Humidity sensors are important for assessment of environmental monitoring and industrial applications. Various kinds of humidity sensors, based on different materials and sensing principles, have been developed. In the present paper, cotton fabrics were impregnated using finishing baths containing different alkoxysilanes: tetraethoxysilane (T+EOS), (3-aminopropyl)triethoxysilane (APTES) and (3-triethoxysilylpropyl)succinic acid anhydride (TESP-SA) in combination with cobalt chloride hexahydrate as humidity indicating substance. The rates of color change of the cobalt chloride-doped fabrics were evaluated by measuring the change of the K/S values at three relative humidity (r.H.) levels (12%, 55% and 86%). The modified specimens were characterized by means of SEM, FT-IR and TGA. The physical-technical properties were assessed (air permeability, flexural rigidity, tensile strength, abrasion resistance). The findings make evident that the sol-gel modification results in a decrease in the rates of color change. The APTES-treated specimen showed no change of color.

**Keywords**: cotton, sol-gel, humidity sensor, organic-inorganic hybrid, cobalt chloride hexahydrate

**INTRODUCTION**

A smart textile is defined as a material that can react to stimuli exerted from the environment. The stimulus may derive from physical or chemical sources. Therefore, the structure of a smart textile must contain a sensor, which is able to sense the stimulus, as well as an actuator, which will respond to the stimulus.¹⁴

The concept of sol-gel chemistry can also be employed to convert textile substrates into smart materials. This technique involves the mixing of precursors in an aqueous or alcoholic medium. Precursors are molecules that contain a central metal or semimetal atom, to which reactive alkoxy groups and/or organic groups are bonded. These reactive groups are subjected to an acidic or alkaline catalyzed hydrolysis and condensation reaction, thus forming a sol and subsequently a gel. Aging, drying and curing procedures enable the production of powders, xerogels, aerogels, fibers, or coatings. The latter renders possible the surface modification of textiles, thus imparting novel properties to the material. Alkoxysilanes represent an important class of organic compounds and have the general formula \((RO)_nSiR_{(4-n)}\) (where \(n = 1, 2, \text{ or } 3\)).⁵⁻⁷ They are excellent chemicals in order to modify the surface of various materials. The alkoxy group RO usually consists of a methoxy or ethoxy group, which can readily be converted to a silanol group via a hydrolysis reaction. The organofunctional R group determines the physical and chemical properties of the modified surface.

A great number of studies describe the modification of cellulosic substrates by means of the sol-gel technology. The mechanical properties of cotton-based materials, such as abrasion resistance and crease resistance, were improved.⁸⁻¹⁰ The sol-gel coating technique also enables the enhancement of the thermal stability and flame retardancy of cellulosic textiles¹¹ Coating with organotrialkoxysilanes endows hydrophilic cotton fabrics with superhydrophobic properties.¹²,¹³ Excellent results could also be achieved in terms of UV-blocking.¹⁴,¹⁵

Humidity sensing is very important, since the amount of water vapor in the air can affect human
comfort as well as many manufacturing processes in industries. Humidity measurement can be conducted using hygrometers (dry and wet bulb hygrometers, dew point hygrometers, and electronic hygrometers). There has been a surge in the demand of electronic hygrometers, often called humidity sensors. Various approaches have been accomplished to study cobalt-based humidity sensors.

The goal of the present study was to consider the application of cotton-bound cobalt(II) chloride as humidity sensor. For this purpose, cotton fabrics were modified with hydrolyzed alkoxysilanes in conjunction with cobalt(II) chloride. The rate of the color change of the as-prepared cotton samples was evaluated at various humidity levels.

EXPERIMENTAL

Materials

Tetraethoxysilane (98%, TEOS) was supplied from Fluka Chemie GmbH, Buchs, Switzerland. 3-Triethoxysilylpropyl)succinic acid anhydride (TESP-SA, 100%) and (3-aminopropyl)triethoxysilane (100%, APTES) were obtained from Wacker Silicone, Burghausen, Germany. Cobalt(II) chloride hexahydrate (>99%, CC), lithium chloride (<99%), calcium (II) nitrate tetrahydrate (<99%), and potassium chloride (<99%) were purchased from Carl Roth GmbH, Karlsruhe, Germany. The chemicals were applied without further purification. Deionized water (DI) was used throughout the investigation. Desized, scoured, bleached and mercerized 100% cotton fabric (weave type: plain, density warp: 52 yarn/cm, weft: 24 yarn/cm, weight: 109 g/m²) was utilized throughout the study.

Preparation of the cotton samples

One-step approach

For the one-step procedure, the treatment solutions were prepared as follows: 30 mmol and 60 mmol, respectively, of each of the alkoxysilanes (TEOS, TESP-SA, APTES) were hydrolyzed in 15 ml DI and 5 ml HCl (c = 0.1 mol/L) at room temperature for 15 h under stirring. The beaker was sealed with a pierced parafilm. Subsequently, the volume of the homogenous solutions was filled up to 50 mL with DI. Then, the pre-weighed cotton samples were thermally treated as described above. Subsequently, the specimens were dipped into a CC solution (1.60 g/100 mL) at room temperature for 12 hours, removed from the beaker and dried at 105 °C for 2 min (Fig. 1).

Two-step approach

For the two-step approach, the alkoxide solutions were prepared as follows: 30 mmol and 60 mmol, respectively, of each of the alkoxysilanes (TEOS, TESP-SA, APTES) were hydrolyzed in 15 ml DI and 5 ml HCl (c = 0.1 mol/L) at room temperature for 15 h under stirring. The beaker was sealed with a pierced parafilm. Subsequently, the volume of the homogenous solutions was filled up to 50 mL with DI. Then, the pre-weighed cotton samples were thermally treated as described above. Subsequently, the specimens were dipped into a CC solution (1.60 g/100 mL) at room temperature for 140 °C for 20 min according to the scheme shown in Figure 1.

Methods

Preparation of saturated salt solutions

Selected known r.H. levels in a closed vessel can be obtained by means of saturated salt solutions. To maintain a constant r.H. in the desiccator, three saturated salt solutions were prepared in a crystallizing dish and transferred to the desiccator: 12% r.H. (lithium chloride: 90 g), 55% r.H. (calcium (II) nitrate hydrate: 70 g), 86% r.H. (potassium chloride: 40 g). The corresponding amount of salt was added to 100 g DI at 50 °C. Then, the solution was allowed to cool down to RT.

Measurement of color change kinetics

The CC-containing cotton samples were cut into small pieces (2 cm x 5 cm) and dried at 105 °C for 20 min. Subsequently, the samples were transferred into the desiccator containing the corresponding humidity solution. After a specified period, one sample was removed and the K/S spectrum was recorded.

Mechanical properties

The stiffness of the fabrics was determined by means of a Taber Stiffness Tester, Model 112, New York, USA according to ASTM D 1388-R6. The flexural rigidity was calculated as described in ASTM D 1388-R6. The measurement of the air permeability was conducted according to DIN EN ISO 9237 using the air permeability tester Textest FX 3030 LDM, Textest Ltd., Zurich, Switzerland. The air flux (L/dm²/min) that passes through the fabric was evaluated at a pressure difference of 100 Pa. Abrasion tests were performed according to DIN EN ISO 12947 (Martindale method): the circular sample was mounted on a specimen holder and subjected to a defined load (12 kpa). Then, it was rubbed against a standard fabric in a translational movement.

Analytical methods

Colorimetric data measurements were conducted with a Spectrophotometer CM-3610d from Konica Minolta, Japan. The color data software CM-S100w
Spectra Magic NX V1.9 was used for data acquisition. FT-IR/ATR spectra were recorded with a Bruker Vector 22 spectrometer using a DTGS detector. The spectra were the result of 50 scans. The spectral resolution was 4 cm\(^{-1}\). A PIKE MIRacle TM ATR accessory equipped with a diamond ATR crystal was used. SEM micrographs were recorded with an electron micro probe analyzer – JEOL JSM-6010LV. An acceleration voltage of 15kV was used. The fibers were sputter-coated with a layer of gold. TG measurements were conducted with a Linseis STA PT1000 thermogravimetric analyzer (heating rate 10 °C/min; scan range 40-900 °C) in air.

Figure 1: Scheme of preparation of alkoxysilane/CC-modified cotton samples

RESULTS AND DISCUSSION

General

In the course of the present study, cotton fabrics were treated with nanosol solutions consisting of a hydrolyzed alkoxysilane (TEOS, TESP-SA, and APTES) in combination with CC. The concentration of the alkoxysilane was 30 mmol/100 mL and 60 mmol/100 mL, respectively. The cotton fabrics were modified in a one-step and two-step procedure, as described in the experimental section. The samples prepared in the course of this study are denoted as follows: RM: raw material; CC: cobalt(II) chloride; 1st: one-step procedure; 2st: two-step procedure; the number indicates the mole of the alkoxysilane. Therefore, the term TEOS-30-CC-1st refers to the cotton sample that was impregnated with a 100 mL formulation consisting of 30 mmol TEOS and cobalt(II) chloride. The term TEOS-30-CC-2st refers to the cellulosic specimen that was first impregnated with a 100 mL formulation containing 30 mmol TEOS, and subsequently dried and cured. In the second step, the as-prepared sample was treated with a solution containing cobalt(II) chloride and dried again.

Rate of color change at various relative humidities

For a better understanding regarding the determination of the rate of color change, the following topics will be discussed: theory of color change of CC, pseudo-first order mechanism, and relationship between concentration of the dye and the reflectance.

Theory of color change

Cobalt is a transition metal that is capable of forming a huge variety of stable, colored metal complexes, most of them with octahedral structure. The anhydrous, blue-colored cobalt(II) chloride changes the color in response to humidity, thus forming the hydrated, pink-colored hexaaquacobalt(II) ion. The reverse process can be obtained by thermal treatment. These phenomena can be explained by d-d orbital electron transition of cobalt(II) and have been verified by means of NIR/UV-Vis absorption spectroscopy in solution.\(^{23-26}\) Diffuse reflectance spectra were also recorded to describe the absorption properties of various solid cobalt(II)
The results make evident that the anhydrous, blue-colored cobalt(II) complex shows three absorption bands in the visible spectral region of 500-700 nm. The hydration of the cobalt(II) compound is accompanied with a decrease of the absorption peaks, which can be observed at 660 nm. In the present study, this decrease was measured as a function of time. The as-obtained data were used to determine the kinetic parameters of the color change on the assumption that the reaction obeys a pseudo-first order mechanism.

**Pseudo-first order mechanism**

Since one component (water) is present in excess, it can be assumed that the concentration of water maintains unchanged during the reaction and therefore only the concentration of cobalt(II) chloride anhydrous decreases exponentially. Thus, the reaction is pseudo-first order with respect to CC.

Kinetically, it obeys the following relationship:

\[ \frac{d[CC]}{dt} = k[CC] \]  

(1)

The integration of this rate equation (Eq. 1) leads to the following expression:

\[ \ln \frac{[CC]}{[CC]_0} = kt \]  

(2)

The value of the reaction rate constant \( k \) can be calculated from the slope of the linear graph: \( [CC] = [CC]_0 e^{-kt} \)

where \([CC]_0 = \) initial concentration of TESP-SA, \( t = 0; [CC] = \) concentration of TESP-SA as reaction proceeds; \( k = \) pseudo-first order rate constant the dimension (unit) is \( \text{[t}^{-1}] \).

The Kubelka-Munk function, which can be derived from diffuse reflectance spectra by Equation 3, is directly correlated with the colorant concentration of a substrate, where \( K \) is the absorption coefficient, \( S \) is the scattering coefficient, and \( R \) is the reflectance factor at a specific wavelength. The Kubelka-Munk theory assumes that the ratio of absorption and scattering coefficients of the colorant is directly proportional to its concentration and is additive in a mixture.

\[ \frac{K}{S} = \frac{(1-R)^3}{2R} \]  

(3)

Therefore, it can be concluded that the portion of the species involved in producing the color on the CC-modified cotton directly correlates with the K/S values.

**Determination of kinetic parameters**

The color change was determined by measuring the reflectance of the dried, anhydrous blue-colored samples during certain time intervals (0-60 min) at various r.H. levels (12%, 55% and 86%). The spectra were recorded at intervals of 5 min for a period of 30 min, and then the intervals were increased to 10 min during the next 30 min. The K/S spectra that were recorded at r.H. 12% are shown in Figure 2a-d: RM-CC (Fig. 2a), TEOS-60-CC-1st (Fig. 2b), TESP-SA-60-CC-1st (Fig. 2c), and APTES-60-CC-1st (Fig. 2d). The diffuse reflectance spectra of the other alkoxysilane-modified cotton samples were all nearly identical and for this reason only the diffuse reflectance spectra of the above mentioned cotton samples are presented. In the spectra of RM-CC (Fig. 2a), TEOS-60-CC-1st (Fig. 2b), and TESP-SA-60-CC-1st (Fig. 2c), three bands at 510 nm, 620 nm and 660 nm can be observed, which are due to d-d electron transitions of anhydrous, blue-colored CC. The K/S values of the bands at 620 nm and 660 nm significantly decrease with increasing exposure times. This phenomenon could also be detected visually. The color turned from blue to pink. Almost no reflectance could be measured in the case of APTES-60-CC-1st, as can be seen in Figure 2d.

Figure 2e shows the depletion of the K/S values for the band at 660 nm in correlation to the exposure time. The curves begin to flatten out after 30 min. In an attempt to evaluate the pseudo-first order rate constant of the color change according to Eq. 2, the data thus obtained were subjected to a linear regression analysis. The values included in the flat part of the curves were excluded. The graphs of the linear regression analysis are depicted in Figure 2f.

Identical measurements and evaluations were performed at 55% r.H. and at 86% r.H. The corresponding graphs are not shown, since the curves displayed an almost identical shape. The values of the coefficient of determination (R²) of the linear regression analysis were calculated and found to be in the range of 0.9218-0.9972.

Figure 3 represents the rate of color change of all CC-modified cotton samples at r.H. of 12%, 55% and 86%. Compared to RM-CC, the findings make evident that the treatment of the cotton fabrics with alkoxysilanes generally results in a reduction of the rate of the color change. The application of the one-step approach or the two-step approach does not give rise to a significant difference.
Figure 2: K/S spectra recorded at r.H. 12\%: RM-CC (2a), TEOS-60-CC-1st (2b), TESP-SA-60-CC-1st (2c), and APTES-60-CC-1st (2d); Decrease in absorbance at 660 nm as a function of time (2e) and graphs of regression analysis (2f)

Figure 3: Rates of color change of all alkoxysilane/CC-modified cotton samples at r.H. 12\%, 55\% and 86\%

A doubling of the concentration of the alkoxysilanes brings about an increase in the rate of color change. The incorporation of APTES results in no color change, irrespectively of the r.H. and exposure time. This phenomenon is explained by the fact that the cobalt ion is coordinated by the amino groups of APTES, thus being incapable of forming an aqua complex.

Physical-mechanical properties of the fabrics
The surfaces of the cotton fabrics have been modified applying different nanosol solutions comprising various alkoxyisilanes (TEOS, TESP-SA, and APTES). The thermal treatment of the nanosol-impregnated cotton samples resulted in an alteration of the physical-mechanical properties of the specimens. Therefore, the modified cotton samples were evaluated in terms of air permeability, flexural rigidity, and tensile strength. The results are shown in Table 1. The findings of the abrasion tests are presented in Figure 4.

Air permeability is an important parameter in the performance of various textile materials, such as clothing and technical textiles. Air permeability is defined as the volume of air in liters that is passed in one minute through 1 dm² of fabric at a pressure difference of 100 Pa. It is governed by various parameters, such as fabric porosity, yarn diameter, and inter-yarn pores, which can be modified by means of chemical finishing.

The findings in Table 1 give evidence that air permeability was increased when the cotton sample was immersed into a TEOS-containing solution, indicating that the interstices had been increased. Since the architecture of the fabric was not changed by the chemical modification of the fabric, it can be assumed that TEOS increased the void volume of the cotton fabric by changing e.g. the capillary structure of the yarns.

The flexural rigidity is an indicator for the stiffness of a textile fabric and it can be interpreted as the resistance of a fabric to bending. Stiffness characteristics of fabrics are influenced by the structure of the fabric as well as by the structure of the constituent yarns. In terms of a good drapability, fabrics with a high flexural rigidity are not preferred for apparel and garment fabrics. However, in the case of industrial applications, the stiffness of a fabric is an important factor. An inspection of the values of the flexural rigidity of the fabrics presented in Table 1 reflects that those cotton samples that were treated with TEOS show the highest resistance to bending. Compared to the RM, a significant decrease of the flexural rigidity can be observed for the cotton sample that was finished with TESP-SA.

The tensile strength is defined as the resistance of a material to longitudinal stress, measured by the minimum amount of longitudinal stress required to rupture the material. The data shown in Table 1 confirm that the tensile strength values of all modified cotton samples are significantly reduced.

Abrasion occurs when a textile surface is subjected to a rubbing process. As a consequence, mass loss can be observed, which may result in the destruction of the fabric. Technical textiles should exhibit excellent abrasion resistance. Figure 4 shows the abrasion curves of the nanosol-modified cotton samples, presenting the mass loss as a function of the abrasion cycles. The findings clearly show that the specimens that were treated with APTES solutions exhibit the best abrasion resistance. The values of the mass loss increased when the cotton samples were finished with TEOS solutions. The TESP-SA treated fabrics exhibit the lowest abrasion resistance. Due to the four hydroxyl groups of TEOS and the organic functionalities of TESP-SA (carboxyl group), crosslinking reactions can occur.

![Figure 4: Abrasion curves of APTES-, TEOS- and TESP-SA-treated cotton samples](image-url)
Table 1
Physical-mechanical properties of cotton samples thermally treated with various alkoxy silane/CC solutions

<table>
<thead>
<tr>
<th>Code</th>
<th>Air permeability, %</th>
<th>Flexural rigidity, %</th>
<th>Tensile strength, %</th>
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<tr>
<td>RM</td>
<td>100.0</td>
<td>100.0</td>
<td>100.0</td>
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<tr>
<td>RM-CC</td>
<td>93.6</td>
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<td>62.1</td>
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<td>77.8</td>
</tr>
<tr>
<td>APTES-60</td>
<td>88.6</td>
<td>404.9</td>
<td>57.2</td>
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<tr>
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<td>333.0</td>
<td>79.6</td>
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<tr>
<td>TEOS-60</td>
<td>171.4</td>
<td>664.4</td>
<td>57.6</td>
</tr>
<tr>
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Consequently, the mobility of the cellulose chains is reduced, resulting in a decrease of the abrasion performance. Since the amino-functionalized APTES is not capable of the formation of additional crosslinks, it can be concluded that the treatment of the cotton fabric with APTES does not significantly affect the abrasion properties.

**Surface morphology**

**SEM**

SEM images have been recorded to study the morphology of the surface of the treated cotton fabrics. Figure 5 shows the SEM images of RM-CC (Fig. 5a), APTES-60-CC-1st (Fig. 5b), TEOS-60-CC-1st (Fig. 5c), and TESP-SA-60-CC-1st (Fig. 5d), indicating that in all four cases a smooth surface is obtained. This phenomenon gives evidence that the nanosol solution has penetrated into the fiber pores.

**FT-IR**

FT-IR/ATR is used to investigate the surface of materials to gain more structural information by analyzing the various vibration modes of the functional groups involved. As a consequence, the FT-IR/ATR spectra of RM-CC (Fig. 6a), APTES-60-CC-1st (Fig. 6b), TEOS-60-CC-1st (Fig. 6c), and TESP-SA-60-CC-1st (Fig. 6d) were recorded and are shown in Figure 6. The absorption bands observed in the spectral region from 1500 cm\(^{-1}\) to 750 cm\(^{-1}\) can be ascribed to the vibration mode of the siloxane units (Si-O-Si) and to the asymmetric bridge mode of the cellulose (C-O-C). The absorption bands at 1427 cm\(^{-1}\) (C-H wagging), 1363 cm\(^{-1}\) (C-H bending) and at 1317 cm\(^{-1}\) (C-H wagging) also stem from the cotton material.

Interesting results can be derived from the absorption bands that are visible in the region from 1500 cm\(^{-1}\) to 1750 cm\(^{-1}\). The peak at 1643 cm\(^{-1}\), which appears in all four spectra (Fig. 6a-d), is due to the OH stretching mode of adsorbed water. Spectrum 6b (APTES-60-CC-1st) shows a prominent absorption band at 1593 cm\(^{-1}\). This vibration mode can be assigned to the N-H deformation mode of the amine group. The band at 1712 cm\(^{-1}\), which can be seen in spectrum 6d (TESP-SA-60-CC-1st), corresponds to the carboxyl carbonyl stretching vibration, proving
the presence of the carboxylic group in the TESP-SA-modified cotton sample.

The absorption band at 2893 cm\(^{-1}\) corresponds to the symmetric stretching mode of the methylene groups. The absorption bands of the hydroxyl groups are presented in Figure 6a-d. The broad band between 3000 cm\(^{-1}\) and 3500 cm\(^{-1}\) is associated with the stretching vibrations of the hydrogen bonds. Those bands with wavenumbers greater than 3400 cm\(^{-1}\) are assigned to intramolecular hydrogen bonds, whereas those at lower wavenumbers than 3400 cm\(^{-1}\) are due to intermolecular hydrogen bonds.\(^\text{38}\) Thus, the shoulder at 3443 cm\(^{-1}\) confirms the presence of the intramolecular hydrogen bonds, whereas the absorption bands at 3328 cm\(^{-1}\) and 3278 cm\(^{-1}\) are due to intermolecular hydrogen bonds.

Figure 5: SEM images of cotton samples: RM-CC (5a), APTES-60-CC-1st (5b), TEOS-60-CC-1st (5c) and TESP-SA-60-CC-1st (5d)

Figure 6: FT-IR/ATR spectra of RM-CC (6a), APTES-60-CC-1st (6b), TEOS-60-CC-1st (6c) and TESP-SA-60-CC-1st (6d)
Thermogravimetric analysis

Thermogravimetric analysis is an excellent technique to obtain information in terms of the thermal stability of materials. The thermal degradation of a cellulosic material is very complex. It is assumed that four processes are involved, when cellulose is decomposed (dehydration and break-down of the cellulose molecule, formation of levoglucosan, decomposition of the dehydrated products to form tarry residues, and decomposition of levoglucosan). Thus, Figure 7 presents the thermograms of RM-CC (TG: Fig. 7a, DTG: Fig. 7e), TEOS-60-CC-1st (TG: Fig. 7b, DTG: Fig. 7f), APTES-60-CC-1st (TG: Fig. 7c, DTG: Fig. 7g), and TESP-SA-60-CC-1st (TG: Fig. 7d, DTG: Fig. 7h). An inspection of the TG curves makes evident that the decomposition rates are remarkably increased at temperatures >300 °C. At 600 °C, significant differences in terms of the char yields can be observed. The results indicate that the highest values for the char yield were obtained for the cotton samples that were finished with an alkoxysilane bearing a functional group (APTES: -NH₂, and TESP-SA: -COOH). The corresponding DTG curves reveal that the degradation rates increase in the following order: RM-CC > TEOS-60-CC-1st > APTES-60-CC-1st > TESP-SA-60-CC-1st.

CONCLUSION

Cotton fabrics were treated using finishing baths containing different hydrolyzed alkoxysilanes: tetraethoxysilane (TEOS), (3-aminopropyl)triethoxysilane (APTES) and (3-triethoxysilylpropyl)-succinic acid anhydride (TESP-SA) in combination with cobalt chloride. A one-step procedure or a two-step procedure was applied. The rates of color change of the cobalt-chloride dotted fabrics were evaluated by measuring the change of the K/S values (660 nm) at three r.H. levels (10%, 55% and 86%) during a specified time period. No change of color could be observed for the APTES-treated specimen. A comparison to RM-CC reveals that the thermal treatment of the cellulosic material with alkoxysilanes results in a decrease in the rates of color change. The modified specimens were characterized by means of SEM, FT-IR, and TGA. The results of the physical-technical tests (air permeability, flexural rigidity, tensile strength, abrasion resistance) prove that the air permeability and the flexural rigidity were increased, when the samples were treated with TEOS. The tensile strength was reduced in all cases. The APTES-finished sample possesses the best resistance to abrasion.

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