

Effect of Cellulase Pretreatment of Raw and Bleached Cotton Fibers on Properties of Hydroentangled Nonwoven Fabrics

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Received 24 February 2006; accepted 16 December 2006

DOI 10.1002/app.26158

Published online 27 March 2007 in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: This study was undertaken to investigate the effect of enzymatic pretreatment of cotton (polysaccharides) fibers on the properties of resulting nonwoven fabric. Enzymatic treatment is known to improve the esthetical properties of fabrics but will likely lead to a reduction in strength. In the case of nonwovens the strength loss can be even more drastic as cellulase may attack bonded areas of the fabric. In this work, raw and bleached cotton fibers were treated with enzyme solutions prior to fabric formation to avoid possible damage to the bonded areas and improve strength retention. These fibers were first modified with commercially available whole cellulases and

monocomponent endoglucanase enzyme solutions. Then they were formed into a fabric and bonded via hydroentangling. Parameters such as bending modulus, fabric tenacity, fiber strength, length and reducing power were measured for each sample. The pretreatment of cotton fibers prior to fabric formation showed that the resulting nonwovens could be stronger and more drapeable than the same fabric composed of untreated fibers. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 105: 492–499, 2007

Key words: enzymes; polysaccharides; fibers; mechanical properties; structure-property relations

INTRODUCTION

The use of enzymes was reported for the first time in 1857 and nowadays, they are widely used in textile industry to replace or partake in traditional processes such as stoning, deinking, scouring, and dyeing.¹ There are many enzymes that are involved in fabric manufacturing; however cellulases have taken a special niche in this area.

Cellulases are a group of proteins that degrade cellulose to glucose by hydrolyzing β -1,4-glucosidic bonds in the polysaccharide molecule. Secreted by bacteria or fungi, they are a crude mixture of several enzymes: endoglucanases (EG), exoglucanases, and β -glucosidases. All three types of enzymes exhibit different activity on cellulose. Endoglucanases cut at random amorphous sites of the cellulose chain, producing new reducing (also called reducing power)² or nonreducing ends and oligosaccharides of various lengths. Exoglucanases attack the reducing or nonreducing ends of the cellulose chain, liberating glucose or cellobiose as major products; and β -glucosidases remain in the solution hydrolyzing small chain reduc-

ing sugars to glucose.³ Most cellulases consist of catalytic and cellulose-binding domains (CBDs). The latter plays an important role in enzymatic hydrolysis. It has been suggested that the presence of CBD promotes the enzyme activity by increasing their concentration at the substrate surface or by releasing a single cellulose polymer from crystalline region via breaking hydrogen bonds.^{4,5}

Typically, cellulases are applied to the fabrics at the final stages of finishing in processes such as biopolishing, scouring, bio-stoning, color brightening etc. One of the disadvantages of this approach would be a significant loss in the fabric strength.^{6–8} Thus, process optimization and proper selection of enzymes should be done to minimize strength loss.^{4,5,9,10}

Several investigators have tried to identify the type of cellulase suitable for the fabric treatment to achieve desirable effects without deterioration of the fabric properties. Kumar et al.⁹ concluded that whole cellulase is good to apply to sturdy fabrics in applications where high level of surface polishing is required. However, for less durable fabrics, endo-rich cellulases were found to be the best. Cortez et al.⁸ came to the same conclusion stating that EG-rich enzyme solutions improve dimensional stability of the fabric with the minimum effect of exo-cellulases. Lenting and Warmoeskeken⁵ explained the effect of EGs by the ability of these enzymes to act preferably on the amorphous regions of the cellulosic fibers rather than crystalline. The latter is the responsible for the fiber

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Contract grant sponsor: Procter and Gamble.

Contract grant sponsor: Nonwovens Cooperative Research Center.

strength. Moreover, the removal of CBD from EGs should promote the activity of endo-cellulases towards amorphous regions.

While enzymatic treatments are well-established for woven and knitted fabrics, they have found little or no use in treating nonwovens. As nonwovens move from low-cost, disposable goods to durable, high performance fabrics, value-added fabrics, the importance of the tactile properties of nonwoven fabrics becomes increasingly important. This especially concerns hydroentangled cotton fabrics. Hydroentangling is a mechanical bonding technology that uses fine high velocity jets of water to displace, twist and/or interlock the fibers in the web. The final outcome is a highly compressed and uniform fabric sheet, which may be strong enough to be finished on conventional textile finishing equipment. To produce durable fabric with relatively high strength requires higher entanglement energies or post-treatment with a binder. The latter is not preferred since it adversely affects the fabric hand. Higher entanglement energy also causes a stiffer and less pleasant hand in cellulosic nonwovens¹¹ with fiber damage being a possibility as well. Thus, the aim of the present study was to examine the potential use of cellulase as a way to reduce the stiffness of the fabric without the reduction or even improvement in its strength. During this work, raw and bleached cotton (polysaccharides) fibers were subjected to enzymatic modification with cellulases. Besides altering fiber wettability,¹² cellulases are capable of altering fiber characteristics such as length, strength and the concentration of reducing ends on its surface. These parameters might affect the hydroentanglement behavior of the fibers as well as fabric strength and drapability. Therefore, the effort to investigate the influence of abovementioned parameters was undertaken. Moreover, to minimize the strength loss in fibers and thus nonwovens, the cotton samples were treated with CBD-free EGs and its effect on the properties of nonwoven was compared to a commercially available multi-component cellulase solution.

EXPERIMENTAL

Materials

Raw and bleached cotton fibers supplied by Cotton Inc. (Cary, NC) were used in this work. Fibers were rinsed and dried to eliminate impurities. The raw cotton was additionally boiled in water at 100°C for 30 min to remove the wax content associated with the fibers. The resulting raw and bleached cotton fibers had a mean fiber length of 18.2 and 11.7 mm with mean fiber tenacities of 177N and 185.4N m g⁻¹, respectively.

Two cellulase solutions, a cellulase mixture, Cellu-soft L, and a monocomponent solution of CBD-free

Endoglucanases (NS 29,050) from Novozyme NA (Franklinton, USA), were used in this work. These cellulases with activities of 750 and 5000 EGU g⁻¹, respectively, were secreted by *Trichoderma reesei* microorganism.

Enzymatic treatment and fabric formation

Bleached and raw cotton fibers were treated with enzymes as indicated in Table I in an incubator shaker (Amerex, USA) with gentle agitation at a temperature of 50°C.

Prior to each experimental run, the fibers were dried and cooled in a desiccator. To avoid interference caused by fiber swelling during hydrolysis, the cotton samples were soaked for 15 h in 150 mL of 50 mM sodium acetate buffer. The pH of the buffer was adjusted to optimum values of 5 or 6.75 for Cellu-soft L and EG solutions, respectively.

To start the enzymatic reaction, a designated dose of enzyme solution was added to slurries preheated to 50°C. After a given time, the enzymolysis was terminated by immediate filtering of the reaction solution and fast immersion of the fibers into hot water at a temperature of 80°C for 10 min. After rinsing with deionized water, cotton samples were squeezed and placed in an oven at (105 ± 2)°C for drying.

Fiber webs with a basis weight of about 60 g m² were then formed with a Hollingsworth carding machine and hydroentangled at identical operating conditions. All samples were passed 4 times through 3 manifolds with a pressure setting of 50 bars.

Characterization

The reducing ends or reducing power of fiber samples after enzymatic treatment was measured with the Formazane method^{13,14} with glucose as a standard. In this method reducing end group of cotton fibers is determined by reaction with triphenyltetrazolium chloride. This reagent forms red methanol-soluble compound, which can be quantified by measuring color intensity.

Tenacity of cotton fiber was measured with Favimat (Textechno, USA) according to ASTM D3822. The tensile strength results were the arithmetic mean of at least 20 fibers per sample. The Advanced Fiber Information System (AFIS) (Zwellerger Uster, Switzerland) was used to assess the length of raw and bleached cotton fibers after enzymolysis.

The effects of enzymatic treatments and hydroentangling on the morphology of raw and bleach cotton fiber surface were examined by scanning electron microscopy (SEM). The micrographs were obtained on a Hitachi S-3200N microscope. Before each measurement, the specimens were coated with gold using a Samsputter-2A Sputter Coater.

TABLE I
Process Conditions for Enzymolysis of Bleached and Raw Cotton Fibers

Cotton	Substrate concentration (% w/v)	Enzyme concentration (% owf)	Reaction time (min)	Labeled as
Cellusoft L				
Raw	1.6	0.25	30	Cell 1.6-0.25-30
	1.6	0.25	120	Cell 1.6-0.25-120
	1.6	1	30	Cell 1.6-1-30
	1.6	1	300	Cell 1.6-1-300
Bleached	1.6	0.25	120	Cell 1.6-0.25-120
	2.2	0.25	120	Cell 2.2-0.25-120
	1.6	0.75	300	Cell 1.6-0.75-300
CBD-free EG solution				
Raw	1.6	0.25	60	EG 1.6-0.25-60
	1.6	0.75	60	EG 1.6-0.75-60
	1.6	0.75	120	EG 1.6-0.75-120
Bleached	1.6	0.25	15	EG 1.6-0.25-15
	2.2	0.25	15	EG 2.2-0.25-15
	1.6	0.25	300	EG 1.6-0.25-300

Nonwoven breaking strength was determined on fabric strips (2.5×15.2 cm) according to ASTM D 5035 with minimum 15 tests per sample. Based on this test, the fabric tenacity was calculated as follows:¹⁵

$$\text{Fabric tenacity} = \frac{0.00981F}{0.0254EW} \quad (1)$$

where F is breaking force (gf), E is elongation (in), and W is basis weight (g m^{-2}).

Bending stiffness test (Cantilever bending method) was performed on the strips of fabrics (2.5×15.2 cm) with minimum 15 tests per sample. Bending modulus used as a measure for fabric drapeability, Q , was then calculated as follows:¹⁵

$$Q = \frac{Wl^3}{1/2 \times t^3} \quad (2)$$

where l is bending length (m) and t is fabric thickness (m).

RESULTS AND DISCUSSION

Preliminary tests

A series of experiments was conducted to investigate the effect of Cellusoft L and EG enzyme solutions of the properties of cotton (polysaccharides) fibers and identify the preferable conditions for the fiber pretreatment prior the fabric formation.^{16,17} These runs were carried out within the cotton and enzyme concentration ranges of 0.8%–2.2% (w/v) and 0–1% on weight of fibers, respectively. The experiments showed that raw cotton fibers were subjected to a more damage by enzymes than bleached. Moreover, Cellusoft L solution being aggressive for raw cotton

was found to suitable for the pretreatment of bleached.¹⁶ Based on these data, the conditions (Table I) were selected for the cotton fiber pretreatment.

Sections below describe the properties of nonwoven fabric composed of enzyme treated cotton fibers. During our testing, it was observed that the properties of these fabrics followed a similarly trend in both machine and cross-machine directions. Therefore, the Tables and Figures reported below reflect the data obtained for machine direction only.

Raw cotton nonwovens

The tensile and bending properties of nonwovens formed from untreated, Cellusoft L and CBD-free EGs pretreated raw cotton fibers are shown in Figures 1 and 2. As can be seen in Figure 1, the tenacity of cotton nonwoven was improved by 43% after raw cotton fibers were treated at 1.6-0.25-30 (for keys see Table I).

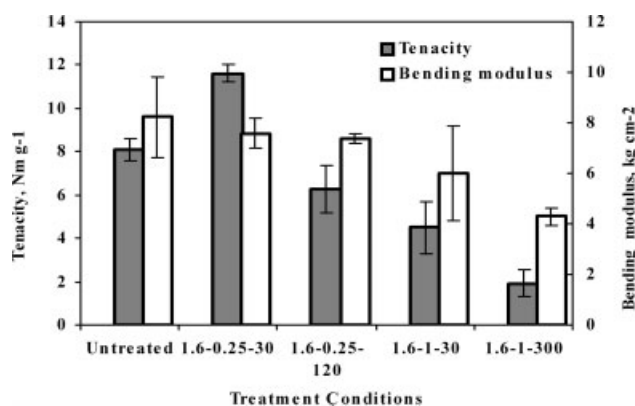


Figure 1 Bending modulus and tenacity of nonwoven fabrics composed of Cellusoft L treated raw cotton fibers at different process conditions: reaction time and cellulase concentration.

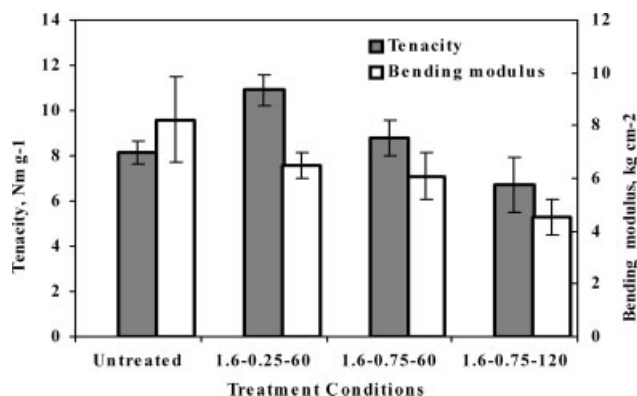


Figure 2 Bending modulus and tenacity of nonwoven fabrics composed of CBD-free EG treated raw cotton fibers at different process conditions: reaction time and cellulase concentration.

However, an increase in reaction time and cellulase concentration decreased the strength of cotton nonwoven. For instance, treatment of raw cotton fibers for 300 min at 1% owf of Cellusoft L lowered the fabric strength by 76.2% in comparison with the untreated fabric. As for the bending modulus, it decreased by only 7% at 1.6-0.25-30. With prolonged treatment time and high concentration of enzymes the drapeability of nonwoven substrate improved further but at the expense of the fabric strength.

Similar trends were observed for the pretreated fabric with CBD-free EGs (Fig. 2). An increase of 35% in strength of nonwoven was observed when the reaction was carried out for 60 min and with the enzyme concentration of 0.25% owf. The bending modulus however lowered by 20% at the same conditions inferring an improvement in the drapeability of the nonwoven fabric. Further increase in reaction time and enzyme concentration did improve the drapeability of the fabric without significant decrease in the strength of the fabric.

To isolate the effects of fiber length, strength and reducing ends after cellulase treatment on the proper-

ties of fabric, the fabric tensile data were divided into two groups: improved or decreased in comparison to untreated sample (Table II). This Table displays the decrease in the fiber characteristics with a minus sign. Among seven fabrics listed, only one of them consisted of fibers with extensive deterioration in their strength, 53%. This in turn led to the lowest tensile strength observed between the studied nonwovens (Fig. 1 and Table II). The rest of the fabrics contained enzymatically treated fibers with strength loss of up to 25.8%. However, an increase in fabric tenacity of 35–43% was observed when the tenacity of single cotton fiber was not reduced by more than 7.8%. Fabrics with the fibers shortened by fragmentation by about 3.8% also exhibited higher strength than the untreated fabric. A further decrease in fiber length caused a deterioration of tensile strength of the nonwoven. Nevertheless, there are two nonwovens composed of raw cotton fibers with similar strength and mean length but were placed into different sections of Table II. These are EG 1.6-0.75-60 and EG 1.6-0.75-120 (for keys see Table I). The major difference lies in the concentration of the reducing ends of enzymatically treated cotton fibers used for these nonwovens. These groups can form hydrogen bonds with hydroxyl groups of neighboring fibers,¹⁸ thus increasing the tenacity of nonwoven fabric. In the case of EG 1.6C-0.75E-60 fabric, the reducing power of cotton fibers increased up to 34.4% after enzymatic process and only by 8.6% for EG 1.6-0.75-120. This could be a reason for EG 1.6-0.75-60 fabric to be stronger than EG 1.6-0.75-120.

Bleached cotton nonwovens

Bleached cotton fibers were treated with the same cellulase solutions as the raw fibers (Table I). Our earlier experiments¹⁶ showed that bleached cotton fibers were generally more resistant to cellulase action than raw cotton. This fact is also reflected in the properties of nonwovens composed of enzymatically-modified

TABLE II
Characteristics of Raw Cotton Fibers After Enzymolysis with Cellusoft L and CBD-free EGs, and Tenacity of Nonwovens Made of These Fibers

Conditions	Enzyme	Changes (%)			Fabric tenacity (Nm g ⁻¹)
		Fiber tenacity	Length	Red. ends	
			Higher		
1.6-0.25-30	Cellusoft L	-7.8	-3.2	20.5	11.6
1.6-0.25-60	CDB-free EG	-7.8	-2.7	18.7	10.9
1.6-0.75-60	CDB-free EG	-24.4	-3.8	34.4	8.8
			Lower		
1.6-0.75-120	CDB-free EG	-25.8	-4.3	8.6	6.7
1.6-0.25-120	Cellusoft L	-24.8	-5.4	33.7	6.3
1.6-1-30	Cellusoft L	-14.9	-6.0	17.8	4.5
1.6-1-300	Cellusoft L	-53	-25.0	14.3	1.9

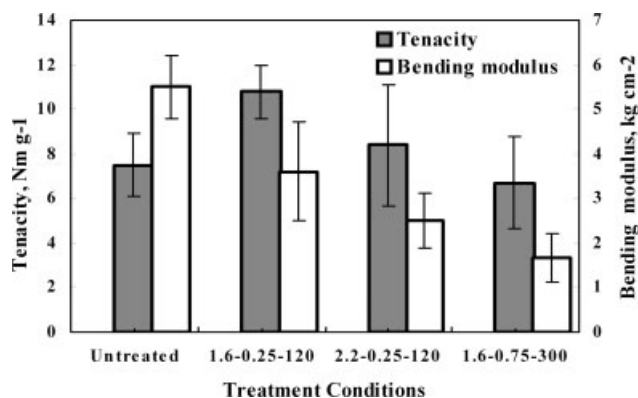


Figure 3 Bending modulus and tenacity of nonwoven fabrics composed of Cellusoft L treated bleached cotton fibers at different process conditions: reaction time, cellulase and cotton concentrations.

cotton. The tensile and bending properties of these fabrics are depicted in Figures 3 and 4. A comparison of nonwovens formed from raw and bleached cotton fibers treated with Cellusoft L at the 1.6-0.25-120 conditions (for keys see Table I) showed that bleached cotton nonwoven possessed better tensile properties. Moreover, with the increase in the reaction time and enzyme concentration, the tenacity of this type of nonwovens dropped not as rapid as it was observed with raw cotton fabric. This effect can be attributed to a difference in the activity of cellulase enzymes towards these two types of cotton fibers. Treatment of raw cotton with Cellusoft L caused strong fragmentation of fibers,¹⁷ whereas the length was unaffected by enzymolysis of bleached cotton fibers, though the strength of single fiber decreased.¹⁶ This suggests that cellulases could only cause scissions on the fiber surface, thus making fibers more flexible and subsequently nonwovens too. The SEM micrographs of enzymatically modified bleached cotton fibers confirm this conclusion (Fig. 5).

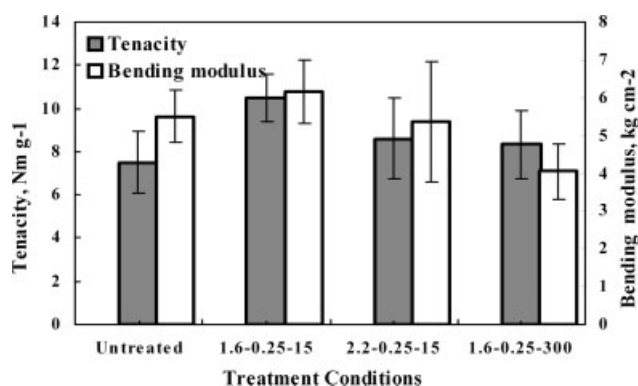


Figure 4 Bending modulus and tenacity of nonwoven fabrics composed of CBD-free EG treated bleached cotton fibers at different process conditions: reaction time, cellulase and cotton concentration.

The CBD-free EGs showed a less drastic effect on the properties of bleached cotton as well as on the properties on nonwoven fabric (Fig. 4). Among the three fabrics prepared from enzymatically treated fibers, all of them had tenacity higher than untreated fabric. The bending modulus however increased to a maximum of 6.4 kg cm^{-2} .

To investigate the effect of pretreated bleached cotton fibers on the properties of nonwovens, the fabrics were segregated based on their strength relative to the un-pretreated nonwovens; the same way as it was done for raw cotton nonwovens. Since fiber mean length of bleached cotton did not change during enzymatic hydrolysis, the "length" factor was omitted and the results are shown in Table III.

As may be noted from the Table, only one nonwoven had tensile strength lower than that of the untreated fabric and it contained fibers that lost up to 49.5% of their original strength. The other five fabrics improved in tenacity. Among them, the decrease in single fiber strength of 1.4% resulted in increase in tenacity to 10.5 N m g^{-1} after hydroentangling (Table III). A further decrease in fiber strength resulted in a slight decrease in fabric tenacity with the exception of the "Cell 1.6-0.25-120" sample. This fabric consisted of fibers that lost 27% of their original strength had the tenacity of 10.8 N m g^{-1} . The assessment of reducing ends for the fibers used in Cell 1.6-0.25-120 nonwoven showed its increase by 45%. This is the highest observed value between the tested fibers and it also could mean that the strength of this nonwoven was improved by the formation of hydrogen bonds (via reducing ends) between fibers in the web.

Since the mean fiber length of bleached cotton was not affected by enzymatic hydrolysis,¹⁶ the drapeability of the fabric, that is, bending modulus, of EG and Cellusoft L pretreated nonwovens can be compared. For this purpose the Cell 2.2-0.25-120 and EG 1.6-0.25-300 fabrics are considered as these nonwovens con-

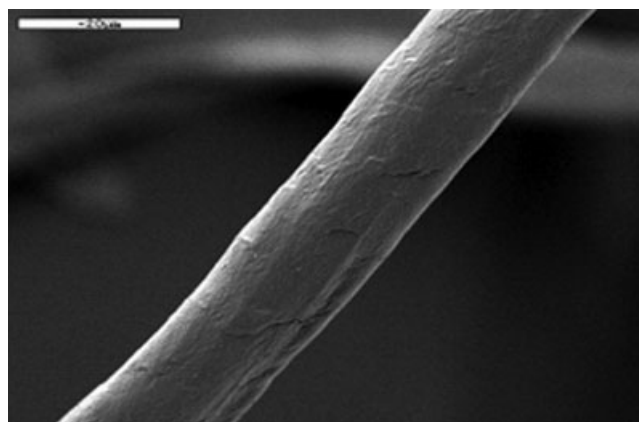


Figure 5 Scanning electron micrographs of the surface of cotton fibers treated with cellulase solutions.

TABLE III
Characteristics of Bleached Cotton Fibers After Enzymolysis with Cellusoft L and CBD-free EGs, and Tenacity of Nonwovens Made of These Fibers

Conditions	Enzyme	Changes (%)		Fabric tenacity (Nm g ⁻¹)
		Fiber tenacity	Red. ends	
1.6-0.25-15	CDB-free EG		Higher	10.5
		-1.4	17	
1.6-0.25-120	Cellusoft L	-27.2	45	10.8
2.2-0.25-15	CDB-free EG	-4.0	5.5	8.6
2.2-0.25-120	Cellusoft L	-28.6	9.7	8.4
1.6-0.25-300	CDB-free EG	-28.2	4.7	8.3
1.6-0.75-300			Lower	6.7
	Cellusoft L	-49.5	14.6	

sisted of fibers that lost about 28% of their strength during their hydrolysis and had small differences in reducing power. These nonwovens also had similar tenacity values after hydroentangling. However, their bending moduli were found to be different. The nonwoven formed from Cellusoft L pretreated fibers exhibited a lower bending modulus, about 2.5 kg cm⁻² compared to 4.1 kg cm⁻² obtained for EG treated nonwoven substrate. This difference can be attributed to the mode of action of these two cellulase solutions. Cellusoft L, the whole cellulase, is able to attack cotton fiber more or less uniformly, hydrolyzing amorphous and crystalline regions, whereas the CBD-free EG solution, predominantly attack amorphous regions.⁵ The ability of Cellusoft L enzymatic system to efficiently hydrolyze all the regions of the cotton fiber led to more effective decrease of bending modulus of resulting nonwoven.

Effect of hydroentangling process

The changes in the nonwoven fabric properties were caused however, not only by the quality of modified cotton fibers but also by the bonding process following the web formation stage. To investigate the effect of water jets on the cotton fibers, the nonwovens were imaged with SEM and several micrographs are presented in Figure 6.

The images reveal that the nonwovens made of enzyme-treated fibers had more cracks present on the fiber surface than those containing untreated fibers [Fig. 6(a,b)]. The appearance of cracks was not widespread for the cotton fibers pictured right after enzymatic process (Fig. 5). We believe that the impact force of the water jets triggered the cracks caused by the enzymes to propagate and visibly increase in size. This also resulted in the fibrillation of some cotton fibers [Fig. 6(c)] which could further entangle around of fiber causing increase in fabric strength.

The changes in fiber characteristics, that is, cracks of different sizes and fibrillation, might have a significant impact on the tensile properties of the nonwoven

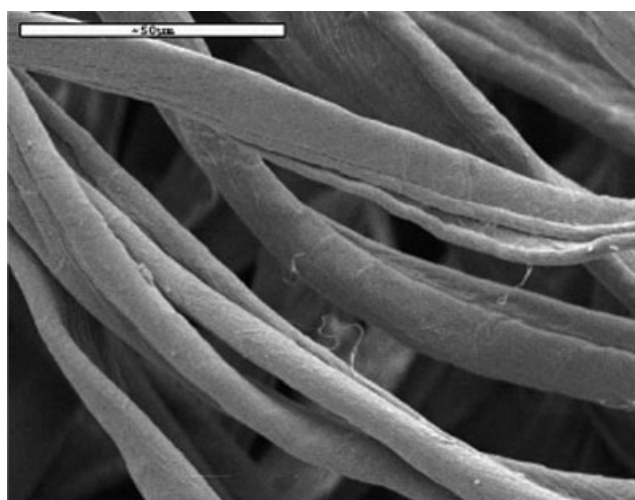
fabric. Although we observed a decrease in single fiber tenacity, the increased water adsorption and flexibility of the cotton fibers after enzymolysis¹⁹ could also cause them to entangle more efficiently and contribute to the observed increase in the tenacity of the nonwoven fabric.

Comparison enzyme-treated nonwovens

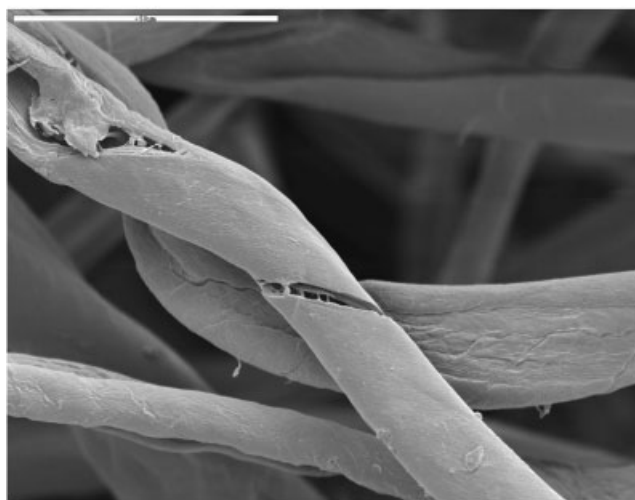
Finally, to analyze the effectiveness of the enzymatic treatment of cotton fibers prior to nonwoven formation, tenacities of nonwovens made of as-received cotton fibers and then treated with the cellulase solutions were compared to the ones prepared from the enzymatically-modified fibers. The conditions and tenacities of the resulting nonwovens are shown in Table IV. As can be seen from this Table, the tenacity of fiber pretreated nonwoven fabrics increase with both Cellusoft L or CBD-free EGs, whereas the treatment of nonwoven fabrics with enzymes after the hydroentangling process caused the tenacity of fabric to decrease or remain unchanged. Application of Cellusoft L however, triggered more damage to the structure of nonwoven substrate. The strength of the raw and bleached cotton nonwoven fabrics dropped by 52 and 31%, respectively. The main reason of this effect on enzyme treated fabric can be attributed to a looser structure of nonwovens and weaker fiber entanglement after enzymatic post-treatment.

CONCLUSIONS

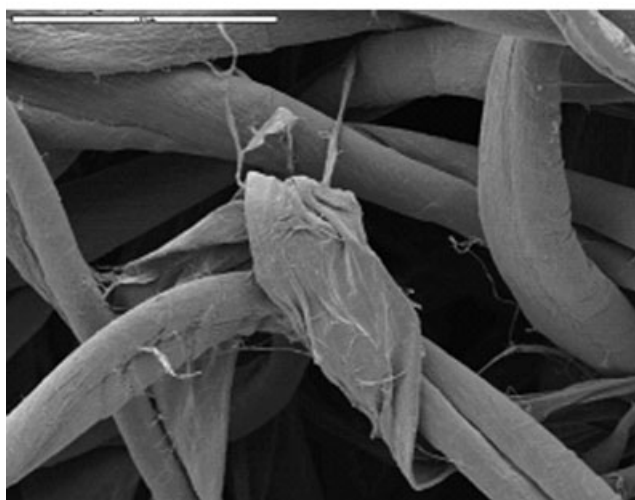
A series of experiments were conducted to investigate the effect of fiber modification with cellulase on the properties of hydroentangled nonwoven fabrics. Our results indicate that careful design of enzymatic pretreatment process could lead to an improved drapability and an increase in strength of cellulosic nonwovens, although a general weakening of the cotton fiber occurred. The following sequence of fiber characteristics, affected by enzymatic pretreatment, might influence on the tensile properties of resulting non-



(a)



(b)



(c)

Figure 6 Scanning electron micrographs of the surface of cotton fibers after hydroentangling: (a) untreated cotton fiber, (b) treated with cellulase, (c) fibrillation of cellulase treated fiber.

TABLE IV
Fabric Tensile Properties of Raw and Bleached Cotton Nonwovens After Enzymatic Treatment of Fiber or Fabric

	Tenacity (Nm g ⁻¹)	
	Raw	Bleached
Untreated	8.1 ± 0.5	7.5 ± 1.4
Cellusoft L	1.6-0.25-30 ^a	1.6-0.25-120
Fabric treated	3.9 ± 0.9	5.2 ± 1.1
Fiber treated	11.6 ± 0.4	10.8 ± 1.2
EG solution	1.6-0.25-60	1.6-0.25-15
Fabric treated	6.2 ± 0.7	7.5 ± 1.8
Fiber treated	10.9 ± 0.7	10.5 ± 1.1

^a Keys: "1.6-0.25" is attributed to 1.6% (w/v) of cotton fibers and 0.25% owf of cellulases; the last number is the reaction time in minutes.

woven fabric: tenacity > mean length > reducing ends.

Fiber modification with cellulases, Cellusoft L and CBD-free EGs, revealed that Cellusoft L had more drastic effect on the properties of cellulosic nonwovens. Extensive treatment, that is, prolonged reaction time and high concentration of enzymes, caused a decrease in strength of the nonwovens. Nevertheless, Cellusoft L, being more aggressive than CBD-free EGs, was more suitable for the treatment of bleached cotton fibers prior hydroentangling. Whereas, CBD-free EGs were more efficient on raw cotton fibers. At certain conditions of pretreatment, the bending modulus of bleached cotton nonwovens was improved without sacrificing the fabric tenacity. The comparison with the fabrics after postenzymatic treatment showed that the enzymatic modification of cotton fibers prior to hydroentangling process could be a possibility for obtaining stronger and more drapeable nonwoven fabrics.

The authors thank Novozymes and Cotton, Inc., for providing materials for this study.

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