Enzymatic Retting of Kudzu Fibers

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ABSTRACT: The enzymatic retting of kudzu fibers was performed with several commercial enzymes, and the effects on the retting were compared with respect to the smoothness of the surface of the fibers and the mechanical properties. The commercial enzymes were classified into two types, that is, cellulase and pectinase. For the cellulase type, enzymatic decomposition occurred almost topochemically because of the cooperation of cellulase with high activity, and then the retting was fully achieved, suppressing the damage to the intercellular matrix (middle lamella) by pectinase. For the pectinase type, decomposition predominately occurred in the middle lamella joining the adjacent fibrous cells. Therefore, the tensile properties of the retted fibers were lowered. In the retting of kudzu fibers, a topochemical process is promising for producing fibers with excellent luster that retain the tensile properties. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 106: 2759–2762, 2007

Key words: biopolymers; enzymes; fibers; mechanical properties; processing

INTRODUCTION

Kudzu, or *Pueraria lobata*, is classified as part of the legume family and is native to Japan and China. The stems are applicable to fiber industries, and kudzu fibers removed from the bast ribbons of the stems are used in making cloth and paper. Yamashita et al.[1] reported the fundamental fiber structure for traditionally water-retted kudzu fibers. Recently, Luo et al.[2] tried to use kudzu fibers as reinforcing materials for composites. When we design apparel, the luster of the fibers is one of the most important elements. In the case of cotton fibers, to enhance the luster, the fibers are mercerized with a caustic soda solution. Among cellulosic fibers, kudzu fibers can provide a specific luster. Such luster will be possibly enhanced by the mercerization treatment. In the case of kudzu fibers taking a multicellular structure, however, a strong chemical will attack the weak intercellular regions, and then the mechanical properties of the fibers will tend to be lowered.[3] Therefore, a retting procedure becomes a key process to producing high-quality kudzu fibers with authentic luster.

Water retting has been traditionally used for the production of kudzu fibers. This retting process depends on the microorganic activities of bacteria living in the daily environment. Therefore, this method is not suitable for the production of high-quality fibers because of the difficulty of controlling microorganic activities.

In this study, several commercial enzymes were prepared, and the effects on retting were compared with respect to the smoothness of the surface of the retted fibers and the mechanical properties.

EXPERIMENTAL

Materials

Kudzu plants grown at a farm of the Kyoto Institute of Technology were harvested from July to August in 2005. Stems 2–3 m from the growing tip were decorticated by hand immediately after the harvest, and the sheath parts containing bast ribbons were stocked in a freezer and used in this study.

The commercial enzymes used in this study were viscozyme L (Novo Nordisk, Chiba City, Japan), ultrazyme 100G (Novo Nordisk), pectinase G (Amano, Nagoya City, Japan), pectinase PL (Amano), and hemicellulase 90 (Amano). The compositions are as follows. Viscozyme L is a blend of pectinase, cellulase, hemicellulase, β-glucanase, arabanase, and xylanase. Ultrazyme 100G and pectinase G basically consist of pectinase from different origins. Pectinase PL is a blend of pectinase and cellulase. Hemicellulase 90 is a blend of pectinase, cellulase, xylanase, and β-glucosiderase. The properties of the enzymes are summarized in Table I.[4]

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TABLE I
Properties of the Enzymes

Code	Activity (u/g)	Substrate	Optimum pH	Optimum temperature (°C)
a	1×10^2	β-Glucan	3.3–5.5	25–55
b	8×10^3	Pectin	3.5–5.5	40–60
c	1.2×10^3	Pectin	3.5–5.0	50–60
d	5×10^4	Pectin	3.5–5.5	50–65
e	9×10^4	Hemicellulose	3.5–6.0	30–55

a = viscozyme L; b = ultrazyme 100G; c = pectinase G; d = pectinase PL; e = hemicellulase 90. The activities were estimated against each substrate.

Enzymatic retting

The separated sheath parts containing bast ribbons (ca. 6 g) were first soaked in a 0.1*M* sodium acetate buffer (pH 4.5) for 30 min at a room temperature, and then the swollen ribbons were put into a solution of each enzyme (5 g/L) prepared with a similar acetate buffer. The retting was performed for 2–8 h at 50–55°C, and a liquor ratio of 17:1 was maintained. After the retting, the fibers, removed from the bast ribbons, were boiled for 20 s and then rinsed in water.

Water retting

The separated sheath parts containing bast ribbons (ca. 30 g) were soaked in water (500 mL) for 1 week at room-temperature in shaded sunlight. After soaking, the partially separated fiber bundles were rubbed between the thumb and forefinger in a water stream, and then the residual cell walls of parenchyma were removed from the fiber bundles.

The separated sheath parts containing bast ribbons were also boiled in water for 30 min to enhance the degree of swelling, and then the effect of swelling on the water-retting process was investigated.

Scanning electron microscopy (SEM)

The surfaces of the retted fibers were coated with gold and examined with a scanning electron microscope (S-3000N, Hitachi, Hitachi City, Japan).

Regular reflections

The retted fibers were aligned on a sample stage of 2×4 cm^2, and the surfaces of the retted fibers were exposed to an incident beam set at 15° from the horizontal. Then, the intensities of the regular reflections parallel and perpendicular to the fiber axis were measured with a UGV-5D (Suga Shikenki, Tokyo, Japan). The regular reflection value depended on the smoothness of the retted fibers. The values were the averages of at least seven tests.

Tensile tests

The tensile tests of the retted fibers were performed at 20°C and 65% relative humidity with a Tensilon A&D (Tokyo, Japan) type STA-1150 automatic tester. The gauge length was 10 mm, and the crosshead speed was 0.5 mm/min. The values were the averages of at least40 tests.

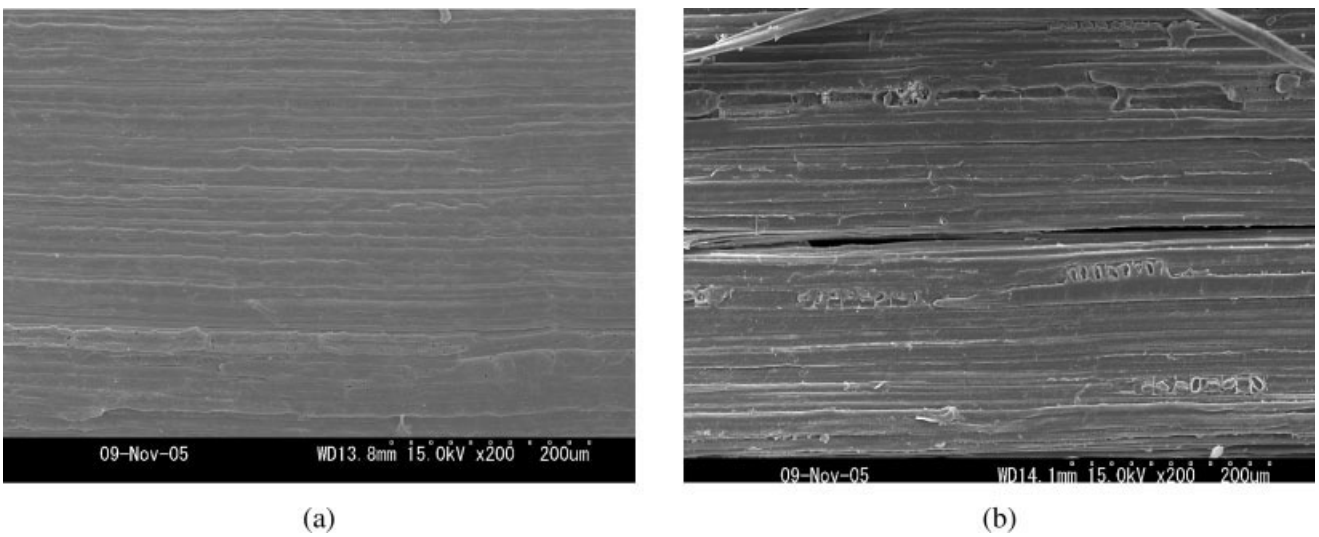

Figure 1 SEM images of water-retted kudzu fibers: (a) without the preboiling treatment and (b) with the preboiling treatment.

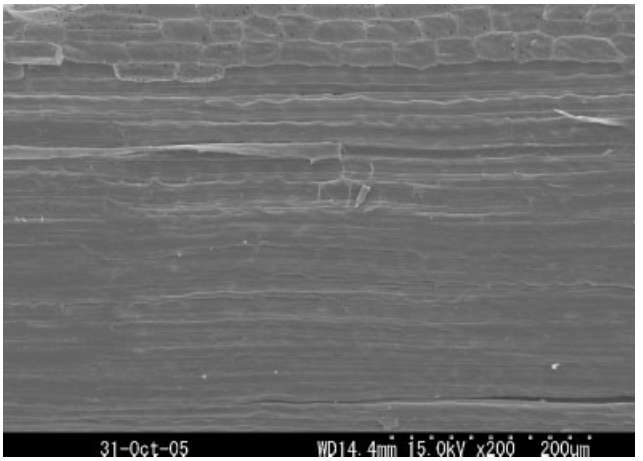

Figure 2 SEM image of kudzu fibers retted with viscozyme L for 8 h.

RESULTS AND DISCUSSION

Surface morphology

Figure 1 shows SEM images for the water-retted kudzu fibers. A smooth surface can be observed for the fibers without the preboiling treatment [see Fig. 1(a)]. The enhancement in the swelling seems to have advanced the hydrolysis of the intercellular matrix by microorganic activities of the bacteria. The many striations along the fiber axis in Figure 1(b) are the scars of the removed intercellular matrix. However, the tensile properties have not been affected by the preboiling treatment (shown later in Table IV). It seems that the scars have not propagated into the inner parts of the fibers.

As for the enzymatic retting process, the surface of the retted fibers becomes smoother with increasing retting time. Figure 2 shows the surface of fibers retted with viscozyme L for 8 h. The residual cell walls of parenchyma have been almost completely removed from the fiber bundles. Similar SEM images were obtained for retting with other commercial enzymes, except for ultrazyme 100G. The treatment time of 8 h is enough for enzymatic retting to produce kudzu fibers with a smooth surface, whereas traditional water retting takes 1 week.

Luster of the retted fibers

To estimate the luster of the retted fibers, the regular reflection was measured. If the residual cell walls of parenchyma remain on the surface of the fibers, they will produce shadows against an incident beam, and then the regular reflection will be lowered. Tables II and III show regular reflections measured parallel and perpendicularly to the fiber axis, respectively. The values for the fibers retted for 8 h with viscozyme L are superior to those for other fibers in both directions. As shown in Table III, the value for fibers obtained via water retting accompanied by preboiling is smaller than that for fibers only water-retted. This is due to the shadows produced by the scars along the fiber axis [see Fig. 1(b)].

Tensile properties

The tensile properties of the retted fibers are summarized in Table IV. In comparison with the tensile strengths for water-retted fibers, the values for the enzymatic retted fibers are extremely smaller, except for viscozyme L and ultrazyme 100G. As for ultrazyme 100G, the regular reflection becomes smaller than those of other retted fibers. It seems that the residual cell walls of parenchyma still remain on the surface of the fibers; that is, the retting process is not fully achieved. As shown in Table I, the activity of ultrazyme 100G is much lower than the value for pectinase PL and is in the same order for pectinase G. However, pectinase G is a refined and produced from *Aspergillus pulverulentus*. Therefore, the retting effects become different, and then the strength of fibers retted with ultrazyme 100G stands at the same level as that of water-retted fibers.

The activities of enzymes are greatly influenced by the pH as well as the temperature of the retting bath. In the case of viscozyme L, cellulase shows higher activity against glucan than with the other enzymes used in this study at pH 4.5.[4] Thus, the fragile cell

TABLE II
Regular Reflections Measured Parallel to the Fiber Axis

Treatment time (h)	Regular reflection (au)						
	a	b	c	d	e	f	g
2	0.25	0.31	0.32	0.32	0.37		
4	0.43	0.60	0.60	0.39	0.52		
6	1.03	0.88	0.64	0.56	0.83		
8	1.35	0.89	1.16	1.00	1.14		
168						1.05	1.00

a = viscozyme L; b = ultrazyme 100G; c = pectinase G; d = pectinase PL; e = hemicellulase 90; f = water retting with preboiling treatment; g = water retting. The incident beam angle was set at 15° from the horizontal.

TABLE III
Regular Reflections Measured Perpendicularly to the Fiber Axis

Treatment time (h)	Regular reflection (au)						
	a	b	c	d	e	f	g
2	0.21	0.30	0.29	0.28	0.36		
4	0.35	0.59	0.56	0.32	0.45		
6	0.96	0.74	0.53	0.51	0.79		
8	1.38	0.80	1.06	0.98	1.01		
168						0.84	1.00

a = viscozyme L; b = ultrazyme 100G; c = pectinase G; d = pectinase PL; e = hemicellulase 90; f = water retting with preboiling treatment; g = water retting. The incident beam angle was set at 15° from the horizontal.

walls of parenchyma attached to the bast fiber bundle will be almost directly hydrolyzed by viscozyme L. On the one hand, other commercial enzymes contain pectinase as their major component. Therefore, it is difficult to attack the cell walls directly. According to the measurements for the chemical composition of kudzu fibers, the fibers contain 13.9% pectin, which is a little more than that of other commercial bast fibers.[1] Pectin is one of the major components of an intercellular matrix.[5] For bast fibers, the matrix works as the jointing material between adjacent fibrous cells. Therefore, if the matrix experiences enzymatic hydrolysis decomposition, the damage will be reflected in a reduction of the tensile properties of the retted fibers. We have divided the commercial enzymes into two types, that is, cellulase and pectinase. The mechanism for each type is represented in Figure 3. For the cellulase type, that is, viscozyme L, enzymatic decomposition occurs from the surface of parenchyma and proceeds almost topochemically because the cellulase with high activity and pectinase work together, and then the retting is fully achieved, suppressing the damage to the intercellular matrix (middle lamella) by pectinase. For the pectinase type, that is, pectinase G, pectinase PL, and hemicellulase 90, enzymatic decomposition predominately occurs in the middle lamella and will degrade the intercellular matrix, leading to the decrease in the tensile properties. As shown in Table IV, the tensile properties for the fibers retted with the pectinase type are lower than those for the water-retted fibers. In the case of ultrazyme 100G, the smaller values for the regular reflection suggest that the retting process us not fully achieved (see Tables II and III). Therefore, the tensile strength is not as lowered.

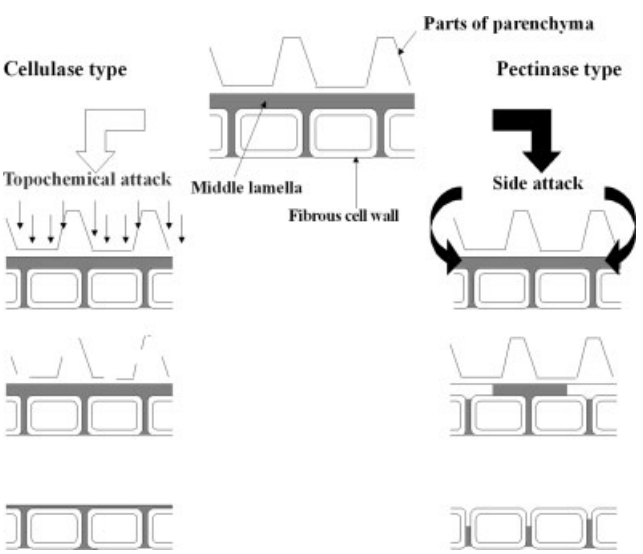

Figure 3 Schematic representation of the enzymatic retting process.

TABLE IV
Tensile Properties of the Retted Kudzu Fibers

	Code						
	a	b	c	d	e	f	g
Strength (N/tex)	0.26	0.27	0.13	0.18	0.17	0.27	0.27
Elongation (%)	4.13	3.15	1.97	3.03	1.52	4.20	3.32

a = viscozyme L; b = ultrazyme 100G; c = pectinase G; d = pectinase PL; e = hemicellulase 90; f = water retting with preboiling treatment; g = water retting.

The process reported in this article is applicable to other bast fibers with lower lignin fractions. Kudzu fibers contain 9.1% lignin. In the middle lamella, the lignin constructs a lignin–carbohydrate complex.[6] Such a rigid structure will show resistance against an enzymatic reaction. Joko et al.[7] reported that biological retting with viscozyme L is not effective for jute fibers, which contain 20–25% lignin.[8] Therefore, this enzymatic retting process will be effective for bast fibers with smaller lignin fractions, such as flax and ramie.[8]

CONCLUSIONS

The enzymatic retting of kudzu fibers has been performed with several commercial enzymes, and the effects on the retting have been compared with respect to the smoothness of the surface of the fibers and the mechanical properties. In the retting of kudzu fibers, a topochemical retting process, which is brought about by the cooperation of active cellulase, is promising for producing fibers with excellent luster without reducing tensile properties.

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