COMPARATIVE INVESTIGATIONS ON THE EFFICIENCY OF DIFFERENT ANCHORING CHEMICALS FOR THE PERMANENT FINISHING OF COTTON WITH CHITOSAN

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Abstract:

Comparative studies on the efficiency of anchor molecules for the finishing of cotton with chitosan are presented. Four different anchors were used: cyanuric chloride (CNC), butane-1,2,3,4-tetracarboxylic acid (BTCA), glycidyloxypropyltrimethoxysilane (GPTMS) and sodium hydroxydichlorotriazine (Na-HDCT). Two types of chitosan with different molecular weights were used. The type of anchor used for fixation of chitosan on cotton fabric had a distinct effect on the antimicrobial efficiency of chitosan. By using increasing the concentration of anchor chemicals, the ability of chitosan to inhibit the growth of bacteria increased. Using GPTMS as an anchor for chitosan improved the ion binding capacity for Cu²⁺-ions more than other anchors. The highest colour strength was achieved when using a low amount of anchors from the chlorotriazine system (up to K/S 3.3), whereas carboxylic or trialkoxysilane anchors at the same ratio of anchoring chemical to glucose units achieved comparatively low K/S values. The polyelectrolyte values of treated cotton fabric were also measured. SEM was used to investigate the surface morphology of the treated cotton fabric samples.

Key words:

Chitosan, textile finishing, crosslinking, polyelectrolyte layer, antimicrobial finish.

Introduction

The biopolymer chitosan (CTS) has been used in an increasing number of applications, including a variety of areas for practical uses in pharmaceutical and biomedical engineering, paper production, textile finishing, wastewater treatment and heavy metal chelation [1–9]. The biopolymer has excellent properties for transition metal adsorption, mainly due to the presence of active amino groups bonded directly to the polymer backbone. However, its adsorbent capacity depends on the degree of deacetylation, on the metal ion and on the pH of the solution [10–12]. Another important benefit is its antimicrobial activity (due to protonation of the amino function). The permanent fixation of chitosan on a cotton surface has been investigated in many fields of application such as bioactive fibres for medicine, the creation of textiles with improved properties for wound dressing and textiles with ion binding capacities for many heavy metals [9,13]. Therefore, an important task lies in the evaluation of the methods used to anchor chitosan permanently onto fibre surfaces in a way that the chitosans will retain their beneficial bulk properties [14].

The scope of the present work is to compare chitosan anchoring strategies on cotton fabric with regard to add-on and to the properties obtained. Besides add-on, the evaluated properties are colouration effects, polyelectrolyte values on the surface and the ion binding capacity of the treated textiles.

Experimental

Materials and chemicals

Chitosans used:

– CTS-1: Chitosan(r) 85/500/A1 (Heppe, Queis, Germany), deacetylation 85 %, Mw 300 kDa.
– CTS-2: Chitosan(r) 100 (Chipro GmbH, Germany), deacetylation > 95 %, Mw 95 kDa, CTS-salt, high aqueous solubility.

Analyzing chemical and analytical reagents used:

– cyanuric chloride, CNC (Fluka),
– butane-1,2,3,4-tetracarboxylic acid, BTCA (Aldrich),
– glycidyloxypropyltrimethoxysilane, GPTMS (Aldrich),
– sodium hydroxydichlorotriazine, Na-HDCT (Taicros aqua, Evonik, Germany),
– non-ionic surfactant Marlipal(r) 013/80 (Sasol, Germany),
– polyelectrolyte standard solutions (Mütek, Germany), e.g., poly-(diallyldimethylammonium chloride) (poly-DADMAC) and poly-(ethylene sulfonic acid) sodium salt (PES-Na),
– reactive dyestuff Intracron(r) Red (Yorkshire),
– other chemicals used were of laboratory grade.

Fabric used:

– plain weave mild scoured bleached cotton fabric with 170 g/m², warp = weft, 27 cts/cm, 295 dtex (wfk, Krefeld, Germany)

Analytical equipment

– polyelectrolyte titration was performed according to the process outlined by Horn [15] and Knittel [16] using a PCD 03 pH particle charge detector (Mütek, Germany),
– colour measurement was done using the K/S apparatus (Datamaster DC3880 Data colour AG, Switzerland),
– scanning electronic microscope DS1-ABT55 (ISI Instruments),
– atomic absorption spectrometer for determination of bound metal ions using Spectra AA-800 equipment from Varian,
– nitrogen content measurements were performed on a VELP Scientifica apparatus UKA130 (Dk 20 heating Digestor) according to the Kjeldahl method.

Preparation and finishing of cellulose fabrics for permanent add-on of chitosan(s):
Chitosan was dissolved in a 1 wt% solution using acetic acid followed by pH adjustment with NaOH to the desired pH value of 6, the anchoring chemical was added while maintaining the pH value.

- CNC was at first dissolved in a minimum amount of dioxane (10 ml). Then, the chitosan solution was cooled with an ice-water mixture and the dissolved CNC was added to the cold chitosan. The fabric was immersed in this solution.
- In the case of BTCA, the fabric was immersed in an aqueous solution containing the crosslinking agent. In addition, 0.6 mole of sodium acetate trihydrate per one mole anchor was used.
- GPTMS and CTS form a clear homogeneous aqueous solution into which the samples were immersed [17].
- Similarly, a Na-HDCT-solution was added to a CTS solution at pH 6 and the fabrics were immersed.

In all cases, the textile was impregnated for 5-10 min and squeezed between rollers to a liquor uptake of 100%. Fixation was performed at 140°C for 10 min after pre-drying.

**Amount of anchoring chemicals:**

The four described protocols were conducted with 0.1, 0.2 and 0.5 moles of anchoring chemicals in a molar ratio to the monomer unit “aminoglucose” of CTS.

**Reactive dying**

Treated fabric samples were dyed with reactive dye, namely the reactive dyestuff Intracron(®) Red using the exhaustion method as follows: the dyeing solution was prepared using with final dye concentration equal to 500 mg/L and LR 1:50. The dyeing temperature was increased to 60°C. The sample was introduced in the dye bath followed by the addition of 50 g/L sodium sulphate; after 20 minutes, 5 g/L sodium carbonate was added with stirring, then after another 20 minutes 40 g/L sodium hydroxide was added and the dyeing process was continued for 50 min. At the end of the dyeing process, the samples were thoroughly washed with hot water and 0.1% Marlipal, then rinsed with cold water and finally dried at ambient conditions. Blank samples were dyed using the same recipe.

**Testing**

**Antimicrobial testing: the “shaking flask method”**

Determination of the antimicrobial activity of immobilised antimicrobial agents was done by the shaking flask test by producing an overnight culture of Escherichia coli (ca. 10^6 bacteria/ml), cutting 0.5 - 2.0 g of the treated fabric into snippets, produce of the overnight culture 1.5 - 3.30 x 10^6 bacteria/ml. Fifty millilitres of this working culture were placed in a 250 ml Erlenmeyer flask with the snippets. After closure and 1 minute of shaking, 1 ml of the solution was taken and diluted. The rest was shaken for 1 hour. Shaking was done at 37°C at 200 rpm. Afterwards, bacteria were plated out and incubated for 1 day. After 1 hour, 1 ml of the next dilution was taken out and plated for 1 day, as well. After 24 hours, the colonies which had formed were counted to calculate the difference between 1 minute and 1 hour, to calculate the bacterial growth rate.

**Ion binding experiments**

1 g of treated fabric was immersed in a solution of the corresponding metal ion (2.10^4 mol/l) e.g. copper sulphate, acidified with sulphuric acid to pH 3 - 4 (to avoid hydroxide formation) and left for two days. The ion concentration of the remaining solution was determined by atomic absorption spectroscopy.

The capacity of adsorption was characterised by calculating the ratio of removed and original Cu^2+ ions. The binding of Cu^2+ by CTS was characterised by the ratio of absorbed ions and the CTS add-on.

**Polyelectrolyte (PEL) measurement (sample conditioning)**

Polyelectrolyte titrations (PEL) were performed with a PES-Na solution. CTS treated fabrics were preconditioned in a solution at pH 4.66 prior to polyelectrolyte measurement. Fabric (1 - 2 g) was immersed in 50 ml of the oppositely charged polyelectrolyte titre (PES-Na) and stirred for 2 h. After separation of the fabric by filtration, 10 ml of the remaining solution was back-titrated with the corresponding oppositely charged polyelectrolyte standard poly-DADMAC (0.001 mol/l). The volume consumed during titration was called V_p. For calibration, 10 ml (PES-Na) was titrated against poly-DADMAC; the volume determined was called V_p. The amount of titre consumption at the endpoint of titration can be used for the calculation of accessible charges according to the following equation: milliequivalent (mequiv) = [(V_p - V_1) x 0.001]/0.2. For the correlation of the volume, which is equivalent to 1 g of cotton sample, one multiplies (V_p - V_1) by a factor of 5.

**Results and discussion**

**Basic considerations for chemical fixation of chitosan on cotton**

The strategies for chemical bond formation and thus permanent anchoring on textile are summarised. Table 1 describes the modes of anchoring chitosan to cellulosic materials (cotton, CO and regenerated cellulose, CV).

<table>
<thead>
<tr>
<th>Table 1. Interactions usable for chitosan finishes on cellulosic materials</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anchoring types</td>
</tr>
<tr>
<td>Crosslinker (durable press resins)</td>
</tr>
<tr>
<td>Ionic Interaction</td>
</tr>
<tr>
<td>Covalent Bonding</td>
</tr>
<tr>
<td>Van der Waal’s interaction</td>
</tr>
</tbody>
</table>

+ possible interaction, - weak/no interaction

Figure 1 shows the permanent anchoring of chitosan on cellulosic fibres either by crosslinking or by covalent bonding. Figures 2 and 3 give the proposed anchoring pathway for chlorotriazines and polycarboxylic acids, respectively.

Figure 1. Strategies of the permanent anchoring of chitosan on cellulosic fibres (either by crosslinking or by covalent bonding).

Cyanuric chloride (CNC) was used to anchor chitosan due to the presence of three reactive chlorine species forming bridges...
between chitosan and the fabric with opportunities for crosslinking. The principal reaction path is given in Figure 2. Sodium hydroxy-2,6-dichloro-1,3,5-triazine (Na-HDCT) is a bifunctional material, which is capable of forming a covalent bond with the hydroxyl groups of cellulose yielding a crosslinking structure in a single step.

Butane-1,2,3,4-tetracarboxylic acid (BTCA) is the most effective formaldehyde-free crosslinking agent. BTCA-treated fabrics also have desirable qualities such as a high level of smooth drying, wrinkle resistance, dimensional stability, formaldehyde-free durable-press finishing, low toxicity and recurableness [18]. Figure 3 shows that BTCA produces crosslinking in cotton through esterification of cellulosic hydroxyl groups and amide formation with the amino groups of CTS under high temperature in the presence of a catalyst.

Glycidyloxypropyltrimethoxysilane (GPTMS) seems to be a good agent for crosslinking CTS and to confer an inorganic domain, thereby maintaining smoothness [19]. The coating of textiles with CTS-GPTMS, an “organic-inorganic hybrid polymer”, is an innovative method for textile finishing, where three-dimensional networks are built of inorganic domains with increased temperature resistance (see Figure 4).

**Weight increase of treated fabrics**

In the first set of experiments, the treatment efficiencies of the different anchors are presented (for two types of chitosan) with regard to add-on achieved and to wettability (measured as drop ingression time).

Table 2 shows that in all cases one observes a moderate to strong decrease of wettability, which is probably due to the hydrophobic parts of the anchoring molecules. Similarly, an
Table 2. Add-on of CTS on cotton fabric and analyses using different anchoring chemicals.

<table>
<thead>
<tr>
<th>Sample Code</th>
<th>Anchor</th>
<th>Amount of anchoring chemical (molar ratio)</th>
<th>Add-on [wt%]</th>
<th>Nitrogen content [%]</th>
<th>Drop penetration time [s]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CO</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>&lt; 1</td>
</tr>
<tr>
<td>CTS-1-CNC1</td>
<td>CNC</td>
<td>0.1</td>
<td>0.48</td>
<td>0.17</td>
<td>20</td>
</tr>
<tr>
<td>CTS-1-CNC2</td>
<td>CNC</td>
<td>0.2</td>
<td>0.60</td>
<td>0.20</td>
<td>30</td>
</tr>
<tr>
<td>CTS-1-CNC3</td>
<td>CNC</td>
<td>0.5</td>
<td>0.80</td>
<td>0.30</td>
<td>32</td>
</tr>
<tr>
<td>CTS-2-CNC1</td>
<td>CNC</td>
<td>0.1</td>
<td>0.40</td>
<td>0.17</td>
<td>6</td>
</tr>
<tr>
<td>CTS-2-CNC2</td>
<td>CNC</td>
<td>0.2</td>
<td>0.77</td>
<td>0.20</td>
<td>8</td>
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<tr>
<td>CTS-2-CNC3</td>
<td>CNC</td>
<td>0.5</td>
<td>0.90</td>
<td>0.22</td>
<td>10</td>
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<tr>
<td>CTS-1-Na-HDCT1</td>
<td>NaHDCT</td>
<td>0.1</td>
<td>1.40</td>
<td>0.30</td>
<td>180</td>
</tr>
<tr>
<td>CTS-1-Na-HDCT2</td>
<td>NaHDCT</td>
<td>0.2</td>
<td>1.30</td>
<td>0.24</td>
<td>180</td>
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<tr>
<td>CTS-2-Na-HDCT1</td>
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<td>0.5</td>
<td>1.26</td>
<td>0.23</td>
<td>180</td>
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<tr>
<td>CTS-2-Na-HDCT2</td>
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<td>0.1</td>
<td>2.00</td>
<td>0.31</td>
<td>22</td>
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<tr>
<td>CTS-2-Na-HDCT3</td>
<td>NaHDCT</td>
<td>0.5</td>
<td>1.00</td>
<td>0.24</td>
<td>48</td>
</tr>
<tr>
<td>CTS-1-BTCA1</td>
<td>BTCA</td>
<td>0.1</td>
<td>0.80</td>
<td>0.25</td>
<td>14</td>
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<tr>
<td>CTS-1-BTCA2</td>
<td>BTCA</td>
<td>0.2</td>
<td>1.30</td>
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<td>56</td>
</tr>
<tr>
<td>CTS-1-BTCA3</td>
<td>BTCA</td>
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<td>1.80</td>
<td>0.29</td>
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<tr>
<td>CTS-2-BTCA1</td>
<td>BTCA</td>
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<td>0.40</td>
<td>0.15</td>
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<tr>
<td>CTS-2-BTCA2</td>
<td>BTCA</td>
<td>0.2</td>
<td>0.80</td>
<td>0.17</td>
<td>22</td>
</tr>
<tr>
<td>CTS-2-BTCA3</td>
<td>BTCA</td>
<td>0.5</td>
<td>1.30</td>
<td>0.25</td>
<td>49</td>
</tr>
<tr>
<td>CTS-1-GPTMS1</td>
<td>GPTMS</td>
<td>0.1</td>
<td>1.20</td>
<td>0.15</td>
<td>11</td>
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<tr>
<td>CTS-1-GPTMS2</td>
<td>GPTMS</td>
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<td>1.40</td>
<td>0.18</td>
<td>60</td>
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<tr>
<td>CTS-1-GPTMS3</td>
<td>GPTMS</td>
<td>0.5</td>
<td>1.70</td>
<td>0.24</td>
<td>120</td>
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<tr>
<td>CTS-2-GPTMS1</td>
<td>GPTMS</td>
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<td>0.35</td>
<td>0.17</td>
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<tr>
<td>CTS-2-GPTMS2</td>
<td>GPTMS</td>
<td>0.2</td>
<td>0.60</td>
<td>0.20</td>
<td>20</td>
</tr>
<tr>
<td>CTS-2-GPTMS3</td>
<td>GPTMS</td>
<td>0.5</td>
<td>0.90</td>
<td>0.22</td>
<td>42</td>
</tr>
</tbody>
</table>

*Values of amount of anchor are in molar ratios to glucan monomer unit.

increase in the ratio of anchor to aminoglucan units diminished wettability. This effect was more pronounced when the CTS of high molecular weight was used. Best wettability was obtained when using CTS 2 with CNC or GPTMS because of high degree of deacetylation of CTS 2 which means an increase in the available amino groups as well as a uniform network formed in the case of GPTMS, producing a thin film of chitosan. It is also indicated in Table 2 that using Na-HDCT as an anchor for chitosan resulted in higher add-on as well as higher nitrogen content (up to 2.3 add-on, 0.4 N% content), which affects in a direct way the affinity of treated cotton fabric to dyeing, as was evident from the results of the K/S data as seen later. In all cases, use of high molecular weight CTS resulted in higher weight gain.

In all cases, the increase in drop ingress time compared to blank CO can be explained by the influence of residual acetyl groups on CTS and by the hydrophobic character of the anchoring molecules. Figure 5 shows SEM micrographs of blank fabric and chitosan treated fabric anchored with different crosslinkers.

All treatments show good film forming behaviour; the smoothest fibre surface was observed using CTS1 rather than CTS2. GPTMS as an anchoring chemical led to better film forming as the surface was coated by Si-O-Si network incorporating CTS. Cracks on the surface of the fibres, like those seen with Na-HDCT, could be a direct consequence of the extent of crosslinking.

**Antimicrobial efficiency**

Figure 6 shows the effect of the concentration of one anchor on the antimicrobial properties of chitosan finished fabrics. The shaken flask data reveal that an increasing amount of anchor, regardless of the type of anchor, increased the effect of chitosan CTS 1 in inhibiting the growth of bacteria. For the low molecular weight chitosan CTS 2, the results revealed that no significant antimicrobial effect was noted from the finished samples when the amount of anchor was below 0.5, and the rate of inhibition reached only 25% at that amount. The amount of anchor needed to be tailored to achieve the best bacteriostatic effect desired for chitosan, and this differed from one chitosan type to another. The results also show that chitosan imparts antimicrobial activity to cotton fabric even when tested under pH 6 and low add-on (< 3 %), and so it could be concluded that the antimicrobial activity of finished fabrics increases with the molecular weight of chitosan and as a result of increasing chain length. It is also shown that even if there is a distinct difference in grafting yield on cotton using different anchors, the antimicrobial suppression seems to be similar.

In a recent work, Tayel et al. demonstrated that dissolved CTS (of fungal origin) had increasing biostatic potential with decreasing CTS molecular weight [20]. According to the present experiments, covalently bonded CTS shows a controversial dependence on molecular weight.

**Ion binding capacity of chitosan finished cotton fabric**

The ability of chitosan for metal ion adsorption is closely related to its principal characteristic: rather high hydrophilicity owing to a large number of hydroxyl and primary amino groups that represent activity as adsorption sites.

The complexing main sites are the amines and secondary alcohol functional groups, since the nitrogen of the amino group and the oxygen of the alcohol have a pair of electrons that can add themselves to a proton or a cation by coordinated covalent bonds. The attraction of the electron pair by the atom nucleus is stronger for oxygen, while on the other hand nitrogen has a greater tendency to donate its pair of electrons to metal ions to form a complex through a coordinated covalent bond. The complexes between metal ions (M⁺) and chitosan are formed according to the mechanism illustrated in Figure 7.

In this mechanism, the metal ions (M⁺) with empty orbitals act as Lewis acids capable of accepting electron pairs. In contrast, the NH₂- and OH-groups that have non-shared electron pairs

act as Lewis bases donating their electron pairs. This behaviour depends on the pH of the solution. If the adsorption system is in a neutral or slightly acid pH, the mechanism shown below is predominant [3,9,21].

As seen in Table 3 and Figure 8, in the case of CTS 1, the highest adsorption was achieved using GPTMS as the anchor. This may be due to more a porous structure of the finish. With the exception of Na-HDCT, there was no effect of anchor concentration. For Na-HDCT, increasing the concentration to 0.5 resulted in a breakdown in adsorption capacity. It should also be mentioned that the matter of porosity of the finish in the case of each crosslinker will be extensively discussed in future work.

As shown in Table 3 and Figure 8 for all kinds of anchors, an increased amount of anchor increases the adsorption capacity (except in the case of CNC and Na-HCDT). This notable decrease in adsorption capacity can be attributed to the blocking of pores. The binding capacity of CTS treated fabrics is on the order of commercial ion exchangers. As has been previously found by Hebeish et al. [23], crosslinked chitosan (by glutaraldehyde) can adsorb copper ions from water and a Cu$^{2+}$ adsorption up to 85% can be achieved.

GPTMS as the anchoring chemical yielded the highest specific Cu$^{2+}$ ion adsorption with nearly the same results for both types of chitosan. This indicates that CTS/GPTMS yields a more flexible structure of the polymeric chitosan chain, which allows for configuration adjustment. Cu$^{2+}$ and better film forming properties were also observed.

High adsorption capacity could be seen using CTS/GPTMS or CTS/BTCA. In comparing the two types of chitosan, it was found that the use of the low molecular weight CTS 2 was more applicable for complexation of the heavy metal ion Cu$^{2+}$. This may be attributed to the availability of free amino groups compared with CTS 1 and to the increased molecular weight of CTS 1. This results in increased network viscosity and in decreased accessibility to internal sites.

The treated samples were dyed using the reactive dye Intracon Red (Figure 9) as a probe for the dyeability of the various CTS treated fabrics.

The colouring effects demonstrate the accessibility of the CTS layers on the textile substrate. Using a triazine ring-containing crosslinker, any ratio of anchor to glucose unit greater than 0.1 reduced reactive dyeing. This may be due to blocking of the amino groups through reactions with chlorine atoms in the triazine moieties; this also may explain the decrease in K/S “dyeability” when the amount of fixed chitosan is increased. Other linking systems show slight improvement in dyeing when surpassing this limit.

As shown in Table 3, increasing add-on as well as the nitrogen content increased K/S values, especially when using BTCA as an anchor for both type of chitosans (the K/S value reached 2.8 in the case of CTS 1/BTCA and was slightly less than 1.58 in the case of CTS 2/BTCA).
Chitosan/BTCA treated fabric was observed to be more hydrophilic than chitosan/CNC and chitosan/GPTMS treated fabrics. It is assumed that an amide bond is formed between chitosan leading to improved dyeability. The highest value for $K/S$ in reactive dyeing was achieved when using a low molar ratio of Na-HDCT as the crosslinker.

Polyelectrolyte measurement (PELT)

Polyelectrolyte measurements made on CTS treated fabrics (Table 3) can be used to assess the availability of ionic charges on the fabric surface which provides information on statistical anchor sites and the possible chain segment mobility of the biopolymer on fibres. Since the probing molecules of a PELT determination are rather high molecular weight polymers, the information depth of PELT titration can be expected to be very much smaller than the information given by diffusing metal ions. It is assumed that polymer chain segment mobility within the outermost surface layer determines any functionality [8].

The charge accessibility increased considerably by using less of the anchoring chemicals, meaning better functionality of the CTS molecules. Table 3 also indicates that the type of anchor affects the value of PELT in both cases of CTS. The highest values of PELT were achieved with CTS/BTCA and CTS/GPTMS.

Conclusion

The objective of this work was to evaluate the effect of different anchor chemicals for the fixation of chitosan on cotton fabrics (cyanuric chloride, Na-hydroxydichlorotriazine, butane tetracarboxylic acid, glycidyloxypropyltrimethoxysilane) on specific properties of the textile finish. Both the type and concentration of anchor chemicals significantly affected the performance properties, antimicrobial properties, ion binding capacity as well as the polyelectrolyte value of the treated cotton fabric.

When discussing the efficiency of anchor chemicals, one has to consider efficiency per gram of treated fabric vs. the efficiency of usage of anchored biopolymer molecules. Usually, a low add-on results in efficient use of CTS molecules, whereas total binding capacity increases with increasing add-on.

The choice of anchor type strongly depends on the property of interest. Regarding add-on, CNC was rather ineffective whereas the other three crosslinkers gave comparatively high results. Polyelectrolyte values and ion binding capacities were best when using BTCA or GPTMS.

It was found that the wettability of treated cotton fabric samples was highly affected by the hydrophobic part of the anchor molecules. The type of anchor used for fixation of chitosan on cotton fabric showed little effect on the efficiency of chitosan as an antimicrobial agent, while an increasing concentration of anchor chemical increased the ability of chitosan to inhibit the growth of bacteria.

The highest colour strength was achieved when using a low amount of anchors from the chlorotriazine system (up to $K/S$...
3.3), whereas carboxylic or trialkoxysilane anchors at the same ratio of anchoring chemical to glucane units gave comparatively low values of K/S. The PELT values of the treated cotton fabric with CTS was highly affected by the different anchor chemicals

Regarding the types of chitosan used, the water-soluble type CTS 2 (higher degree of deacetylation) was more effective for the abovementioned properties when related to the amount of CTS fixed.

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