

IMPARTING MULTI-FUNCTIONAL PROPERTIES TO COTTON FABRIC BY MEANS OF SOL GEL PROCESS

Noureddine Abidi

Eric Hequet

Luis Cabrales

Fiber and Biopolymer Research Institute, Department of Plant and Soil Science

Texas Tech University

Lubbock TX

Abstract

In this study, lightweight 100% cotton fabric was successfully modified by the sol-gel process to impart high Ultraviolet Radiation (UVR) scattering property to the fabric surface. Active ingredient was tetraethyl orthotitanate ($\text{Ti}(\text{OCH}_2\text{CH}_3)_4$). The cotton fabric was padded with the nanosol solution, dried at 60°C , and cured at 150°C . Scanning Electron Microscopy showed continuous and uniform film on the fiber surface. Excellent UVR scattering was obtained with all treated fabrics. Increasing titanium content in the nanosol lead to increased UVR protection. This is attributed to the increase of the refractive index of the film formed on the fabric surface. Excellent durability of the treatment was obtained, which could be attributed to the establishment of covalent linkages between the $-\text{OH}$ groups of the cellulose and $-\text{OH}$ groups on the $\text{Ti}-\text{O}-\text{Ti}$ network. Cotton fabric functionalized with titania nanosol exhibited very good self-cleaning properties when exposed to UV radiation.

In addition, cotton fabric was successfully functionalized with vinyltrimethoxysilane in order to impart water repellency and wrinkle recovery and to introduce onto the fabric surface vinyl groups ($-\text{CH}=\text{CH}_2$) that could be easily initiated for copolymerization reaction with various monomers. The introduction of active groups on the fabric surface was evidenced from the Universal Attenuated Total Reflectance FT-IR (UATR-FTIR) spectrum of the treated fabric. The spectrum shows two peaks located at 1410 cm^{-1} and 1600 cm^{-1} ($\text{C}=\text{C}$ stretch). Additional peak located at 756 cm^{-1} attributed to $\text{Si}-\text{O}-\text{Si}$ symmetric stretch was also observed.

Introduction

The ultraviolet radiation (UVR) is composed of three types: UV-A (315 nm to 400 nm), UV-B (290 nm to 315 nm) and UV-C (100 nm to 290 nm). The UV-C radiation is absorbed by the ozone layer, however, the UV-A and UV-B reach the earth surface and cause serious health problems such as skin cancer, sunburn, and photo-aging (Reinert et al. 1997 ; Hilfike et al. 1996 ; Crews et al. 1999 ; Srinivasan and Gatewood, 2000 ; Eckhardt and Rohwer, 2000 ; Zhou and Crews 1998). Therefore, special attention has been focused recently on the ultraviolet transmission of textiles because of the growing demand in the marketplace for lightweight apparel that offers protection from UVR, while fostering comfort. Modifying fabrics to reduce the UVR transmission to the wearer is a relatively new application. To quantify the protection from the UVR, the term SPF (Sun Protection Factor) is widely used for cosmetic sunscreens (Hilfike et al. 1996 ; Xin et al. 2004). However, for fabrics the use of the term UPF (Ultraviolet Protection Factor) is preferred (Xin et al. 2004). This factor is based on an in-vitro measurement and is defined as the ratio of the average effective UVR calculated for an unprotected skin to the average UVR irradiance calculated for a skin protected by the fabric (Xin et al. 2004). Effective UVR irradiance is defined as the product of the relative erythral spectral effectiveness by the relative energy value of the solar spectral irradiance reaching the skin (Xin et al. 2004).

Textile surface modification provides a way to impart new and diverse properties to textiles while retaining comfort and mechanical strength. Chemical compounds containing silicon-oxygen bonds (such as polydimethylsiloxane) are used in the textile industry as finishing agents: antistatic, antisoil, anticrease (Tsukda et al., 2001). The presence of siloxane bonds ($\text{Si}-\text{O}$) imparts interesting properties such as: improved thermal stability, resistance to oxidation, retention of physical properties over a wide range of temperature, water repellency, and active surface properties, due to the high flexibility of the $\text{Si}-\text{O}$ bond (Tsukda et al., 2001).

Materials and Methods

The 100% cotton fabric used in this study was desized, scoured, and bleached. The fabric characteristics were: 79.4 ends, 65.4 picks, yarn count of 23.4×22 tex, and a weight of 161.98 g.m^{-2} (4.8 oz.yd^{-2}). The chemicals used to

prepare the sol, tetraethyl orthotitanate ($\text{Ti}(\text{OCH}_2\text{CH}_3)_4$), ethanol ($\text{C}_2\text{H}_5\text{OH}$), acetic acid (CH_3COOH), hydrochloric acid (HCl) (37.7%) were purchased from Fisher Scientific (Houston, Tx). Vinyltrimethoxysilane 97% ($\text{CH}_2=\text{CH}-\text{Si}(\text{OCH}_3)_3$) was purchased from Sigma-Aldrich Co. (Milwaukee, Wi). All chemical reagents were used as received.

Cotton fabric samples were dipped into the titania nanosol, soaked for 5 min, and passed through a two-roller laboratory padder (BTM 6-20-190) at a speed of $4 \text{ yd} \cdot \text{min}^{-1}$ and an air pressure of $2.76 \times 10^5 \text{ Pa}$. The padded fabric samples were then dried at 60°C for 10 min by passing through a Ben Dry-Cure Thermosol Oven (IT500 with 18-in working width) at $0.3 \text{ yd} \cdot \text{min}^{-1}$ to evaporate the solvent (ethanol) and then cured in the same oven at 150°C for 5 min at a speed of $0.7 \text{ yd} \cdot \text{min}^{-1}$.

Results and Discussion

Cotton fabric functionalized with titania nanosols

Analysis of variance shows a significant effect of the amount of the tetraethyl orthotitanate in the solution on the fabric percent add-on after rinsing and drying. The increase in add-on results in a significant increase in the ultraviolet protection of the fabric. There is 300% increase in the UPF of the fabric after treatment with a solution containing 1 ml of tetraethyl orthotitanate. After treatment with a solution containing 4 ml of tetraethyl orthotitanate, the UPF increases by 760% and the fabric provides excellent protection from the UV radiation (UPF ratings 50+). In this case, the amount of the UV radiations passing through the fabric is reduced to less than 1.6%. The relationship between the UPF and the amount of tetraethyl orthotitanate is: $\text{UPF} = -1.7945 * ([\text{Ti}(\text{OCH}_2\text{CH}_3)_4])^2 + 21.328 * ([\text{Ti}(\text{OCH}_2\text{CH}_3)_4]) + 8.6953$, $R^2 = 0.99$.

The durability of the treatment to repeated home laundering was evaluated by performing 40 washing-drying cycles according to the standard test method AATCC 124. There is an unexpected and significant increase of the UPF during laundering cycles (the UPF increased by 270% after 40 laundering cycles).

Self-cleaning cotton fabric

Figures 1-a and 1-b show the SEM micrographs of the control fabric (no treatment) and the fabric treated with titania nanosol. Titania dioxide coating on the fabric surface could be seen on the treated sample (at $\times 100$ magnification). Higher magnification ($\times 3,000$) of the treated fabric shows deposition of thin film of titania nanosol on the fiber surface.

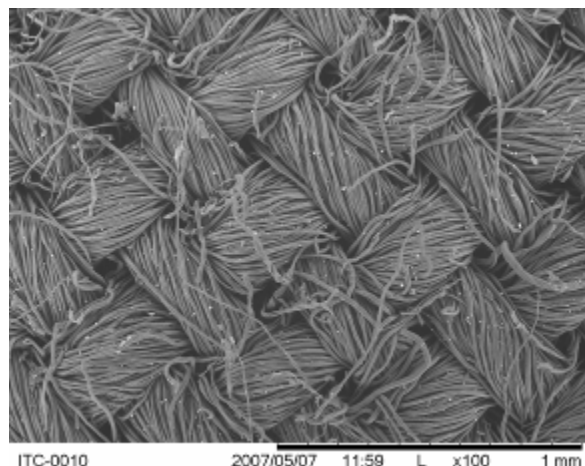


Figure 1-a: SEM of untreated fabric.

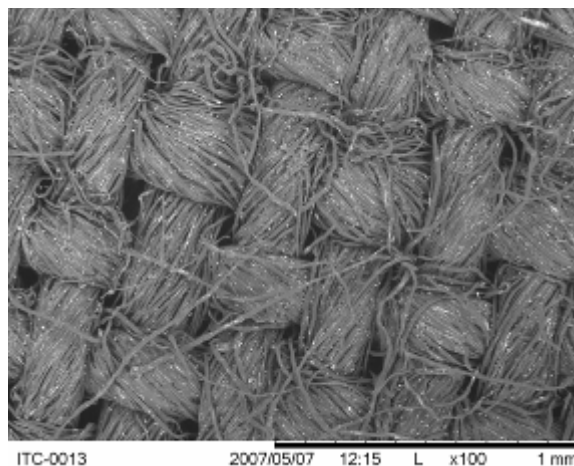


Figure 1-b: SEM of titania nanosol treated fabric.

To test the efficiency of stain removal, the fabrics treated with titania nanosol were stained with coffee and red wine. The stained fabrics were then exposed to UV light for different periods of time.

As shown in figure 2, exposure of coffee-stained fabric (treated with titania nanosol) to UV radiations for 28 h leads to the decomposition of the organic stain. The ΔE_{cmc} decreased from 33.4 to 0.6 and GS passed from 2 to 5. ΔE_{cmc}

of 0.6 and GS of 5 mean that the human eye can not see a difference between white unstained cotton fabric and the treated stained fabric and exposed to UV radiations.



Figure 2: Comparison between cotton fabric coated with titania nanosols before exposure to UV radiations (left, $\Delta E_{\text{cmc}} = 33.4$, $\text{GS} = 2.0$), cotton fabric treated with titania nanosols stained with concentrated coffee and exposed to UV radiations for 28 h (center, $\Delta E_{\text{cmc}} = 0.6$, $\text{GS} = 5.0$), and control white fabric with no treatment and no stain (right).

Figure 3 shows a control fabric (no treatment) stained with red wine. After exposure to UV radiations during 28 hours, the $\Delta E_{\text{cmc}} = 8.0$ and $\text{GS} = 3.5$. However, the fabric treated with titania nanosol showed a better level of fading with a $\Delta E_{\text{cmc}} = 1.6$ and a $\text{GS} = 5$. At this level of ΔE_{cmc} and GS , the human eye can not see a difference between the white control fabric and the titania nanosol treated fabric exposed to UV radiations. This behavior is attributed to the photocatalytic activities of the titania film deposited on the fabric surface. It should be pointed out that, these fabrics have also a very good UPF ($\text{UPF} > 50$).

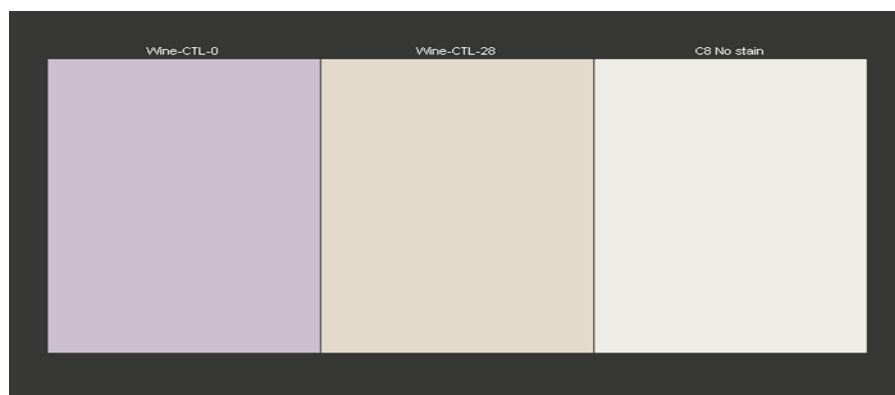


Figure 3: Comparison between control fabric stained with red wine before exposure to UV radiations (left, $\Delta E_{cmc} = 16.1$, $GS = 2.0$), stained with red wine after exposure to UV radiations for 28 h (center, $\Delta E_{cmc} = 8.0$, $GS = 3.5$), and control white fabric with no treatment and no stain (right).

Cotton fabric functionalized with VTMS

The statistical analysis of the results shows significant effect of the VTMS concentration on the percent weight increase. The relationship between the amount of VTMS cross-linked to the cotton fabric (%weight increase) and the VTMS concentration in the solution is: %weight increase = $0.013 + 4.371 * [(CH_3-O)_3Si-CH=CH_2] - 0.308 * [(CH_3-O)_3Si-CH=CH_2]^2$, Adjusted $R^2 = 0.967$, $F(2,6) = 118.222$, $p < 0.001$, Standard Error of Estimate = 0.8596. The non-linear relationship is attributed to the unavailability of cellulosic OH groups for cross-linking with the OH groups of the VTMS at higher concentration (saturation phenomenon).

To assess the hydrophobicity of the VTMS-functionalized cotton fabric, the water contact angles were measured. The coefficient of Variation (CV%) for contact angle measurements was comprised between 2.6% and 4.5%. The statistical analysis showed a significant effect of the VTMS concentration on the water contact angle. The untreated bleached cotton fabric surface was hydrophilic (contact angle = 0). At low VTMS concentration ($< 1.114 \text{ mol.l}^{-1}$), the treated fabric remained hydrophilic. Then, the increase of the VTMS concentration leads to significant increase of the contact angle and the cotton fabric surface becomes hydrophobic (contact angle $> 100^\circ$).

The statistical analysis showed significant effect of the increase in VTMS concentration on the wrinkle recovery angle. The treatment of the cotton fabric with a solution containing 3.603 mol.l^{-1} of VTMS increased the wrinkle recovery angle by 80%. This means that the tendency of the functionalized cotton fabric to recover from a deformation is higher than for the untreated fabric, thus, producing a wrinkle free fabric.

Acknowledgment

The authors would like to thank the Texas Department of Agriculture, Food and Fibers Research Grant Program for providing the financial support for this project.

References

- Crews, P.C., S. Kachman, and Beyer, A.G. AATCC Review 1999, 31(6), 17.
- Eckhardt, C. and H. Rohwer. Textile Chemist and Colorist & American Dyestuff Reporter 2000, 32(4), 21.
- Hilfiker, R., W. Kaufmann, G. Reinert, and E. Schmidt. Textile Res J 1996, 66(2), 61.
- Reinert, G., F. Fuso, R. Hilfiker, and E. Schmidt. AATCC Review 1997, 29(12) 36.
- Srinivasan, M., and B.M. Gatewood. Textile Chemist and Colorist & American Dyestuff Reporter 2000, 32(4), 36.
- Tsukda, M., T. Arai, S. Winkler, G. Freddi, and H. Ishikaw. J Appl Polym Sci 2001, 79, 1764
- Xin, J.H., Daoud, W.A., and Y.Y. Kong. Textile Res J 2004, 74(2), 97.
- Zhou, Y., and P.C. Crews. AATCC Review 1998, 30(11), 19.