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Enzyme enhanced solid-state fermentation of kenaf core fiber for storage and pretreatment

P.T. Murphy [a,*], K.J. Moore [b], T.L. Richard [c], C.J. Bern [a]

[a] Department of Agricultural and Biosystems Engineering, 100 Davidson Hall, Iowa State University, Ames, IA 50010, USA
[b] Department of Agronomy, 1567 Agronomy Hall, Iowa State University, Ames, IA 50010, USA
[c] Department of Agricultural and Biological Engineering, 225 Ag. Engr. Building, Pennsylvania State University, University Park, PA 16802, USA

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Abstract

Kenaf is an annual fiber crop adaptable to a wide range of climates and soil types. This study investigated the use of kenaf core fiber as a feedstock for enzyme-enhanced fermentation. Triplicate kenaf core fiber samples were treated with enzymes having cellulase:hemicellulase activity ratios of 0:1, 0.015:1, 0.45:1, and 2.54:1 at a rate of 5010 IU/kg dry matter hemicellulase activity, vacuum-sealed, and incubated at 37 °C for 21 d. Samples were analyzed for pH, water soluble carbohydrates, organic acids, and hemicellulose and cellulose concentrations. All treatments produced a pH less than 4.0, which is sufficient for stable storage. Treatments with 2.54:1 and 0.45:1 produced the highest water soluble carbohydrate and lactic acid concentrations. Enzymes with no or low cellulase activity produced results similar to the control. Utilizing enzyme mixtures with high cellulase activity is an effective pretreatment method for ensiled kenaf core fiber.

Keywords: Kenaf; Ensilage; Storage; Pretreatment; Fermentation

1. Introduction

1.1. Kenaf

Kenaf (*Hibiscus cannabinus* L.) is a warm-season annual fiber crop mainly adapted for cultivation in tropical and sub-tropical areas. It is native to parts of Africa and is an important cordage crop in many developing countries. Closely related to cotton, okra, and hibiscus, kenaf is similar in appearance to hemp. Kenaf can be grown in a wider range of climates and soil types than any other commercial fiber crop (Dempsey, 1975). Most production in the United States has been limited to Mississippi, Texas, Florida, and California, but it has been grown for fiber production as far north as Iowa. It requires 150 days to complete maturity, so seed production is limited to frost-free areas in Texas, California, and Florida (Sullivan, 2003).

Kenaf is harvested for its stalk, which can reach heights of 2.5–6 m, depending on environmental conditions. The stalk is composed of two distinct fibers, bast and core, which comprise approximately 35% and 65% of the stalk mass, respectively (Columbus and Fuller, 1999). Bast is characterized as a bark, containing long fibers, with the core being physically similar to balsawood, containing soft, short fibers.

Since the early 1990s the area planted to kenaf in the United States has increased significantly and during the period from 1992 to 1997, planted area increased from 1600 to 3200 ha (USDA-ERS, 1997). More recent estimates place planted area levels at 6000 ha (NCIS, 2002). Accurate acreage and production values are difficult to establish because most kenaf is grown under contract and kenaf is not part of any current government farm program.

Kenaf is typically allowed to dry in the field, with a killing frost needed to initiate the drying process. This is necessary

* Corresponding author. Tel.: +1 515 231 2081; fax: +1 515 294 6633.
E-mail address: pmurph@iastate.edu (P.T. Murphy).

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because moisture concentrations above 20% moisture content on a wet basis (w.b.) greatly reduce separation efficiency of the bast and core (Columbus and Fuller, 1999). In areas with a mild winter, desiccants or defoliants may be used to hasten drying (Bowyer, 1999). Currently, whole-plant kenaf is harvested with unmodified or slightly modified sugarcane harvesters, forage harvesters and baling equipment, but in cotton production areas, cotton modulation equipment is also used (Webber et al., 2002).

When kenaf is not used for whole plant applications, such as paper pulping or composite building materials, bast and core are mechanically separated after harvest. Each fiber has its own distinct uses, so the greatest economic returns are produced when fibers are separated (Columbus and Fuller, 1999). Bast fiber is considered to be a high quality papermaking material, with chemically refined bast suitable for replacement of softwood pulp in products like newsprint and multiple tissue paper grades (Bowyer, 1999). Core fiber has much lower quality uses, such as oil absorbent, livestock bedding and as a replacement for vermiculite and Styrofoam packaging material (Young, 1992).

1.2. Solid-state fermentation

Because of the limited range and low value of products produced from core material and its high lignocellulose content, core fiber has potential as a biomass conversion feedstock. Fermentation and enzymatic processes have been thought to hold the most potential for conversion of biomass to industrial products, such as acetic acid, acetone, butanol, and lactic acid (NRC, 2000), and fuels, such as ethanol and biodiesel (NRC, 1999). Richard et al. (2001) found the use of solid-state fermentation to be an effective method for pretreatment and limited bioconversion of corn stover in order to improve biomass characteristics for downstream conversion processes.

Solid-state fermentation, when used for biomass storage and pretreatment, is similar to the ensilage technology traditionally utilized by ruminant producers to preserve high fiber feedstuffs for year-round use. Ensilage is referred to as solid-state fermentation because materials are handled as solids as opposed to a fermentation system for ethanol, which is in a liquid phase. Ensilage is characterized as primarily a lactic acid fermentation process. During the initial stage of fermentation, excess oxygen is consumed producing an anaerobic environment. During the second stage, from one day to three weeks, soluble carbohydrates are converted to lactic and acetic acid, ethanol, mannitol, acetaldehyde, and carbon dioxide by anaerobic bacteria (Roberts, 1995). This period is characterized by a significant decrease in pH. After three weeks, significant acetic and lactic acid accumulation result in pH declining to a level which inhibits further microbiological growth ($pH < 4.5$) and the ensilage is considered to be stable. If lactic and acetic acid levels are not sufficient, secondary fermentation will occur. During this process, lactic acid is converted to secondary fermentation products, such as butyrate and iso-butyrate, by clostridia. This process is characterized by an increase in pH and the presence of propionate, butyrate, and iso-butyrate. Secondary fermentation is considered to be detrimental to the ensilage process because butyric acid is a weak acid for preserving silage and its formation causes significant dry matter losses from the silage (Jaster, 1995).

Enzyme additions can be used to enhance the ensilage produced by hydrolyzing structural carbohydrates (hemicellulose and cellulose) into additional fermentable sugars (Jaster, 1995; McDonald et al., 1991). Ren et al. (2006) and Richard et al. (2002) found that enzyme-enhanced fermentation is a suitable pretreatment method for corn stover to be used for further processing. Enzymes have also been demonstrated to improve fermentation in other high lignocellulose materials, like whole-plant wheat (Adogla-Bessa and Owen, 1995), orchardgrass, and alfalfa (Nadeau et al., 2000), but this approach has not been applied to kenaf core fiber.

The purpose of this project was to determine concentrations of fermentation products in enzyme-treated kenaf core fiber preserved using solid-state fermentation, and possible strategies for use of enzymes in a kenaf core fiber ensilage system for storage and pretreatment.

2. Methods

2.1. Experimental design

Whole kenaf plants were harvested from a plot near Brooklyn, Iowa in early December 2003, after a killing frost and subsequent period of field drying. Plants were allowed to dry an additional week indoors to improve bast and core fiber separation. Separation and fractionation of the fibers was done with a portable shredder (MTD Products, Inc.). The resulting material was then sifted and the large bast particles removed, producing a lower quality core fiber, containing small amounts of bast fiber.

The material was adjusted from an initial moisture concentration of 10–15% (w.b.) to 60% by addition of water. Samples were treated with four commercially available enzymes having cellulase:hemicellulase activity ratios ranging from 0:1 to 2.54:1 (Table 1). Enzymes were applied at a hemicellulase rate of 5010 International Units (IU)/kg dry

Table 1
Source and cellulase:hemicellulase activity ratios for enzymes used

Enzyme	Source	Cellulase: hemicellulase activity ratio
MULTIFECT® Xylanase	Genencor International, Inc. (Cedar Rapids, IA)	0:1
ENZECO® Xylanase S 200	Enzyme Development Corporation (New York, NY)	0.015:1
SAFIZYM® FL 300	Saf Agri (Soustons, France)	0.45:1
MULTIFECT® A40	Genencor International, Inc. (Cedar Rapids, IA)	2.54:1

matter. A no-enzyme control was included as an additional treatment, making five total treatments.

For each treatment, triplicate 500-g samples were vacuum-sealed in medium-strength polyethylene bags to insure an anaerobic environment and then incubated at 37 °C. Samples were destructively analyzed at 21 days of incubation and the following analyses conducted: dry matter, pH, water soluble carbohydrates, organic acids, and fiber fractions. Additional triplicate zero day control samples were also analyzed to make comparisons to the initial material.

2.2. Analyses

Dry matter (DM) of the sub-samples was determined by drying 100 g of material at 60 °C in a forced air oven for 72 h (ASAE Standards, 2003). Dried samples were ground using a Wiley mill (Thomas Scientific, Inc.) to pass through a 1-mm screen. Dry matter was also determined for ground samples by drying 1 g of sample at 103 °C in a forced air oven for 4 h to make moisture corrections for water soluble carbohydrates and fiber fraction analyses. pH was measured using a pH electrode. Samples were prepared with a 10:1 mass dilution (H_2O: sample) and allowed to stand for 30 min prior to measurement.

Water soluble carbohydrates (WSC) were determined using a modification of the method described by Guiragossian et al. (1977). Water extracts were prepared by shaking 0.25 g of dried, ground material with 100 ml of distilled water for 30 min. Extracts were filtered through #54 filter paper. To an aliquot of the filtrate, 5% phenol and sulfuric acid was added and the solution's absorbance measured at 490 nm using a spectrophotometer equipped with a rectangular 10 mm light path cell. Sample values were calculated from a standard curve prepared using equimolar concentrations of glucose and xylose to determine carbohydrate levels.

Organic acid concentrations were analyzed using a method similar to that described by Moore et al. (1985). Water extracts were prepared by shaking 50 g of undried sample with 200 ml of water for 4 h, refrigerating for 20 h, and filtering through four layers of cheese cloth. An aliquot of the filtrate was acidified with 25% metaphosphoric acid and centrifuged for 15 min at 15,000 rpm. The resulting supernatant was collected and analyzed using gas–liquid chromatography. Separations of acetate, propionate, butyrate, and iso-butyrate were made using a SP-1200/H_3PO_4 column (Sigma–Aldrich Co.) operated at 120 °C using N_2 as the carrier gas at a flow rate of 30 ml/min with an injection block temperature of 170 °C. Quantification of the acids was done using a flame ionization detector operating at 180 °C. Lactate concentrations were determined using a similar procedure. Separation was done using a SP-1000/H_3PO_4 column (Sigma–Aldrich Co.) with the initial oven temperature set at 100 °C for 1 min and increased to 120 °C at a rate of 10 °C/min. Prior to injection, lactate was methylated by combining 2 ml of filtrate, 4 ml of methanol, and 0.8 ml of 50% aqueous H_2S in a test tube and heating for 30 min at 60 °C (Supelco, 1998). After cooling, 2 ml of water and 2 ml methylene chloride were added to the test tube and the resulting bottom layer of methylene chloride was sampled. All other operating conditions were the same as described above.

Samples were analyzed to determine neutral detergent fiber (NDF), acid detergent fiber (ADF), and acid detergent lignin (ADL) concentrations using the filter bag method (Vogel et al., 1999). Extractions were made sequentially from the same sample. Hemicellulose concentration was expressed as the difference between NDF and ADF, and cellulose concentration as the difference between ADF and ADL. Decreases in hemicellulose and cellulose concentrations during fermentation indicate the occurrence of enzymatic hydrolysis and the production of additional fermentable carbohydrates.

Statistical significance of treatment effects were assessed with Statistical Analysis Systems software (SAS, 2003) using the ANOVA procedure and the least significant difference (LSD) test. Differences were determined to be significant at $p \leqslant 0.05$.

3. Results and discussion

Acidification occurred with all treatments, with the largest pH declines occurring in the 0.45:1 and 2.54:1 enzyme treatments (Fig. 1). The 0:1 and 0.015:1 ratios produced values that were not different from the ensiled control. All treatments progressed below the pH 4.0 level by day 21, which is considered adequate for stable storage and to inhibit clostridium growth. Differences in pH between treatments can be attributed to the relative levels of organic acids produced. Ren et al. (2006) also observed a decrease in pH with enzyme-treated corn stover compared to untreated corn stover. Storage pH values of 3.9–4.5 and 4.8 were reported for enzyme-treated and untreated silages, respectively.

Water soluble carbohydrate levels ranged from 1.8% to 4.3% on a dry matter basis (d.b.) (Fig. 2). During the fermentation period, fermentable sugars were significantly

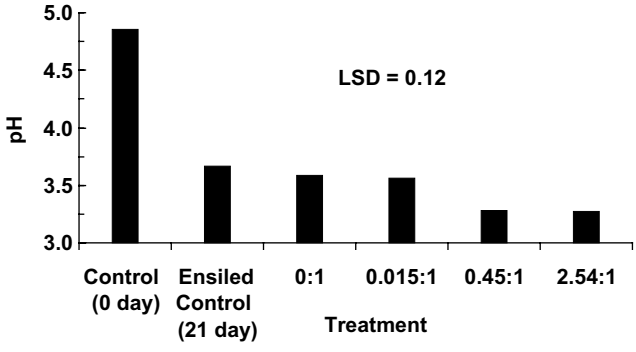

Fig. 1. Sample pH values for enzyme treatments ($n = 3$). $LSD_{0.05}$ is appropriate for comparison of treatments.

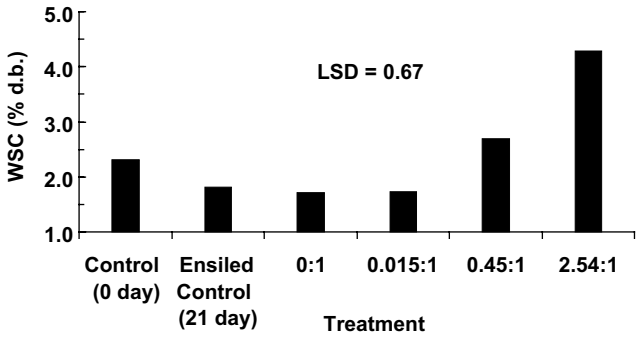

Fig. 2. Water soluble carbohydrate concentration values for enzyme treatments ($n = 3$). $LSD_{0.05}$ is appropriate for comparison of treatments.

higher with the 2.54:1 treatment compared to the other treatments. All other treatments had concentrations that were not found to be different from the day zero control; however the 0.45:1 treatment was higher than the day 21 control, 0:1, and 0.015:1 treatments.

A constant or increasing level of soluble carbohydrates over the fermentation period, as observed with the 2.54:1 and 0.45:1 treatments, respectively, is not necessary in a conventional storage ensilage system, but is important for a pretreatment ensilage system designed for treating biomass feedstocks. This indicates that structural carbohydrates are being hydrolyzed to soluble carbohydrates at a rate that is equal to or in excess of the rate of conversion for soluble carbohydrates to organic acids. In a pretreatment ensilage system, where the level of fermentable sugars can become limiting over time, organic acid production rates will become highly dependent on the conversion rate of hemicellulose and cellulose to soluble carbohydrates.

Organic acid profiles for all treatments were dominated by lactate and acetate (Table 2). Organic acid data is not presented for the day zero control because concentrations of fermentation products, including organic acids, are assumed to be negligible in plant materials that have not previously undergone fermentation. The 0.45:1 and 2.54:1 treatments produced higher lactate concentrations than all other treatments, at 2.5% and 3.0% (d.b.), respectively. The lactate concentration averaged about 1.5% (d.b.) for the other treatments. Acetate levels were not found to be different among any of the treatments and averaged approximately 1.1% (d.b.) for all treatments.

The 0.45:1 and 2.54:1 treatments were found to contain lower concentrations of butyrate. Butyrate levels for the day 21 control, 0:1, and 0.015:1 treatments were approximately 0.6% (d.b.). Propionate and iso-butyrate levels were similar for all treatments, with the only difference found between the 2.54:1 and ensiled control treatments. The higher levels of butyrate suggest the presence of clostridium bacteria and the occurrence of secondary fermentation in the control, 0:1, and 0.015:1 treatments. This agrees with the lower lactate concentrations noted with the same treatments. The presence of significant amounts of butyrate in most treatments, despite their low final pH values, suggests that clostridial fermentation may have been actively occurring in the early stage of ensilage. Clostridial fermentation likely continued until lactic acid fermentation dominated and a subsequent reduction in pH occurred. Inoculation of the initial material with lactic acid bacteria may be a likely solution to this problem (Pitt, 1990) and a potential topic for future research in this area.

Other researchers have also observed increases in lactic acid with application of enzymes on lignocellulosic substrates. Ren et al. (2006) treated fiberized corn stover with the same high cellulase enzyme (2.54:1) as was used in this experiment. Lactate concentrations of 3.3% and 0.9% (d.b.) were observed on enzyme-treated and non-treated samples, respectively, at 21 days of ensiling. Nadeau et al. (2000) reported lactate concentrations of 6.5% and 5.9% (d.b.) in orchardgrass and alfalfa silages, respectively, treated with a commercial cellulase enzyme. These values are higher than those observed in this study; however initial fermentable carbohydrates were also higher with the orchardgrass and alfalfa at 4.6% and 4.7% (d.b.), respectively, compared to 2.3% (d.b.) for the initial kenaf core fiber.

Hemicellulose and cellulose concentrations are displayed in Table 3. Differences in hemicellulose concentrations were only observed between the day zero control and the 2.54:1 treatment, while for the cellulose the only difference was between the day 21 control and the 2.54:1 treatment. In general, fiber fractions were similar among all enzyme treatments and controls, averaging 11% and 52% (d.b.) for the hemicellulose and cellulose, respectively.

The results indicate kenaf core fiber to be a suitable biomass feedstock for solid-state fermentation. All treatments

Table 2
Organic acid concentration values for enzyme treatments ($n = 3$)

Treatment	Lactate (% d.b.)	Acetate (% d.b.)	Propionate (% d.b.)	Isobutyrate (% d.b.)	Butyrate (% d.b.)
Ensiled control (21 day)	1.73	1.03	0.07	0.21	0.56
0:1	1.43	1.08	0.03	0.21	0.50
0.015:1	1.64	1.10	0.03	0.26	0.70
0.45:1	2.51	1.05	[a]	0.16	0.31
2.54:1	2.95	1.21	[a]	0.20	0.07
$LSD_{0.05}$	0.73	0.40	0.05	0.12	0.12

$LSD_{0.05}$ is appropriate for comparisons within a column.
[a] Values below detection level.

Table 3
Hemicellulose and cellulose concentration values for enzyme treatments ($n = 3$)

Treatment	Hemicellulose (% d.b.)	Cellulose (% d.b.)
Control (0 day)	11.9	52.6
Ensiled control (21 day)	11.0	53.1
0:1	11.5	52.6
0.015:1	11.5	52.5
0.45:1	10.7	49.9
2.54:1	10.0	49.4
$LSD_{0.05}$	1.98	3.58

$LSD_{0.05}$ is appropriate for comparisons within a column.

produced ensilage that was relatively stable, with only limited amounts of butyric fermentation occurring in the control, no-cellulase, and low-cellulase treatments.

Application of high cellulase activity enzyme mixtures greatly increased fermentation by hydrolyzing cellulose to fermentable carbohydrates. Overall, lower cellulase activity enzymes produced results similar to the control. Because all enzyme treatments contained the same level of hemicellulase activity, increases in water soluble carbohydrates and organic acids, particularly lactic acid, can be attributed to increased cellulase activity. Supplementing hemicellulase activity with high cellulase activity ratios is the preferred enzymatic pretreatment method for fermentation of kenaf core fiber compared to purified hemicellulase enzymes and no enzymes. A purified cellulase enzyme, containing no hemicellulase activity, is not commercially available, so comparisons to purified cellulase treatments were not possible in this study.

The cause for the ineffectiveness of the hemicellulases is unknown, but it is likely a function of the accessibility of the hemicellulose in the cell wall. Hemicellulose and lignin are closely associated in the kenaf cell wall and are located between the parallel chains of cellulose molecules (Stout, 1985). Because of the much higher cellulose content compared to hemicellulose content (approx. five times more) the surface area available for interaction of the enzymes with the hemicellulose may be considerably limited.

4. Conclusions

Results indicate solid-state fermentation, enhanced with enzymes, to be a suitable method for storage and pretreatment of kenaf core fiber. All treatments produced pH values below pH 4.5, which is sufficient for proper storage. Treatments with 2.54:1 and 0.45:1 cellulase:hemicellulase ratios produced the highest water soluble carbohydrate and lactic acid concentrations. Treatments with no or low cellulase activity produced results that were similar to the control treatment. Treatments with higher cellulase activity were able to generate more fermentable sugars and a higher level of conversion to lactic acid. The use of high cellulase activity enzyme mixtures is a suggested enzymatic pretreatment strategy for fermentation of kenaf core fiber.

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