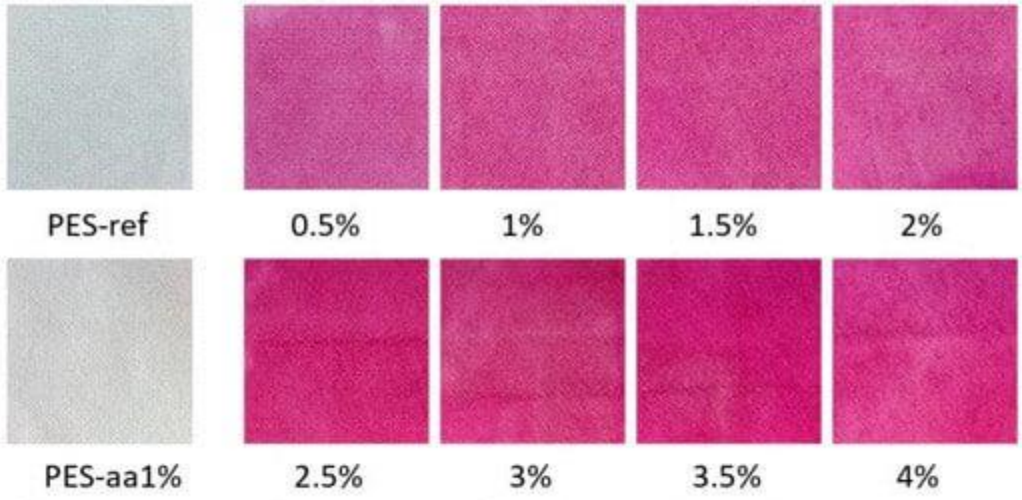
To ascertain the elements distribution on the textiles' surfaces, REM in conjunction with EDS was employed. The mean values and standard deviation (SD) are displayed in Table 2.

Nitrogen is absent from the chemical structures of cotton and polyester, whereas it is present in chitosan [15]. It was anticipated that as the chitosan concentration rose, so would the quantity of nitrogen on the textiles' surfaces. Nevertheless, nitrogen was absent from all samples, despite the fact that carbon and oxygen were discovered in varying amounts in each (Table 2). Since the EDS technique is not very sensitive to the chemical element nitrogen, it is evident that the amount of nitrogen added to the chitosan was insufficient to be detected in the samples that were created. While significant chitosan concentrations were detected subjectively using SEM analysis, these trials demonstrate that SEM/EDS analysis was not appropriate for quantitative chitosan detection, supporting the authors' earlier findings [28]. As a result, an additional analytical technique for chitosan detection on textiles was assessed.

3.1.2. Dyeing with Remazol Brilliant Red F3B

Remazol Brilliant Red F3B, a reactive dye, was used for the dying process. Due to ionic interactions, the anionic sulphonyl groups in the dye should interact with the amino groups in the chitosan [32], but not with the textile fibres directly. Images of the coloured PES (Figure 4a) and CO/PES (Figure 4b) textiles are displayed in Figure 4.

(a) polyester textile



(b) cotton/polyester textile

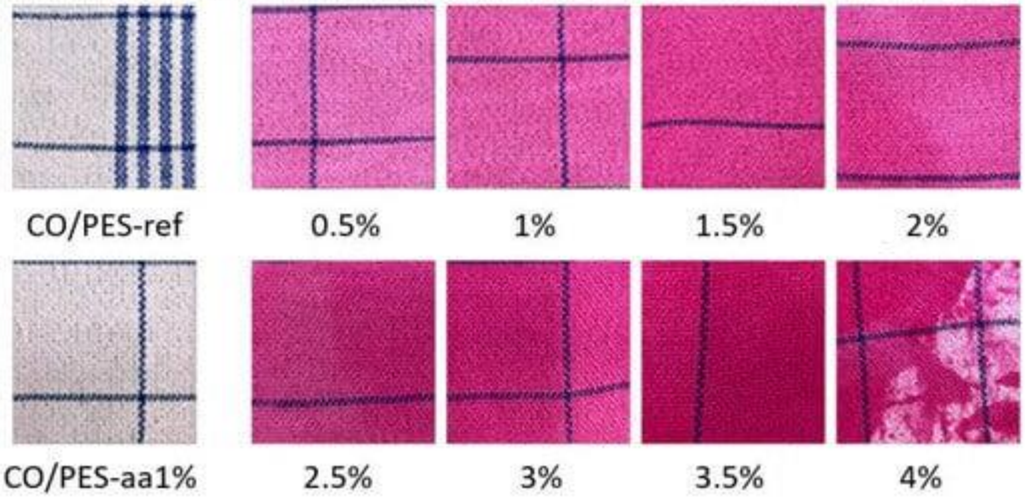


Figure 4. Different concentrations of chitosan were used to functionalize polyester (a) and cotton/polyester (b) fabrics, and Remazol Brilliant Red F3B was used to dye them. Textiles used as a reference, soaked in 1% acetic acid but devoid of chitosan. Pictures are sourced from [31] with permission.

Reactive dyes are often used to stain cotton fibres in alkaline circumstances. This process involves the interaction of the hydroxyl group in cellulose with appropriate functional dye groups [26, 27]. Because polyester fibres don't have these functional groups, they are frequently coloured at high temperatures using dispersed dyes, which cause the dye molecules to move into the amorphous regions of the polymer chain.

Figure 4 demonstrates that functionalized CO/PES fibres were not observably discoloured. For samples treated with 1% acetic acid (aa), the same holds true. Cotton fibres often have a strong affinity for reactive dyes, however in this case, the dyestuff does not attach to cotton very well due to the low dyeing temperature of just 50 °C and the short dying duration of only 5 min. The CO/PES references, on the other hand, have a faint reddish colouring that makes it possible to measure the samples more precisely and visually (please compare "ref" in Figure 5b).Pure polyester fabrics do not react well to reactive dye, as would be expected, and no discoloration was seen in this instance (see also "ref" in Figure 5a).

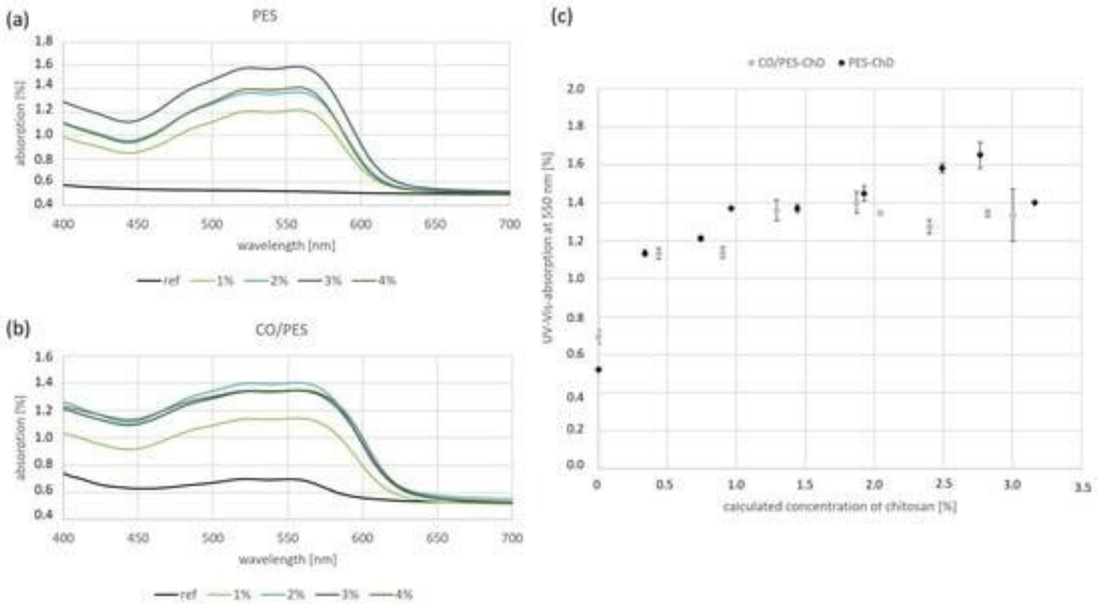


Figure 5. UV-Vis spectroscopy of samples of coloured chitosan. (a) Staining PES sample UV-Vis spectra. (b) Staining CO/PES sample UV-Vis spectrum. (c) Absorption at various chitosan concentrations at the 550 nm absorption peak. From three measurements, the mean and standard deviations are computed and shown. Image edited using information from reference [31].

All chitosan-functionalized textiles show generally very uniform staining, with the exception of the CO/PES-4% sample, which has a sizable portion that is unstaining. This is explained by the fact that not much of the highly viscous solution was used during the padding procedure. Table 3 presents a comparison between the chitosan concentration that was applied in the padding bath and the chitosan concentration that was determined utilising liquor pickup on the textiles.

The computed data show that when the bath's chitosan concentration rose, so did the textiles' chitosan content when PES was used. For CO/PES, however, this was not the case. A lot less chitosan was used on the fabrics than was anticipated because of the low spirits uptake rate of 4% ChD in this instance.The stained samples were subjected to quantitative tests using UV-Vis spectroscopy. The UV-Vis spectra of the stained fabrics are displayed in Figure 5 (Figure 5a: PES samples; Figure 5b: CO/PES samples). The values at the Remazol Brilliant Red F3B absorption maximum at

550 nm are compared for different chitosan concentrations. The concentration of chitosan on the textiles, as determined by liquor pickup (refer to Figure 1 and Table 3), is used in Figure 5c.

As the chitosan content increases, Figure 5's absorbance values for staining with Remazol Brillant Red F3B demonstrate a general rising tendency. For low chitosan concentrations, the three measurements' standard deviations were negligible. The standard deviations increased and the staining became more uneven at increasing chitosan concentrations (see also Figure 4). The maximum chitosan concentration of 4% (estimated 3.2%), however, did not produce the darkest staining for polyester. The absorption values for the samples obtained with 2.5% (calculated as 1.9% chitosan) were similar. This could be due to unequal chitosan application, as seen by the uneven staining in Figure 4 and the SEM images in Figure 2.

Despite the determination being done in triplicate, this leads to variations in the absorbance readings. The chitosan content on textiles may not rise steadily with the chitosan concentration in the finishing solution, as suggested by the variations in liquor absorption (see Figure 1). Higher chitosan concentrations do not result in an increase in colour depth when used with cotton/polyester; instead, a type of saturation happens from around 2% chitosan and up. Once more, uneven finishing is evident from Figure 4b's discoloration. The viscosity of highly concentrated chitosan solutions makes even application challenging. The SEM pictures on Figure 3 show unsolved chitosan.

The findings demonstrate that staining with Remazol Brillant Red F3B is appropriate for use in chitosan detection on both pure PES and CO/PES mixed textiles: the references were not stained, but the chitosan-functionalized samples were. The presence of primary amino groups in chitosan, which may create electrostatic interactions with dye anions in their protonated form (NH3)+ or covalent connections with reactive dyes, is responsible for the stainability of the chitosan-functionalized textiles [33].

Reactive red dyes have been used in a number of previously published investigations to stain chitosan-functionalized textiles [25, 34, 35]. These experiments used more powerful reaction conditions, with temperatures between 40 and 60 °C and reaction periods of 30 to 160 minutes, and they produced similar results. Al-Bahra had previously examined the same conditions—a mild dyeing temperature of 50 °C and a brief reaction time of 5 min—for cotton [29], but they were applied to polyester fabrics. With this technique, chitosan on cotton and polyester fabrics may be quickly and accurately detected.

When chitosan is applied to PES and CO/PES textiles using SEM/EDX, the outcomes are not up to par. Nonetheless, a reliable and quick detection technique for both materials was created during the tested dyeing process, negating the need for the textiles to be pre-treated.

3.2. Different Application Methods of Chitosan and Addition of Silanes

In a second experimental series, several application techniques were examined and chitosan-functionalized textiles with silanes as the binder were analysed. In addition to determining if the washing fastness could be enhanced, these tests sought to ascertain whether amino- and glycidoxy silanes were appropriate for altering the surface characteristics of chitosan fabrics, as previously reported for tetraethoxy silane (TEOS) [19]. An overview of the functionalization of PES and CO/PES textiles is provided in Table 4. The samples underwent analysis both immediately upon functionalization and five washing cycles later to ascertain the characteristics' washing fastness. "B" stood for the cleaned samples and "A" for the unwashed ones.

3.2.1. SEM/EDS Analysis

SEM/EDS analysis was performed on newly functionalized samples (designated with "A") and samples that had been washed five times (designated with "B"). SEM pictures of the various samples are displayed in Figure 6.

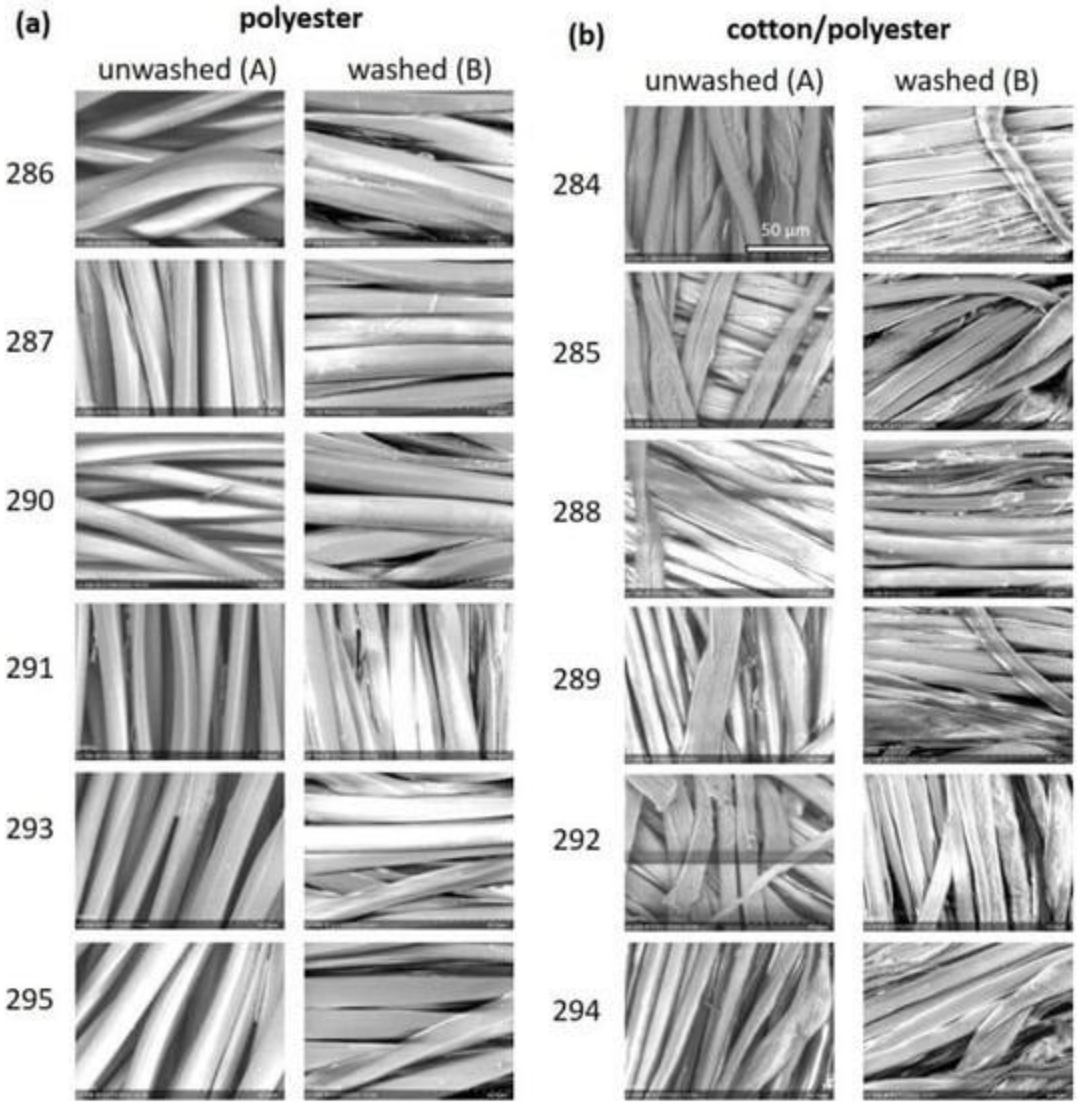


Figure 7. Samples 284B and 288B SEM images paired with orange silicon (Si) EDS mapping (scale bars = 100 µm).

The silicon was distributed uniformly throughout the samples, not just in certain places. As a result, the silane finish's EDS mapping was unable to produce any convincing proof. Overall, it was challenging to identify chitosan on the textile surfaces using EDS because functionalization only marginally altered the distributions of the chemical components carbon and oxygen, while nitrogen was undetectable (see Table 2). But silicon was applied to the surface during the functionalization process using silanes, and silicon isn't present in either chitosan or the textile materials' polymeric structure. SEM/EDS allowed for the spectroscopic identification of silicon on the textiles' surfaces, despite the fact that mapping is not a good method for doing so. This may be applied to the detection of silane functionalization. Figure 8 displays the
the respective EDS spectra.

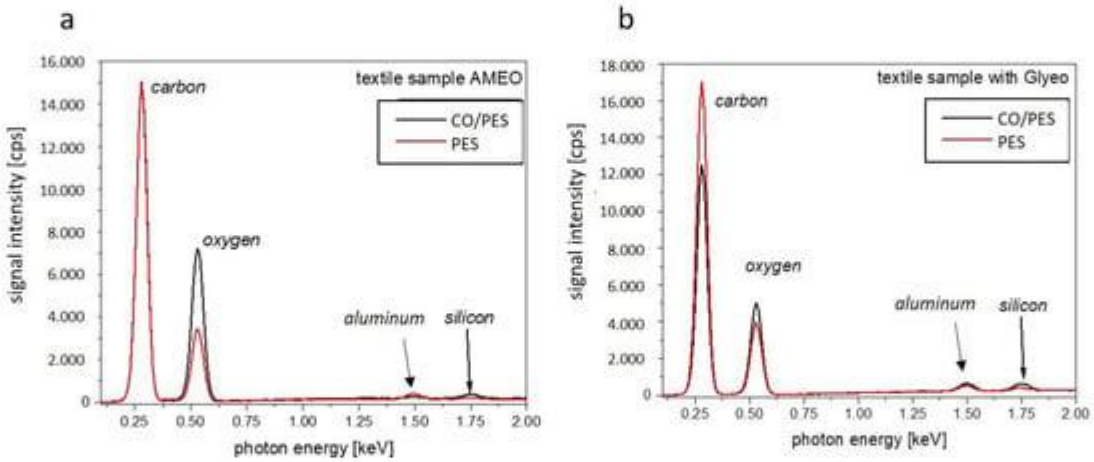


Figure 8. EDS spectra of textiles functionalized with AMEO (**a**) and with GLYEO (**b**).

The most noticeable signal is the carbon peak (0.27 keV), which is followed by the oxygen peak (0.53 keV). Cotton and polyester have varying C to O peak ratios due to their varied chemical structures (see also Table 2). The silicon signal at 1.75 keV in both spectra (Figure 8a,b) indicates the presence of GLYEO and AMEO, respectively, on the textiles. An artefact from the aluminium sample holder utilised for the EDS measurement was identified, and a peak at 1.48 eV that may be attributed to aluminium was discovered.

Figure 9 compares the silicon contents on the textiles' surfaces for the different finishes described in **Table 4**.

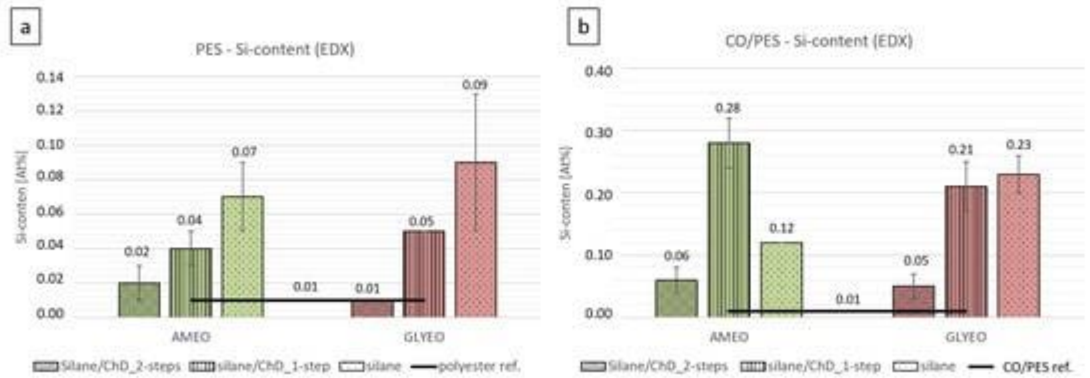


Figure 9. Contents of silicon (Si) in variously functionalized polyester (a) and cotton/polyester (b) fabrics. Three measurements' mean values and standard deviations—represented as error bars—are shown. In the absence of an error bar, the standard deviation is 0.00%.

There were noticeable variations in the PES (Figure 9a) and CO/PES (Figure 9b) values. On the polyester samples, very little silane was found, despite the fact that the silane concentrations in the finishing solutions were the same for both materials (Figure 9a).

The range of the normalised Si content was 0.02% to 0.09%. There were hardly any differences between AMEO and GLYEO in this area. The Si contents on the polyesters were between a quarter and a third lower than on cotton. This resulted from polyester's inherent hydrophobicity, which was brought on by its chemical makeup.

Consequently, it was discovered that polyester fabrics had a decreased affinity for aqueous finishing solutions, which was around 20% lower than that of cotton (see Figure 1).

Following the two-step AMEO application process, the silicone concentration on the textile surface for the cotton/polyester samples (Figure 9b) was also very low, at 0.06% (AMEO). This appeared to be caused by AMEO's decreased attraction for cotton fibres, as demonstrated by its 0.12% application when AMEO was used alone. Significantly more silicon (0.28%) was found when the one-step application method was used. Chitosan and cotton have a high chemical affinity because of their comparable structures. In order for AMEO to adsorb on chitosan, hydrogen bonds may occur between the hydroxyl groups of the hydrolyzed AMEO and the amino and hydroxyl groups on the chitosan.

Because of the chitosan's affinity for cotton, AMEO may thus be applied to the textile in a one-step process more successfully. Such adsorption was inhibited in the two-step process.The one-step method of finishing cotton/polyester with chitosan and GLYEO resulted in minimal amounts of silicon (0.05%) being found on the textile surface. Conversely, utilising the one-step method to apply GLYEO or GLYEO combined with chitosan resulted in silicon levels of 0.23% and 0.21%, respectively. Cotton fibre hydroxyl groups [36,37] or chitosan may interact with the epoxy ring of GLYEO to cause an opening and reaction. Chitosan can obstruct the silane during the two-step application procedure, rendering it unnoticeable.

The chitosan samples (296 and 297) and the textile references (298 and 299) both had very low silicon levels (0.01%), which are not included in the graph. These levels fall inside the measurement error's range.

Following washing, the silicon levels dropped and, for the majority of the samples, fell within the measurement error range. Sample 289B (GLYEO/ChD 1-step), on the other hand, nevertheless exhibited a very low silicon content (0.04%). However, sample 294B (GLYEO) consistently contained 0.23% silicon content before to washing and 0.24% silicon content following washing. This implies that silanes applied in this manner are not appropriate for use as chitosan binders. Nonetheless, there is a high affinity between the cotton/polyester fibre and GLYEO. Using SEM pictures, this cannot be visually validated due to the overall low concentration (see Figure 6).

3.2.2. Dyeing with Remazol Brilliant Red F3B

Chitosan may be identified by staining with Remazol Red, according to the observation of textiles with varying chitosan concentrations (refer to Figures 4 and 5). The second round of trials likewise employed this technique. The samples were stained, and the coloured samples underwent UV-Vis measurements. Images of fabrics that underwent various functionalization processes and were immediately dyed with Remazol Brillant Red F3B following functionalization are displayed in Figures 10 (PES) and 11 (CO/PES). Additionally displayed are pictures of fabrics that were cleaned five times prior to staining (Figures 10a and 11a). The observed UV-Vis spectra's absorption maxima at 550 nm are displayed in Figures 10b and 11b.

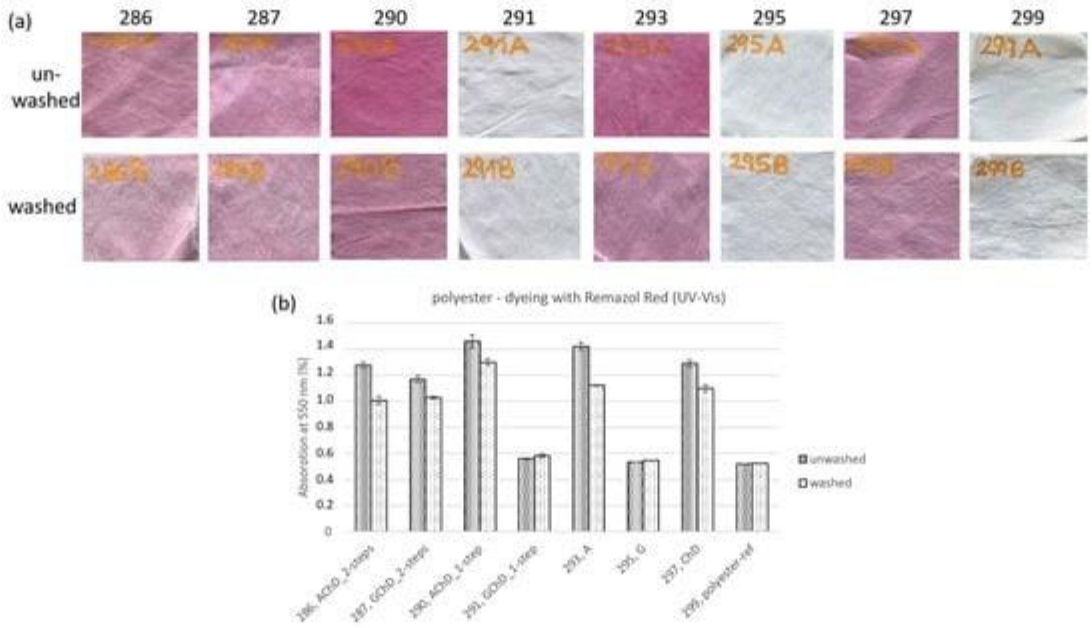


Figure 10. Polyester textiles functionalized in various ways and dyed with Remazol Brilliant Red F3B; (a) images of samples both cleaned and unwashed; (b) absorption of dyed samples at 550 nm in the UV spectrum.

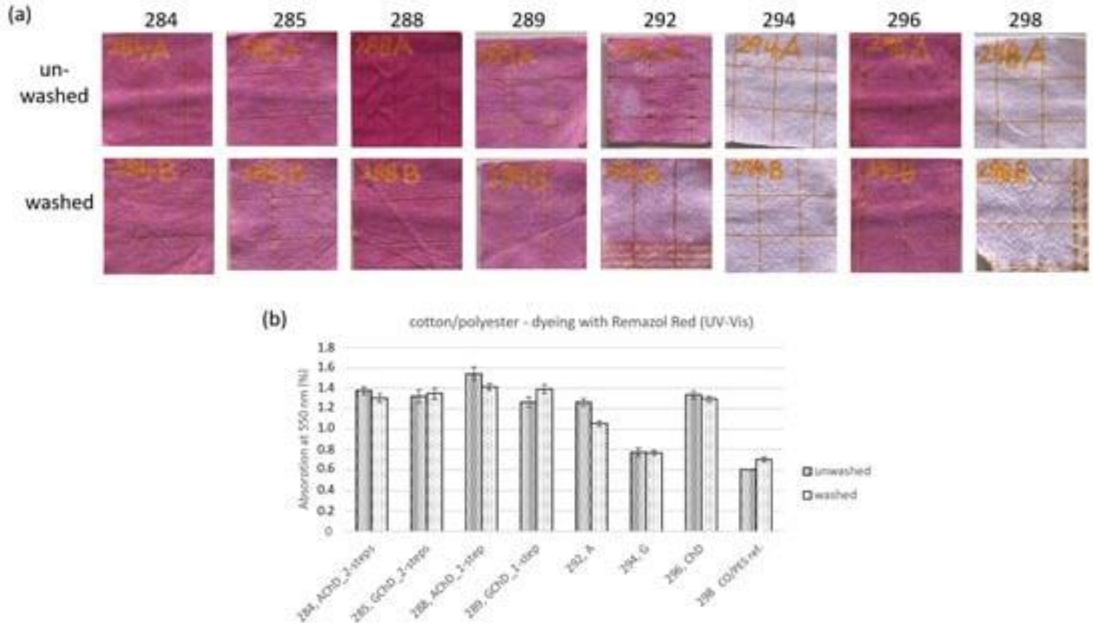


Figure 11. Fabrics made of cotton and polyester functionalized in various ways and dyed with Remazol Brilliant Red F3B; (a) images of samples both cleaned and unwashed; (b) UV-Vis absorption of samples that have been dyed at 550 nm.

In comparison to the GLYEO samples using polyester textiles, the AMEO-functionalized textiles displayed much stronger colouring (Figure 10a). The greatest dye absorption was obtained using the one-step application method using AMEO plus chitosan (samples 290) and functionalization with AMEO alone (sample 293). Sample 296 had somewhat poorer absorption than sample 297, which was finished with a single application of AMEO plus chitosan, and sample 297, which was ended with chitosan alone. This is because, according to a research by Huang [32], the free amino groups found in chitosan can react with the anionic dye molecules. It seems that AMEO, which also contains free amino groups, has a similar impact.

Only sample 287—the one that underwent the two-step application process—was discoloured after using GLYEO. Some samples had very little or no discoloration at all. The Remazol Red discoloration was less intense after washing. Samples 291–299 (Figure 10) that exhibited nearly little staining prior to washing did not vary from the other samples.

With the exception of the GLYEO-treated samples (294), and the cotton reference (298), the majority of the cotton/polyester samples (refer to Figure 11a) were strongly coloured with Remazol Brilliant Red F3B. Similar to polyester, sample 288 produced the greatest staining after applying AMEO and chitosan in a single phase. This sample also had the highest silicon concentration found on the textile surface (see Figure 9b). The staining depths of the other samples were similar. After washing, the samples' colours were just somewhat lighter. Remarkably, samples treated with chitosan alone (sample 286), chitosan with AMEO and GLYEO (sample 285), or both in two steps (sample 286) demonstrated washing stability (see Figure 11b).

The findings demonstrate that chitosan had a very high washing fastness on CO/PES fabrics and that neither GLYEO nor AMEO could further increase this property. Following washing, polyester fabrics exhibited decreased dye absorption, suggesting a decrease in the chitosan content of the material. However, this could not be made better by adding AMEO or GLYEO.

3.2.3. Water Uptake

The TEGEWA drop test was used to examine the functionalized materials' hydrophilic/hydrophobic characteristics. Here, measurements were made of the coloured water droplet's dispersal and sink-in time. Brief sink-in periods suggest that the materials are hydrophilic [19]. Table 5 presents the findings.

Figure 12 shows examples of PES samples and Figure 13 of CO/PES samples.

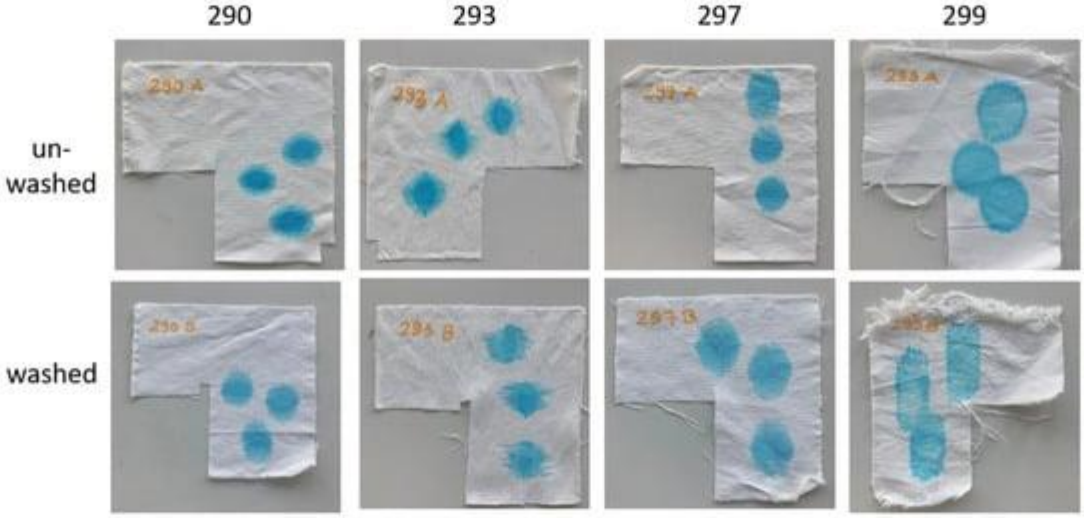


Figure 12. Water uptake on polyester samples before and after washing.

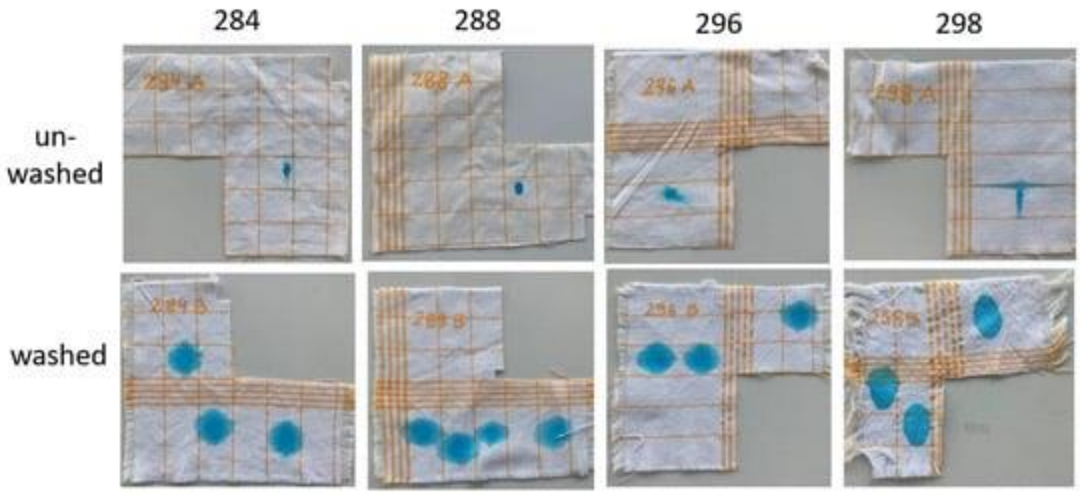


Figure 13. Water uptake on cotton/polyester samples before and after washing.

On the unwashed PES samples, the blue dye solution distributed almost evenly and in a circular pattern. The yarn density, varied by 2 yarns/cm in the warp and weft direction (see Table 1), did not alter the droplet's absorption. One to two seconds was the range of sink-in times. While all other ways of functionalization caused the textiles to become more hydrophobic, GLYEO alone or in combination with chitosan (one-step) somewhat boosted the fabric's hydrophilicity. The impacts of the functionalization were more evident when comparing the droplet spreading, even though the sink-in periods varied in very modest ranges (see Figure 12). Only little alterations in the spreading and sink-in periods were observed following washing.

The droplet dispersed much more in the warp direction than in the weft direction in the unwashed CO/PES samples. Samples 294A and 285A had sink-in periods ranging from 4 to over 24 minutes, which is an extremely lengthy duration. This outcome may have been influenced by an unidentified pretreatment and finer yarn counts in the warp direction. This impact was minimal after washing, and the spreading in the warp and weft directions was more uniform. The sink-in periods were slashed to a mere 0.5 to 1 second.

Increased hydrophilicity of functionalized fabrics was expected to result from applying GLYEO [19]. These samples, which were functionalized with GLYEO, had the highest hydrophilicity and met our expectations by having the lowest sink-in times for both cotton (sample 294A) and polyester (sample 295A). On the sink-in time, AMEO had very little influence. It is anticipated that the hydrophilicity of functionalized textiles utilising polyester would rise when chitosan products with a high DD, such as the ChD utilised in this work (DD of 90%)—as seen in Table 5—are utilised.

On the other hand, because chitosan has an acetylation group, its chemical structure is less polar than cotton's, making the CO/PES mix fabric's surface more hydrophobic. Furthermore, the use of chitosan reduces air permeability, which is regulated by porosity and affects the water absorption qualities. With respect to the CO/PES textile, the air permeability decreased to 507 ± 12 L/m2/s for the unfunctionalized material (refer to Table 1) and to 123 ± 3 L/m2/s for the PES textile, from 185 ± 9 L/s2/m. Depending on how they were applied, textiles made more hydrophobic by combining chitosan with silanes.

This result is most likely caused by the functionalization chemicals' varying affinities for the polymeric textile material, as previously mentioned. These findings highlight the various ways in which chitosan and silane combinations may be used to modify the hydrophilic and hydrophobic characteristics of textiles made of polyester and cotton. Improving the water-uptake capabilities of polyester samples ought to lower the samples' electrical resistance, which will raise the samples' antistatic qualities [19]. Soiling with dry soil is dependent on electrostatic interactions in addition to surface microstructure [38, 39]. Soiling can be decreased by lowering the electrostatic resistance. On the other hand, cotton (or cotton/polyester) textiles' rapid soiling in wet soils can be decreased by improving their hydrophobic qualities.

4. Conclusions

This work describes the functionalization of cotton/polyester and pure polyester fabrics using chitosan, AMEO, or GLYEO silanes, as well as the assessment of a reliable method for chitosan analysis. A variety of techniques were used to modify and examine the fabrics' characteristics. EDS spectroscopy did not support the SEM pictures' assertion that the fabrics' surfaces had large amounts of chitosan (3.5% or more). Remazol Brilliant Red F3B reactive dye adsorption verified the presence of chitosan on the surface. With varying chitosan concentrations, this technique worked well for differentiating between functionalized and non-functionalized fabrics.

The fabrics treated with chitosan exhibited high washing resistance, as demonstrated by the trials. Fabric hydrophilicity was enhanced by silanes without the need for chitosan, although functionalization stability was not improved. By increasing the sink-in time in the TEGEWA drop test, chitosan rendered fabrics hydrophobic. The findings of this study indicate that the hydrophilic qualities of polyester and cotton textiles may be modified by functionalization, and recipes using chitosan can be one of such methods. Future developments of novel bio-based polymer combinations with inorganic binder systems may be made through this first stage, which might ultimately result in the creation of sustainable antibacterial materials with altered hydrophilic characteristics.

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