FTIR Analysis of Crosslinked Cotton Fabric Using a ZnSe–Universal Attenuated Total Reflectance

Noureddine Abidi,[1] Eric Hequet,[1] Chris Turner,[2] Hamed Sari-Sarraf[2]

[1]*International Textile Center, Texas Tech University, Lubbock, Texas 79409-5019*
[2]*Department of Electrical and Computer Engineering, Texas Tech University, Lubbock, Texas 79409-3102*

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ABSTRACT: Two cotton fabrics were treated with increasing amounts of a textile-finishing agent (1,3-dimethyl-4,5-dihydroxy-2-imidazolidinone) to impart durable press properties. The Universal Attenuated Total Reflectance Fourier Transform Infrared (UATR–FTIR) with a ZnSe–Diamond composite crystal was used to determine the amount of the crosslinking agent effectively linked to the cellulose after the required laundering cycles. Textile performance testing conducted on treated and untreated fabrics demonstrated the effectiveness of the treatment applied. The results obtained showed very good correlation between AATCC grading, automatic image analysis of fabric smoothness, textile performance testing, and the amount of finish as evaluated by the UATR–FTIR. The ZnSe–Diamond composite FTIR accessory was proven to be a fast and precise nondestructive technique to evaluate the amount of the crosslinking agent linked to the cellulose macromolecules. © 2005 Wiley Periodicals, Inc. J Appl Polym Sci 96: 392–399, 2005

Key words: crosslinking; FT-IR; fibers; fabric wrinkle; resin

INTRODUCTION

Cotton is made up of cellulose macromolecules with repeating anhydroglucose units. On each unit, there are three available hydroxyl groups. These hydroxyl groups serve as sites for water molecules absorption by establishing many hydrogen bonds with the cellulose macromolecules. A severe limitation of fabrics made from cellulosic fibers is their tendency to wrinkle. In general, wrinkles occur when the fiber is bent. In this process, hydrogen bonds between the cellulose macromolecules in the amorphous regions of the fibers break, thus allowing the chains to slip past one another. The hydrogen bonds then reform in new places and hold creases in the fiber and fabric. The basic idea behind the resistance of cotton fabric to wrinkles is to restrict the slippage of cellulose chains.[1] Appropriate chemical treatment of cotton fabric enables the establishment of covalent links between the cellulosic chains in the amorphous regions of the fibers, thus restricting the slippage of the cellulosic chains. For many years, the textile industry has been using *N*-methylol–based products with very low formaldehyde release as the crosslinking agent. The most common method for finishing the cotton fabric is the pad–dry–cure process.[2–5] This method consists of impregnating the sample in an aqueous solution containing the crosslinking agent and the appropriate catalyst, padding the impregnated fabric to 90–100% wet pick-up, drying, and then curing.

Durable press–referred to as smoothness–is a term used for apparel that requires little or no ironing after home laundering and has wrinkle resistance properties during daily wear. These garments are becoming a prominent consumer item. The protocol for ascertaining a smoothness grade of a fabric is outlined in the American Association of Textile Chemists and Colorists (AATCC) Test Method (TM) 124.[6] This standard test is designed to evaluate the smoothness of fabric specimens after five cycles of repeated home laundering. Once three specimens per fabric have been through five standard washing–drying cycles, three technicians visually evaluate their appearance. For these evaluations, the specimen is laid on a solid surface that stands at an incline of 5° from vertical under specified lighting conditions. The specimen is then compared to six standard replicas, which are 3-D plastic models, showing varying degrees of smoothness and having grades 1 (very wrinkly), 2, 3, 3.5, 4, and 5 (very smooth). The specimen is assigned the grade of the replica it most closely resembles.

Infrared spectroscopy has been used to confirm the effectiveness of the reactions between the crosslinking agent and the OH groups of the cellulose polymer. Morris et al.[7] used near infrared for quantitative determination of polycarboxylic acids on cotton fabrics. The method used requires grinding cotton fabric in a Wiley mill to pass a 20-mesh screen and pressing about 1.8 mg of cotton fabric with 350 mg of KBr

Correspondence to: N. Abidi (n.abidi@ttu.edu).

(potassium bromide). The carbonyl absorbance around 1700–1750 cm^{-1} was used to quantify the amount of polycarboxylic acids on cotton fabric. Wei and Yang[2] used infrared spectroscopy as a tool for predicting the performance of durable-press–finished cotton fabric. The method used for preparing the sample for infrared measurements required grinding the cotton fabric into powder using a Wiley mill to improve the sample uniformity. These methods are destructive, labor intensive, and require a skilled operator to get satisfactory results.

In this work, we have used the Universal Attenuated Total Reflectance–Fourier Transform Infrared (UATR–FTIR) with a ZnSe–diamond crystal to assess the chemical finishing of cotton fabrics. The advantage of the UATR–FTIR technique is that no sample preparation is required, it is not a destructive method (the measurements are performed directly on the fabric), and it does not require a skilled operator. The textile performance tests were conducted on treated cotton fabrics and the results were correlated to the amount of the finish (as measured with UATR–FTIR) that is effectively present after the required five laundering cycles. In addition, we have correlated the AATCC grades, the automatic image analysis of fabric smoothness, and the conditioned wrinkle recovery angles of the treated fabrics to the corresponding FTIR measurements.

EXPERIMENTAL

Materials

Two desized, scoured, and bleached 100% cotton fabrics were used throughout this study. They were manufactured at the International Textile Center, Texas Tech University. The two fabrics are identified herein as C1 and C2. The characteristics of fabric C1 were 100 ends, 85 picks, yarn count of 16.4 × 14.8 tex (36 × 40 English count), and a weight of 118.7 g/m^2 (3.5 oz/yd^2). The characteristics of fabric C2 were 40 ends, 56 picks, yarn count of 59 × 59 tex (10 × 10 English count), and a weight of 230.56 g/m^2 (6.8 oz/yd^2).

The crosslinking agent used was 1,3-dimethyl-4,5-dihydroxy-2-imidazolidinone. It is a glycoxal based textile-finishing resin known as Dimethylureaglyoxal (DMUG) ($C_5H_{10}N_2O_3$) with Ultra Low Formaldehyde content. A magnesium chloride ($MgCl_2$) solution (trade name catalyst 531) was used to catalyze the crosslinking reaction. Both DMUG and Catalyst 531 were purchased from OMNOVA Solutions (Chester, SC) and used as received.

Fabric treatment

The fabric specimen (52 cm × 52 cm) was immersed in an aqueous bath treatment containing x% of DMUG, x/4% of catalyst 531, and 1% of wet aid (Tergitol). All of the concentrations are expressed as a percent weight of the bath. The concentration, x, of the crosslink agent was varied between 1 and 20%, with a 1% increment from 0 to 12%, then 15 and 20%. The impregnated fabric then was passed through a two-roller laboratory padder (BTM 6–20-190) at a fabric speed of 4 yards/min and an air pressure of 2.76×10^5 Pa. The weight pick-up was in the range of 90–106% for C1 and 96–119% for C2. The sample was dried in a Benz Dry-Cure Thermosol oven (IT500 with 45.72 cm, 18-in, working width) at 100°C for 190 s. Finally, the fabric was cured in the same oven at 150°C for 90 s. For both fabrics and for each % of DMUG, three specimens were treated. Two replications were performed totaling 180 fabric specimens (2 fabrics × 2 replications × 3 specimens × 15 treatments).

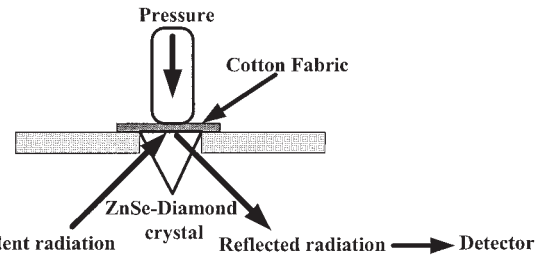

Figure 1 Principle of the Universal Attenuated Total Reflectance Fourier Transform Infra-red (UATR–FTIR).

Fabric evaluation

UATR–FTIR measurements. Perkin–Elmer Spectrum-One spectrometer equipped with a UATR–FTIR accessory was used to record the FTIR spectra of the control and the treated fabrics. The UATR–FTIR consists of a composite ZnSe–diamond crystal that allows collection of the FTIR spectra directly from the sample without any special preparation. It is an internal reflection accessory that is used with Spectrum-One for simplifying the analysis. The midinfrared detector is deuterated tri-glycine sulfate (DTGS). The cotton fabric samples were placed on top of the ZnSe–diamond crystal. Pressure was applied on the sample to ensure a good contact between the sample and the incident IR beam, preventing loss of the IR incident radiation (Fig. 1). The IR spectra were collected at a spectrum resolution of 4 cm^{-1}, 32 scans, over the range of 4000 to 650 cm^{-1}. A background scan of the clean ZnSe–diamond crystal with no sample and no pressure was acquired before acquiring the spectra of the sample. The FTIR measurements were performed on each specimen after five successive washing and tumble-drying cycles (9 readings per specimen × 3 specimens for each concentration = 27 FTIR spectra for each concentration). The objective was to evaluate the quantity of

DMUG that is effectively crosslinked to the cellulose chains.

Smoothness appearance evaluation. The standard test for smoothness appearance was performed according to AATCC TM 124.[6] This test method consists of five subsequent laundering and tumble-drying cycles. The treated fabrics were stitched to prevent unraveling and washed as directed in the AATCC TM 124. Laundering was conducted at a wash temperature of 41 ± 1°C for 10 min, with 66 ± 0.1 g of AATCC standard detergent without optical brighteners. Tumble-drying was set for durable press conditions (30 min). At the end of the washing cycles, the individual samples were placed on a perforated screen for conditioning at 65 ± 2% RH and 21 ± 1°C for 24 h, as directed by ASTM D 1776 (Standard Practice for Conditioning and Testing Textiles). Three trained observers, using AATCC standard replicas, performed the smoothness appearance grading (also referred as Durable Press rating). All AATCC grading was performed before the FTIR measurements and the textile performance tests.

Fabric smoothness appearance assessment using automatic imaging system. In addition to the standard test described above, an objective evaluation of the fabric smoothness was performed using an automatic imaging system. The detailed description of the system was reported in previous work.[8–11] This system is based on a smart CMOS camera combined with a laser-line projector that is capable of acquiring high-resolution range images of the fabric specimens. Customized image analysis algorithms utilize topographical analysis techniques to locate the wrinkles. Localized features of small cross-sectional profiles of those wrinkles then are extracted at each edge point. The individual wrinkle measurements result in a highly detailed quantitative description of the fabric wrinkles. Among the attributes extracted from analyzed images, we selected three attributes: the total number of edge points (which is the sum of all edge points localized in the region of interest), the average profile height (which is a simple arithmetic average of the wrinkle height), and the surface area (which is the total surface area of the 3-D surface of the 8 in. × 8 in. region of interest).

Wrinkle recovery angle measurement. The wrinkle recovery angle (WRA) was measured according to the AATCC TM 66. The wrinkle recovery of a fabric is defined as the property of a fabric that enables it to resist the formation of wrinkles when subjected to a folding deformation. Five specimens per sample for both the warp and fill directions were tested. The WRA results were reported in degrees, as the sum of both warp and fill directions.

Water content. The moisture content and the moisture regain of the control and treated fabrics were evaluated by gravimetric method according to ASTM D 2495. In addition, the FTIR vibration band corresponding to the adsorbed water located around 3280 cm^{-1} was integrated from 3000 to 3700 cm^{-1} to get the integrated intensity I_{3280}.

Breaking strength and tearing strength measurement. The breaking strength (strip) of the treated fabrics was performed using the Testometric Universal Tensile Tester according to ASTM D 5035. Five specimens per sample for the warp direction and eight specimens for the fill direction were tested. The tearing strength of the treated fabrics was evaluated using the Digital Elmendorf Tearing Tester FX3750 according to ASTM D 1424. Five specimens per sample for both the warp and the fill directions were evaluated.

Abrasion resistance evaluation. The abrasion resistance of the treated fabrics was determined using the Martindale 404 instrument according to ASTM D 4966. Four specimens per sample were tested.

Dye uptake evaluation. The effect of the chemical crosslinking on the dye uptake was evaluated by measuring the ΔE according to AATCC Evaluation Procedure 6. For that purpose, the specimens were dyed with 1% of direct dye C.I. blue 80. Spectrophotometer MacBeth EC3000 (2.54 cm window view, illuminant D_{65}, and 10° observer) was used to measure ΔE. The dyed untreated cotton fabric was used as a control (three readings per specimen).

RESULTS AND DISCUSSION

FTIR integrated intensity versus %DMUG

Figure 2(a) shows representative FTIR spectra of untreated and treated cotton fabric C1 with increasing amounts of DMUG. The comparison between the spectra shows the presence of an additional peak around 1710 cm^{-1} for treated fabrics. This band is attributed to –C=O stretching vibrations and is indicative of the presence of DMUG on the treated fabric. Similar spectra were recorded for the treated fabric C2 [Fig. 2(b)]. It should be pointed out that the FTIR spectra were recorded without any sample preparation.

The vibration located around 1710 cm^{-1} was integrated from 1750 to 1670 cm^{-1} to obtain the integrated intensity (I_{1710}) for each DMUG concentration and for each fabric. Figure 3 shows the plot of the integrated absorption versus the percent of DMUG initially in the crosslinking solution. The nonlinear relationships show high degrees of correlation between the concentration of the crosslinking agent DMUG in the solution and the concentration of the DMUG effectively establishing a crosslink between cellulose chains. For fabric C1, the prediction equation is $I_{1710} = 458.2\ (\%DMUG)^2 + 0.73$, with adjusted $R^2 = 0.96$. For fabric C2, the prediction equation is $I_{1710} = 294.5\ (\%DMUG)^2 + 0.77$, with adjusted $R^2 = 0.95$. The decreasing slopes of the curves are due to the unavailability of cellulosic OH groups for crosslinking with the OH groups of the

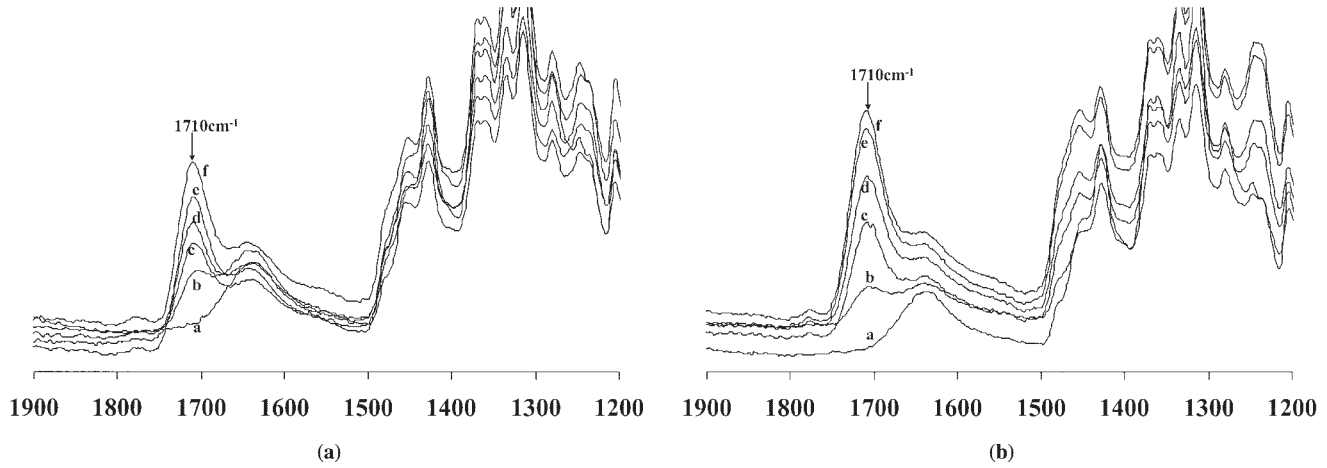

Figure 2 (a) UATR–FTIR spectra of the control and the treated cotton fabrics C1: a, control; b, 2%; c, 10%; d, 12%; e, 15%; f, 20%. (b) UATR–FTIR spectra of the control and the treated cotton fabrics C2: a, control; b, 2%; c, 10%; d, 12%; e, 15%; f, 20%.

DMUG (saturation phenomenon). Furthermore, the FTIR results show higher DMUG concentration on fabric C2 than on fabric C1. This is attributed to the lighter weight of fabric C1, associated with finer yarns and resulting in lower weight pick-up. Indeed, on average, the weight pick-up was 93.4% for C1 and 108.1% for C2. The FTIR measurements of the quantity of crosslinking agent were performed after the required five laundering and tumble-drying cycles. Therefore, the effect of the chemical treatment on fabric appearance as well as on fabric properties will be correlated with these measurements and not with the percentage of the crosslinking agent initially in the formulation.

AATCC grades versus UATR–FTIR

Figure 4 and Table I show the relationships between the integrated intensity I_{1710} as measured with UATR–FTIR and the AATCC grades of the two fabrics. As expected, there is an increase of AATCC grades (i.e., smoother fabrics) with increasing DMUG concentrations.

Wrinkle recovery angles versus UATR–FTIR

The relationship between the conditioned wrinkle recovery angles for both fabrics C1 and C2 and the integrated intensity I_{1710} of the carbonyl band are shown in Figure 5. Very good linear relationships between the wrinkle recovery angles and I_{1710} were obtained for both fabrics C1 and C2 (Table I). The coefficients of determination R^2 are 0.97 and 0.94 for C1 and C2, respectively. This means that the tendency of the fabric to recover from a deformation increases linearly with the increase of the amount of the crosslinking. However, because of the unavailability of crosslinking sites (–OH groups), a saturation phenomenon may occur above 20%. Furthermore, since

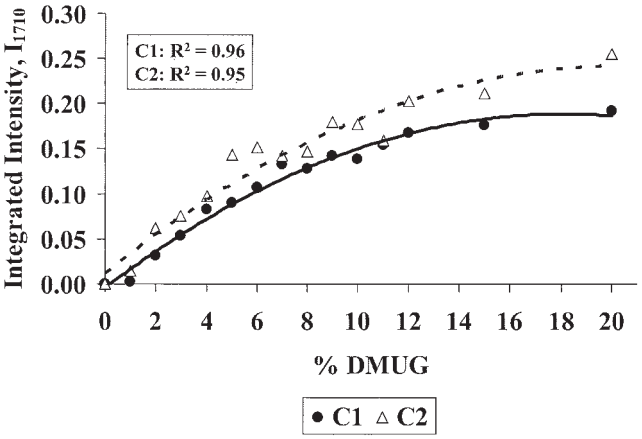

Figure 3 FTIR integrated intensity I_{1710} versus %DMUG for the fabrics C1 and C2.

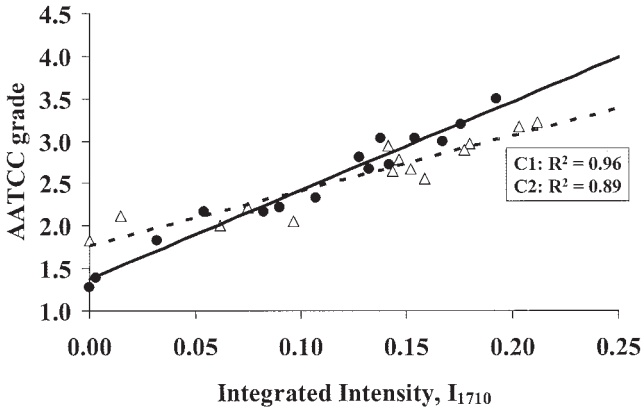

Figure 4 AATCC grade versus FTIR integrated intensity I_{1710} for fabrics C1 and C2.

TABLE I
Prediction Equations

Fabric ID	Prediction equation	Adjusted R^2
C1	AATCC grade = $0.0922 \times I_{1710} - 0.1231$	0.96
	WRA = $509.3 \times I_{1710} + 184.1$	0.97
	$I_{3280} = 64.84\ (I_{1710})^2 - 26.76\ I_{1710} + 6.35$	0.97
	TEP = $-0.391 \times (\text{WRA})^2 - 48.04$ WRA + 56913	0.95
	APH = $-0.0596 \times$ WRA + 20.515	0.91
	SA = $4 \times 10^{-5} \times (\text{WRA})^2 - 0.0262 \times$ WRA + 7.126	0.91
C2	AATCC grade = $0.1382 \times I_{1710} - 0.2294$	0.89
	WRA = $258.69 \times I_{1710} + 219.31$	0.94
	$I_{3280} = 36.16\ (I_{1710})^2 - 16.07\ I_{1710} + 5.27$	0.75
	TEP = $-300.98 \times$ WRA + 98009	0.87
	APH = $-0.001 \times (\text{WRA})^2 + 0.316$WRA -9.913	0.89
	SA = $6 \times 10^{-5} \times (\text{WRA})^2 - 0.0459$*WRA + 11.036	0.94

WRA, Wrinkle recovery angle; [(W) + (fill-F) degree], I_{1710}, integrated intensity of the peak at 1710 cm^{-1}; I_{3280}, integrated intensity of the peak at 3280 cm^{-1}; TEP, total edge point; APH, average profile height; SA, surface area.

the water molecules in the cellulose macromolecule establish hydrogen bonds with –OH groups, the unavailability of –OH groups will result in a decrease of moisture content and moisture regain of the treated fabrics.

Water content

Figures 6(a) and (b) show the evolution of the amount of adsorbed water (integrated intensity I_{3280}) versus the amount of the crosslinking agent (integrated intensity I_{1710}) for the fabrics C1 and C2, respectively. A nonlinear decrease of the amount of absorbed water was observed when the amount of the crosslinking agent increases (Table I). The integrated intensity I_{3280} of fabrics C1 and C2 treated with 20% resin decreased by 47.3 and 37.2%, respectively. When evaluated by ASTM D 2495, the moisture content of treated fabrics (20% of resin) decreased by 16.1 and 17.5% for C1 and C2, respectively. However, the moisture regain decreased for the same fabrics by 14 and 30.4%. The decrease in water content and moisture regain is explained by the decrease of the number of OH groups available for H_2O absorption via hydrogen bonding.

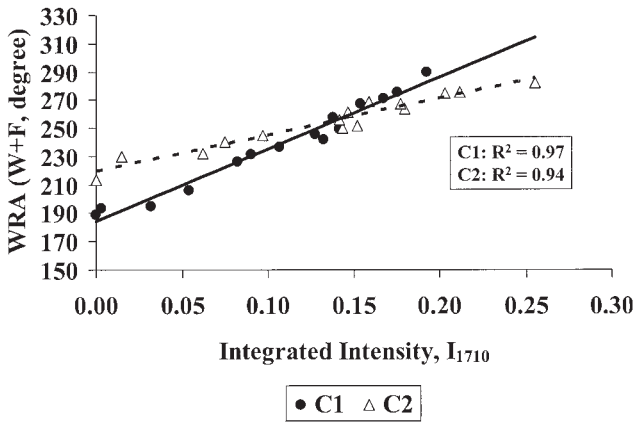

Figure 5 Wrinkle recovery angle (W + F, in degree) versus FTIR integrated intensity I_{1710} for fabrics C1 and C2.

Fabric smoothness as evaluated by automatic image analysis system versus WRA

As stated above, the wrinkle recovery angle measurements of the treated fabric provide an indication of the ability of the fabric to recover from a crease or a deformation. Therefore, the higher the angle (expressed in degrees), the fewer wrinkles are on the fabric surface. Thus, an analysis of the topography of the fabric surface should correlate with the wrinkle recovery result. Figures 7(a–c) show, respectively, the evolution of the total edge points, the average profile height, and the surface area with wrinkle recovery angle for fabric C1. The prediction equations and the coefficients of correlation are presented in Table I for both fabrics C1 and C2. All coefficients of correlation are highly significant, meaning that the approach used for automatic assessment of surface wrinkles is valid.

Strip strength, abrasion, and Elmendorf tear strength tests versus UATR–FTIR

Figures 8(a) and (b) and Table II show the relationship between the integrated intensity I_{1710} as measured with UATR–FTIR and the strip strength of the two fabrics in both the warp and filling directions. There is a decrease in fabric strength with increasing DMUG concentrations. For C1 there is a 58.2 and 69.2% decrease in strength for the warp and filling directions, respectively, between the control (no DMUG) and the fabric treated with 20% DMUG. For C2 there are very similar results, with 64.6 and 69.7% decrease in strength for the warp and filling directions, respec-

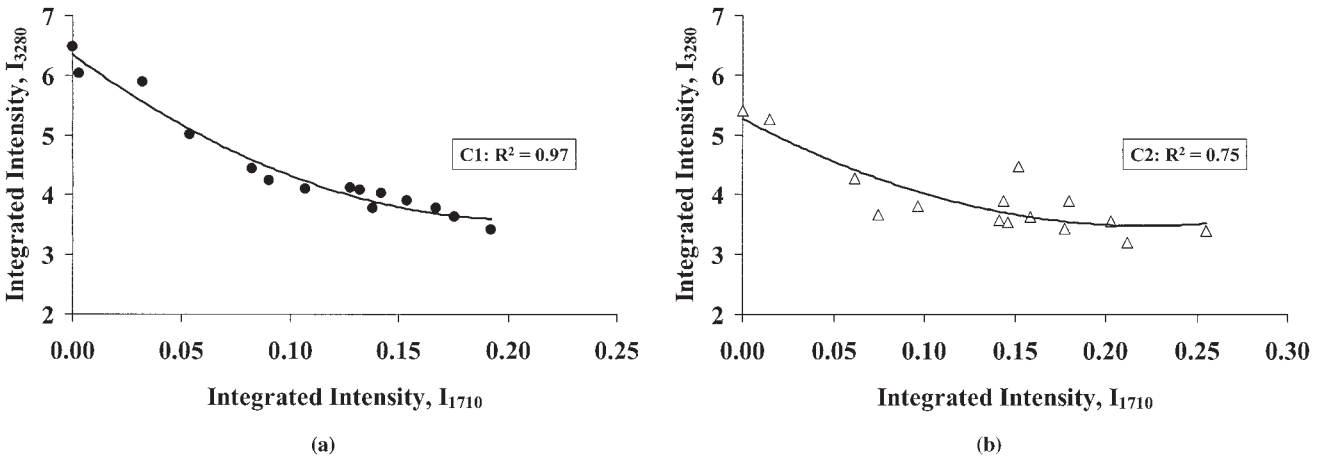

Figure 6 (a) Integrated intensity I_{3280} versus integrated intensity I_{1710} for fabric C1. (b) Integrated intensity I_{3280} versus integrated intensity I_{1710} for fabric C2.

tively, between the control (no DMUG) and the fabric treated with 20% DMUG.

Figures 8(c) and (d) and Table II show the relationships between the integrated intensity I_{1710} as measured with UATR–FTIR and the Elmendorf tear strength of the two fabrics in both the warp and filling directions. As expected, there is a decrease in fabric tearing strength with increasing DMUG concentra-

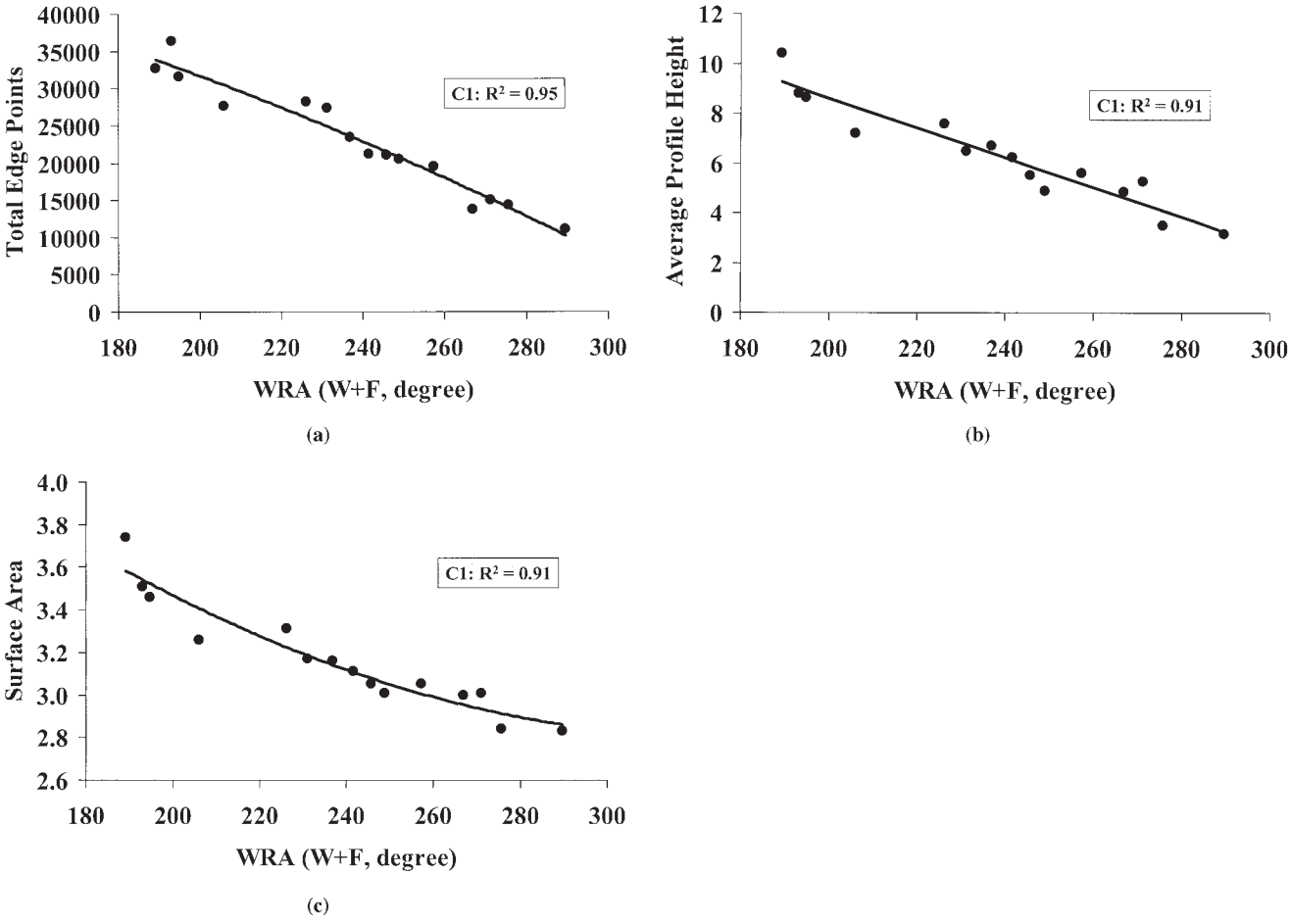

Figure 7 (a) Total edge points versus wrinkle recovery angle (W + F, degree) for fabric C1. (b) Average profile height versus wrinkle recovery angle (W + F, degree) for fabric C1. (c) Surface area versus wrinkle recovery angle (W + F, degree) for fabric C1.

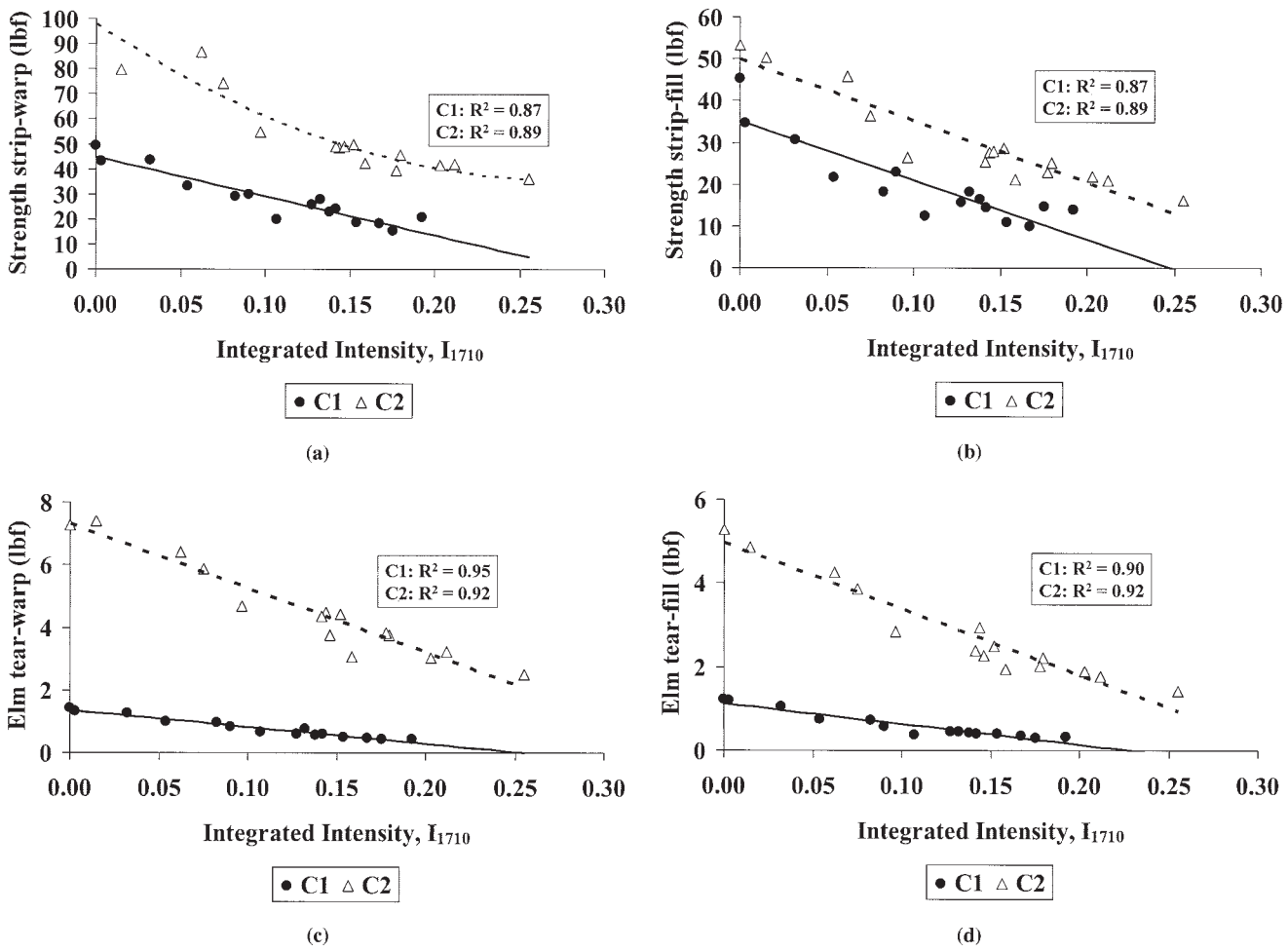

Figure 8 (a) Fabric tensile strength (warp) versus FTIR integrated intensity I_{1710} for fabrics C1 and C2. (b) Fabric tensile strength (fill) versus FTIR integrated intensity I_{1710} for fabrics C1 and C2. (c) Fabric tear strength (warp) versus FTIR integrated intensity I_{1710} for fabrics C1 and C2. (d) Fabric tear strength (warp) versus FTIR integrated intensity I_{1710} for fabrics C1 and C2.

tions. For C1 there is a 68.8 and 72.8% decrease in strength for the warp and filling directions, respectively, between the control (no DMUG) and the fabric treated with 20% DMUG. For C2 there are very similar results, with 65.7 and 73.2% decrease in strength for the warp and filling directions, respectively, between

TABLE II
Prediction Equations: Treated Fabrics versus FT–IR Integrated Intensity I_{1710} for Fabrics C1 and C2

Fabric ID	Prediction equation	Adjusted R^2
C1	Strip W = $-0.0056 \times I_{1710} + 0.264$	0.87
	Strip F = $-0.0055 \times I_{1710} + 0.217$	0.77
	Elm W = $-0.1782 \times I_{1710} + 0.247$	0.95
	Elm F = $-0.1845 \times I_{1710} + 0.217$	0.90
	Abrasion cycles = $10^6 \times (I_{1710})^2 - 313058 \times I_{1710} + 25638$	0.96
	$\Delta E = 0.0059 \times I_{1710} - 0.022$	0.94
C2	Strip W = $0.00005 \times (I_{1710})^2 - 0.01 \times I_{1710} + 0.525$	0.89
	Strip F = $-0.006 \times I_{1710} + 0.315$	0.88
	Elm W = $-0.045 \times I_{1710} + 0.339$	0.92
	Elm F = $-0.0584 \times I_{1710} + 0.3$	0.92
	Abrasion cycles = $121{,}103 \times (I_{1710})^2 - 121{,}100 \times I_{1710} + 21{,}966$	0.96
	$\Delta E = 0.0065 \times I_{1710} - 0.007$	0.87

the control (no DMUG) and the fabric treated with 20% DMUG.

The resistance of the fabric to abrasion decreased drastically. Both fabrics had an abrasion resistance of 26,500 cycles when untreated against only a few hundred cycles with the 20% DMUG treatment. There is a 97.5 and 99.3% decrease for fabrics C1 and C2, respectively. The relationship between the abrasion resistance cycles and the integrated intensity I_{1710} is shown in Table II.

The decrease of the fabric strength (both tear and strip strengths) and the abrasion resistance is attributed to the fact that the establishment of crosslinks between the cellulosic chains reduces the chains' slippage and their flexibility.[1] The crosslinking also leads to the stiffening of the cellulosic macromolecular network and fiber embrittlement.[12–14]

Dye uptake versus UATR–FTIR

Table II shows the relationships between the integrated intensity I_{1710} as measured with UATR–FTIR and the ΔE of the two treated fabrics dyed with direct C.I. blue 80. As expected, there is a decrease in dye uptake with increasing DMUG concentrations. This is attributed to the lack of available sites for the dye molecules. Indeed, the swelling of the treated fabric in the water is inhibited and the fabric is less absorbent. Because of the covalent crosslinking between the cellulosic OH groups, the dye molecules cannot penetrate the internal fiber area. Therefore, the dye uptake is lower for treated fabric than for the control.

CONCLUSION

Two cotton fabrics were treated with increasing amounts of a textile-finishing agent to impart durable press properties. The UATR–FTIR was used to evaluate the amount of the crosslinking agent effectively crosslinked to the cellulose chains after the required five laundering tumble-drying cycles. Textile performance testing was conducted on both treated and untreated fabrics and the results were correlated to the amount of the crosslinking agent as evaluated by UATR–FTIR. The UATR accessory with the ZnSe–diamond composite crystal was proven to be a reliable and nondestructive technique for determining the amount of the crosslinking agent linked to the cellulose macromolecules. This technique may, therefore, be used for quality control purposes.

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