EFFECT OF ENZYMATIC HYDROLYSIS ON THE MEASURED PRIMARY HAND QUALITIES OF COTTON FABRICS P. Radhakrishnaiah, Xiaomin Meng and Gan Huang Georgia Institute of Technology Atlanta, GA G. Buschle-Diller and William K. Walsh Auburn University Auburn, AL A. P. S. Sawhney USDA, ARS New Orleans, LA

Abstract

The effects of enzyme treatment on the strength and abrasion resistance, surface appearance, subjective hand qualities, and dveing characteristics of cellulosic textiles have been widely studied and reported [Diller et. al., 94; Kumar et. al., 94; Kundu et. al., 93;]. Literature also gives significant information [Koo et.al., 94; Bhatawdikar et. al., 92; Choe et. al., 97] on how exactly the properties of cellulosic textiles are affected when they are enzyme treated before and after being subjected to other wet treatments such as desizing, scouring, bleaching, mercerization, and dyeing. However, the effects of fabric agitation on the hand related fabric properties, and the influence of these property changes on the primary hand qualities such as Koshi (stiffness), Numeri (smoothness), and Fukurami (fullness and softness) have not been widely studied and reported. Changes occurring in the thermal comfort performance of the treated fabrics have also not been fully investigated. This work reports the measured changes in the mechanical and surface properties of plain weave fabrics subjected to enzyme treatment with and without mechanical agitation. It also describes the influence of the property changes on the measured hand and appearance qualities.

Some of the mechanical properties and the tactile qualities of the treated fabrics differed by as much as 50 %, compared to that of the untreated fabric. One way analysis of variance revealed that almost all the measured properties (except initial thickness, percentage thickness compression, and bending rigidity) of the treated fabrics differ from that of the untreated fabric. Results also revealed that there are some unique differences in properties between the fabrics subjected to enzyme treatment with and without mechanical agitation, implying that the level of mechanical agitation employed during treatment can significantly alter the finishing effects derived, including the thermal comfort performance of the treated fabrics.

Introduction

Buschle-Diller and her co-workers looked at the changes produced by enzymatic hydrolysis on cotton, linen, ramie, and viscose rayon fabrics that were treated with a cellulase enzyme. They observed that the drop in yarn strength is progressive with increasing weight loss for cotton and viscose rayon fabrics. Mechanical tests on the fabrics showed significant changes in fabric stretchability and stiffness, as well as in the mobility of yarns within the samples. Kumar and Putrell showed that the cellulase enzyme has a slightly different effect on the properties of different cellulosic fabrics. On both viscose ravon and Iyocell (Tencel), cellulase altered the handle and drapeability, and removed the surface fuzz. Cellulase also reduced the tendency of rayon to pill, and it reduced fibrillation of Iyocell. Kundu and his associates revealed that enzyme treatment of jute fibers leads to an increased pore volume (46.2%), a larger surface area of lignin accessible to the oxidant (30%), and hence to improved bleaching. They also noted that enzyme treatment increases transverse swelling (14%), and reduces bulk torsional rigidity (12.5%), making the fibers more flexible and, therefore, softer.

Koo, Ueda, Wakida, Yoshimura, and Igarashi investigated the rate of catalytic hydrolysis of cotton fabrics by a cellulase enzyme in the presence of dyes and surfactants. They found that the presence of both direct and reactive dyes on the substrate inhibits the catalytic reaction of the enzyme, whereas a vat dye does not. The enzyme was found to be more active on mercerized cotton and less active on unmercerized cotton. The treated fabrics showed reduced tear strength and their dye uptake was also found to be lower. Bhatawdekar et al attributed the reduced susceptibility to enzymolysis of the alkali treated fibers to the specific morphological state of the fibrils. Choe et al showed that the amount of weight loss obtained in the cellulase treatment of cotton fabrics depends on whether or not the fabric has been mercerized, on yarn linear densities, and on the fabric structure. Other things being equal, weight loss was found to be greater for mercerized than for unmercerized fabrics. It was also greater for finer as opposed to coarser yam counts, and knit fabrics lost more weight than woven fabrics did. The authors also looked at the effect of pre-existing dyes on cellulase activity and concluded that all the three dyes used (direct, reactive, and vat dyes) inhibit the cellulase reaction. The concentration of the dye on the fabric was found to be the most critical factor governing the weight loss.

The present work seeks to define the level of changes occurring in the low stress-mechanical and surface properties of fabrics, in response to enzyme treatment with and without mechanical agitation of fabrics. The goals of the work therefore, are to: i. Define the level of changes in mechanical and surface properties, ii. Translate property differences into hand quality differences, iii. Ascertain the

Reprinted from the Proceedings of the Beltwide Cotton Conference Volume 1:735-739 (1998) National Cotton Council, Memphis TN

statistical significance of the resulting changes, iv. Relate changes to thermal comfort performance, and v. Define the influence of mechanical agitation on the finishing effects derived.

Materials and Methods

Test Fabric

For this work, we used a 100% cotton sheeting fabric that weighed 165 g/m². The fabric had a plain weave construction with 56 ends and 56 picks per inch, and it was desized, scoured and bleached as per standard procedures, before it was subjected to enzyme treatment.

Enzyme Treatment

We treated the above fabric with a cellulase enzyme, and the treatment was carried out with and without mechanical agitation. A mildly shaking incubator was used for the treatment involving no agitation, while for the agitating treatment, we used a laboratory model launderomat. Preliminary trials involving weight loss measurements suggested that a 4% weight loss could be achieved approximately after 6 hours of treatment in the shaking incubator, and after two hours of agitating treatment in the launderomat. Thus we used a six-hour treatment time for the incubator, and two-hour time for the launderomat. The enzyme concentration (10% on the weight of the conditioned fabric), treatment temperature $(37\pm1^{\circ}C)$, liquor ratio (1:100), and pH (4.9) were maintained the same for the two types of enzyme treatment. The pH was maintained at the required level using a 0.05 M sodium acetate buffer solution. At the end of treatment, the treated samples were immersed in 100% acetone to deactivate the enzyme. The deactivated samples were then washed in distilled water, air dried, and conditioned under standard atmospheric conditions. The treated and conditioned samples were weighed again to make sure that the weight loss of each individual sample was within the range of (4+0.15)%.

Low-Stress Mechanical And Surface Properties

We used the Kawabata Evaluation System for Fabrics (KES-F) to measure the low-stress mechanical and surface properties of the treated and untreated fabrics0. Table I lists the property parameters evaluated under tensile, shear, bending, compression, and surface tests while Table II describes the test conditions used for the measurement of fabric properties. We carried out four warp and four filling tests to determine the average values of the test parameters listed in Table I. We also conditioned the test specimens at $65 \pm 2\%$ RH and $20 \pm 1^{\circ}$ C before measuring their properties.

Thermal Properties

We used Kawabata's new thermal tester "Thermolabo-II" [14] to compare the warm/cool contact sensation offered by the fabrics. We also used this tester to measure the energy dissipation (rate of heat and moisture flow) through the fabrics. The measurement of warm/cool contact sensation

is based on the fact that fabrics maintained at room temperature tend to momentarily absorb certain amount of heat from the body every time they come into contact with the body. The rate of flow of heat which is believed to reach a peak value (Q_{max}) approximately 0.2 sec. after contact is established by the fabric [14], has been found to relate to the warm/cool feeling offered by the fabric. The thermolabo device uses a constant heat capacity hollow metal box (T-Box) in place of the human body and measures the maximum rate of heat flow (Q_{max}) from the T-Box to the fabric surface.

The energy dissipation through the fabric was measured using a constant temperature hot plate (BT-Box) which also is a part of the "Thermolabo" device. The difference in the electrical power required to maintain the hot plate at the body temperature with and without the test specimen placed on the hot plate is a measure of the energy dissipation through the fabric. We obtained the energy dissipation values under both dry and wet contact conditions to understand the heat transfer behavior of the fabrics when they are in contact with dry and wet skins.

In the dry method, the fabric was directly placed on the surface of the hot plate and the amount of energy dissipated through the fabric in unit time was measured as a function of the electrical energy supplied to the hot plate to maintain it at the body temperature. In the wet method, a porous paper subjected to controlled wetting was placed on the hot plate and the fabric specimen was then placed on the top of the paper. The wet paper was used to simulate the wet skin. In general, energy dissipation values obtained in the wet state can be expected to be higher than that obtained in the dry state because of the additional energy expended in the form of latent heat of vaporization of water.

Other Fabric Properties

The air-permeability of the fabrics was measured on the Metafem Airpermeability Tester, using a single orifice for all three fabrics.

Diffusion resistance (the rate of diffusion of water vapor through the fabric) was measured using the Shirley Water Vapor Permeability Tester. The test procedure involved comparing the rate of diffusion of water vapor from a control dish covered by a standard fabric with the rate of diffusion from another control dish that is covered by the standard fabric and a test specimen. Four different thicknesses of the air layer between the water surface and the standard cover fabric were used to define the underlying relationship between the thickness of the enclosed air layer and the rate of loss of water vapor from the dish. This relationship was then used to express the resistance of the test specimen to water vapor diffusion, in terms of the height of the air column offering an equivalent resistance to the diffusion of water vapor. The diffusion resistance values given in table IV, therefore, represent the height in centimeters of the still air column that offers diffusion resistance equivalent to that of the test fabric.

Drape coefficient was measured using the Cusick Drape Tester. Percentage drape coefficient was computed as the ratio of the area of the draped specimen to the area of the flat specimen. A lower value of the drape coefficient is thus indicative of better or more efficient draping behavior.

Computation of Hand Values

Sixteen of the eighteen property parameters listed in Table II were used in Kawabata's hand prediction equation for men's winter suit applications [Kawabata et. al., 1980] to compute the primary hand qualities of the experimental fabrics. Table IV describes the primary hand qualities associated with this end-use and the fabric properties that relate to the individual hand qualities.

Computation of Total Appearance Value, Formability and Springiness Properties

Kawabata et. al., first developed a regression equation in the year 1980 for the prediction of the total appearance value of men's suiting materials. Later, the authors [Kawabata et. al., 1989; Niwa, Nitta, and Kawabata, 1985] refined their original equation, based on the experience gained in evaluating hundreds of suiting fabrics processed by an apparel company over a period of five years. The refined equation for the prediction of total appearance value is based on the understanding that the appearance of a sewn garment is a function of formability, springiness, total hand, and drapeability characteristics of the fabric, and that each of these characteristics can be objectively evaluated from a set of fabric mechanical properties. The following refined equation developed by the authors was used to compute the appearance value listed in Table IV.

TAV = Total Appearance Value = -1.3445 + 0.2841 Y1 + 0.5747 Y2 + 0.3068 Y3 + 0.2071 Y4, where Y1= Formability, Y2= Springiness, Y3 = Drapeability, and Y4 = Fabric quality parameter calculated from total hand value (THV-S).

Y1, Y2, Y3, and Y4 are parameters that relate to the measured fabric properties and they are computed from the sixteen property parameters.

Results and Discussion

Effect Of Enzyme Treatment on Fabric Properties

Low-Stress Mechanical and Surface Properties: From Tables IV and V, it can be seen that only a few measured properties (TO, EMC%, and B) are unaffected by the enzyme treatment. It can also be seen from Table IV that the fabric properties most affected by enzyme treatment are the tensile elongation (EM%), tensile resilience (RT%), tensile energy (WT), tensile linearity (LT), compressive resilience (RC%), linearity of the compression curve (LC), shear rigidity (G), shear hysteresis (2HG), surface friction (MIU),

and mean deviation of surface friction (MMD). The treated fabrics became more extensible and less resilient under tensile deformation, as indicated by an increase in tensile elongation of about 45%, and a drop in tensile resilience of 23%. The treated fabrics also became more resilient to compressive deformation (21%). Linearity of the compression curve, which is an indication of the ease of compression, increased (29%) after the enzyme treatment and so is the fabric thickness measured under compressive loads of 0.05 g/sq.cm and 50 g/sq.cm. The treated fabrics also showed higher values for shear rigidity (15%) and shear hysteresis (23%), and the surface of the treated fabric became smoother after enzyme treatment, as indicated by the increased surface friction (16%) and reduced variability of the same along the surface of the fabric. The changes in compression and shear properties as well as the differences in measured drape coefficient and air permeability values (Table IV) suggest that the fabric becomes tighter and thicker as result of swelling of fibers in enzyme treatment. The results thus confirm the observations of Kundu et al on fiber swelling. The results also agree with that of Diller and her associates in that the fabric becomes more stretchable and less resistant to stretch after enzyme treatment.

Primary Hand Qualities: We used Kawabata's primary hand equations for men's winter suiting materials to evaluate three different tactile qualities of the experimental fabrics. Even though these equations were developed specifically to predict the hand qualities of winter suiting fabrics, their application for the hand prediction of fabrics representing different chemical/mechanical treatments can serve the purpose of understanding the effect of the particular treatment on the individual tactile qualities. The applicable range of values for the three primary hand qualities listed in Table IV is 0-10. Higher the value of the primary hand quality within the 0-10 range, greater is the intensity of this particular hand (tactile) feeling. It can be seen from the table that enzyme treatment makes the fabric smoother (numeri), fuller and softer (fukurami), and less stiff (koshi).

Appearance and Other Related Properties: t-test on the formability and springiness values listed in Table IV suggest that these properties are not significantly different for the three different fabrics, at 95% confidence level. In terms of formability and springiness, therefore, the treated fabrics behave similar to that of the untreated fabric. The total appearance value of the fabric treated with mechanical agitation was found to be significantly different from that of the other two fabrics, at 95% confidence level. Treatment with mechanical agitation, therefore, appears to result in a slightly inferior appearance of the sewn garment made from the treated fabric.

Thermal Properties: The Q_{max} values in Table IV indicate the transient heat flux or the maximum rate of flow of heat from the body to the fabric surface after contact is established between the two. Higher the Q_{max} value (i.e.,

greater the peak value of the momentary heat flow), cooler will be the contact sensation. Based on the average Q_{max} values and their standard deviations, it can be inferred that the fabric representing enzyme treatment with agitation gives a warmer contact sensation compared to the other two fabrics. This result, in fact, is in agreement with earlier work [14] in that a ruffled or wrinkled fabric surface offers a lower contact area to the skin (hot plate), and hence a lower Q_{max} value. The difference in the dry energy dissipation values of the three fabrics was found to be insignificant at 95% confidence. However, the wet energy dissipation values of the two treated fabrics were significantly higher than that of the untreated fabric, implying that the treated fabrics may provide a slightly better heat dissipation from the body under hot and humid weather conditions.

Other Fabric Properties: The treated fabrics showed better drapeability compared to the untreated fabric. The treated fabrics also showed reduced airpermeability compared to the untreated fabric. Tightening of the fabric structure due to fiber swelling appears to be mainly responsible for the reduced airpermeability and the improved drapeability of the treated fabrics. The fabric corresponding to no agitation showed slightly reduced resistance to water vapor diffusion while the fabric representing treatment with agitation showed substantially increased resistance to water vapor diffusion. At a first glance, it appears that the results of the diffusion test contradict that of the energy dissipation test. However, a close look at the two tests would reveal that the tests are drastically different and so are the results obtained from them. The diffusion test measures the resistance offered by the fabric to the diffusion of water vapor at room temperature, keeping an initial gap of several millimeters between the fabric and the surface of the still water column. The energy dissipation test, on the other hand, measures the rate of flow of heat and moisture through the f results provided enough evidence to conclude that mechanical agitation of fabrics during enzyme treatment affects not only the tactile and aesthetic qualities of the treated fabrics but also their thermal comfort performance. fabric when the fabric is actually in contact with a moist hot plate that is maintained at body temperature. Thus one test measures the resistance to water vapor diffusion under non-contact conditions while the other test measures the rate of flow of heat and moisture when the fabric is in contact with a hot surface. The results of the two tests cannot, therefore, be compared.

Influence Of Fabric Agitation on the Properties of the Finished Fabric

Referring to Tables IV and V it can be seen that the fabric properties that are significantly affected by mechanical agitation are compressive resilience (RC%), tensile linearity (LT), shear modulus (G), shear hysteresis (2HG5), warm/cool feeling, airpermeability, diffusion resistance, drape coefficient, and the appearance of the finished garment. Compared to the fabric treated without mechanical agitation, the fabric treated with agitation shows reduced compressive resilience, reduced resistance to tensile deformation (lower tensile modulus), reduced airpermeability, increased resistance to the diffusion of water vapor, reduced drapeability, slightly inferior appearance of the finished garment, and a slightly warmer contact sensation. The difference in these and other measured properties made a significant impact on the objectively evaluated tactile qualities of numeri (smoothness), and Fukurami (fullness and softness).

Summary and Conclusions

We subjected a tightly woven medium weight 100% cotton fabric to enzyme treatment with and without mechanical agitation. We evaluated the fabrics using KES-F and other testing instruments, and compared the properties of the treated fabrics with that of the untreated fabrics. We also compared the properties of the fabrics representing the two types of enzyme treatment, one with agitation, and the other without agitation.

We noticed major changes in the low-stress mechanical behavior of the treated fabrics. Among the properties most affected by enzyme treatment are the tensile elongation (EM%), tensile resilience (RT%), tensile linearity (LT), compressive resilience (RC%), linearity of the compression curve (LC), shear rigidity (G), shear hysteresis (2HG), surface friction (MIU), and mean deviation of surface friction (MMD). A few of these properties changed by as much as 50% after enzyme treatment. Enzyme treatment also made the fabrics smoother, softer and fuller, and less stiff to bend and stretch. The treated fabrics showed higher energy dissipation under wet contact conditions, implying that they may offer a slightly superior thermal comfort performance under hot and humid weather conditions. The treated fabrics also showed better drapeability and reduced airpermeability compared to the untreated fabric.

Between the fabrics representing the two types of enzyme treatment, the one representing treatment with agitation accounted for reduced compressive resilience, reduced resistance to tensile deformation, reduced airpermeability, increased resistance to the diffusion of water vapor, reduced drapeability, slightly inferior appearance of the finished garment, and a slightly warmer contact sensation. The results provided evidence to conclude that mechanical agitation of fabrics during enzyme treatment affects not only the tactile and aesthetic qualities of the treated fabrics but also their thermal comfort performance. The level of mechanical agitation, therefore, can be expected to influence the finishing effects derived. In deciding whether mechanical agitation is to be used or not, and if it is used, what level of agitation is appropriate, one must pay close attention to the nature of the tactile character desired, the structural features of the fabric being treated, and the type of equipment used for enzyme treatment.

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Table I. Mechanical and surface properties involved in KES tests Test parameter Definition of parameter Unit .Tensile Elongation at 5N/cm tension % EM LT Linearity of stress-strain curve None WT Energy to extend fabric to 5N/cm tension J/m² RT Tensile Resilience % Shear Shear rigidity at 39.4 mrad shear strain N/m G 2HG Hysteresis at 8.7 mrad shear strain N/m Hysteresis at 87 mrad shear strain 2HG5 N/m Bending Bending rigidity at 1.5 cm⁻¹curvature В μnm mN 2HB Hysteresis at +0.5 cm⁻¹ curvature Compression LC Linearity of the compression curve None WC Compression energy at 5 kPa pressure J/m² RC Compressive Resilience % EMC %Thickness compression % TO Fabric thickness at 5 Pa pressure mm TM Fabric thickness at 5 kPa pressure mm Surface Coefficient of surface friction MIU None MMD Mean deviation of MIU None SMD Mean deviation of fabric surface profile μm

Table II. Instrum	nent settings and loading conditio	ns for fabric evaluation
Compression	Rate of compression	0.02 mm/sec
	Maximum force	5 kPa
	Area compressed	2.0 cm^2
Bending	Rate of bending	0.5 cm ⁻¹ /sec
	Maximum curvature	$\pm 2.5 \text{ cm}^{-1}$
	Sample size (LxW)	20 cm x 1 cm
Surface	Rate of traverse	1 mm/sec
	Tension on sample	0.1 N/cm
	Normal force, friction	0.5 N
	Contact force, roughness	0.1 N
	Distance measured	3 cm
Shear	Rate of shearing	0.417 mm/sec
	Maximum shear angle	+140 mrad
	Tension on sample	0.1 N/cm
	Sample size (LxW)	5 cm x 20 cm
Tensile	Rate of extension	0.1 mm/sec
	Sample size (LxW)	5 cm x 20 cm
	Maximum tensile force	5 N/cm

Fabric mass per unit area

g/m²

Table III. Primary hand qualities associated with men's winter suit application

Hand expression		Properties influencing hand quality	
Japanese term KOSHI	English equivalent Stiffness	A fabric having a compact weave density and made from elastic yarn gives a high koshi value	
FUKURAMI	Fullness & Softness	A feeling coming from a combination of bulky, rich and well formed impressions	
NUMERI	Smoothness	Feeling arising from smoothness and softness	

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PROPERTY	NO TREATMT	TREATMT-E	TREATMT-EM	
Tensile				
EM, %	6.64	9.36	9.78	
RT, %	51.65	39.20	38.48	
LT	0.83	0.73	0.69	
WT, J/sq.m	11.64	15.84	16.04	
Shear				
G, N/m	1.883	2.260	2.092	
2HG, N/m	3.242	4.186	3.879	
2HG5, N/m	7.380	7.619	7.330	
Bending				
B, μNm	7.45	6.77	7.16	
2HB, mN	0.530	0.785	0.775	
Compression				
LC	0.34	0.45	0.43	
WC, J/m ²	0.27	.036	0.34	
RC, %	41.96	54.21	48.39	
TO, mm	0.687	0.717	0.701	
Surface				
MIU	0.235	0.277	0.277	
MMD	0.083	0.066	0.057	
SMD	8.916	8.010	7.140	
Other	Measured	Properties		
Qmax (W/cm ²⁾	0.125	0.117	0.124	
Dry Energy (W)	1.230	1.223	1.273	
Wet Energy (W)	3.584	3.804	3.936	
Drape Coeff. (%)	85.7	63.65	75.02	
Airpermeability	73.54	58.14	48.55	
Diffus Res. (cm)	0.31	0.274	0.579	
Computed	Attributes			
Koshi	2.184	0.531	0.673	
Numeri	3.664	7.191	8.366	
Fukurami	1.030	5.359	5.877	
Formability	3.86	4.08	4.10	
Springiness	0.38	0.76	0.10	
TAV (New)	11.01	11.86	9.83	

Table IV. Measured properties and computed fabric quality attributes

Table V. Results of One Way Analysis of Variance

	ANOVA Between			
	Three Treatments			
	(No treatment,		ANOVA Between	
	Treatment-E, and		Treatment-E and	
Property	Treatment-EM)		Treatment-EM Only	
		Significance		Significance at
		at 95%		95%
	F	Confidence	F	Confidence
Compression				
LC	9.54	Yes	1.45	No
WC	31.04	Yes	2.47	No
RC%	19.52	Yes	20.86	Yes
EMC%	2.69	No	0.83	No
TO	2.55	No	1.19	No
TM	30.67	Yes	1.77	No
Tensile				
LT	8.61	Yes	10.19	Yes
WT	70.85	Yes	0.17	No
RT%	923.4	Yes	2.97	No
EM%	111.9	Yes	2.73	No
Shear		1		
G	33.24	Yes	12.52	Yes
2HG	19.79	Yes	3.25	No
2HG5	5.18	Yes	13.23	Yes
Bending		1		
В	1.28	No	0.65	No
2HB	8.45	Yes	0.01	No
Surface		1		
MIU	12.21	Yes	0.10	No
MMD	14.14	Yes	1.30	No
SMD	16.67	Yes	5.86	No
Thermal				
Q _{max}	4.17	No	5.92	Yes
Wet Energy	17.29	Yes	3.54	No
Other				
Diffusion Res.	8.34	Yes	14.7	Yes
Drape Coefft.	28.4	Yes	19.8	Yes
Airpermeability	286.5	Yes	66.2	Yes