# Microwave Pretreatments of Switchgrass Leaf and Stem Fractions to Increase Methane Production

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The objective of this study was to determine the effectiveness of microwave pretreatments on methane production from two switchgrass tissues (leaf *vs*. stem). The methane production from the leaf fraction was significantly affected by the microwave final temperature, while production from the stem fraction was affected by the combination of the microwave final temperature and heating rate. Thus, the highest methane yield from the leaf (134.81 mL $CH_4$/g of volatile solids (VS)) was obtained at 100 °C, while the highest yield from the stem (99.35 mL $CH_4$/g VS) was obtained at 150 °C, with a heating rate of 10 °C/min. Although methane production from the leaf fraction was merely enhanced by 9.1% after microwave pretreatments, the time required to reach 80% of ultimate methane production was reduced by 12 days. For the stem fraction, methane production was improved by 5.2% after microwave pretreatment, and the time to obtain 80% of ultimate methane production increased.

*Keywords: Switchgrass; Lignocellulose; Microwave pretreatment; Anaerobic digestion*

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## INTRODUCTION

Switchgrass (*Panicum virgatum* L.) is a perennial $C_4$ grass with great potential as a bioenergy resource (Xu *et al*. 2011; Zambare *et al*. 2011). Morphologically, switchgrass consists of roots, stems (including nodes and internodes), leaves (including leaf blades and leaf sheaths), and, at the reproductive growth stage, flowers, including the peduncle (Mann *et al*. 2009; Hu *et al*. 2011b). The chemical composition and structure vary among these different organs (Sarath *et al*. 2007; Hu *et al*. 2010). Therefore, the plant parts used as lignocellulosic feedstock greatly affect the characteristics of the pretreated biomass, and ultimately affect the quality and quantity of biofuel produced. Hu *et al*. (2011a) reported that the reduction in the degree of polymerization (DP) of cellulose and the extent of the increase in cellulose crystallinity differed between the leaves and internodes of Alamo switchgrass after hydrothermal pretreatment. As a result, the cellulose-to-glucose yield after the pretreatment was lower for nodes than for leaves of switchgrass. Similarly, the cellulose-to-glucose yield from the freeze-dried stems (including leaf sheaths) of switchgrass was 20% lower than the yield from leaves, after a dilute sulfuric acid pretreatment (Yang *et al*. 2009). For banana wastes (Kamdem *et al*. 2013) and wheat straw (Motte *et al*. 2014) treated with anaerobic digestion, the types of plant parts strongly affected the amount of methane produced. Thus, it is very important for the improvement of biofuel production that different pretreating strategies were required, depending on the structure characteristics of different morphological fractions of lignocellulosic biomass.

Microwave technology is a physical pretreatment in which materials are treated with electromagnetic waves with frequencies ranging from 300 MHz to 300 GHz (Huang *et al*. 2008). Microwaves can penetrate and couple energy directly to the materials. This results in rapid, uniform, and volumetric heating, which reduces processing times and saves energy (Thostenson and Chou 1999). In previous studies, when microwave technology was used to pretreat switchgrass, there was no change in the ultimate volume of methane produced, but the time required to obtain 80% of the maximum methane volume was reduced by 4.5 days after a 150 °C pretreatment (Jackowiak *et al*. 2011b). Similar trials with wheat straw showed that methane production was improved by 28% after a microwave pretreatment at 150 °C (Jackowiak *et al*. 2011a). However, when a microwave pretreatment was applied to *Pennisetum* biomass, the total methane yield was decreased by 12%, and the maximum production rate was decreased by 18% (Li *et al*. 2012). The adverse effect of microwave radiation on anaerobic digestion may be due to the pretreatment, which resulted in the release/formation of heat-induced inhibitors, such as phenolic compounds and furan derivatives (Jing and Lue 2007).

The major objective of this study was to investigate whether microwave pretreatments affected the leaf and stem fractions of switchgrass differently in terms of methane production *via* anaerobic digestion. In addition, biomass solubilization and fiber composition of the fractions were evaluated after the pretreatments.

## EXPERIMENTAL

### Materials

The Alamo variety of switchgrass was grown at the Shangzhuang Experiment Field, Beijing, China. The plants were harvested at the reproductive growth stage in September, 2013. The plant biomass was air-dried, the leaves and stems were manually separated, and finally each fraction was ground to pass through a 1-mm screen. The moisture contents of leaf and stem were 4.45% and 2.99%, respectively.

### Experimental Design

The experiment was conducted with an orthogonal design. To analyze the effects of microwave pretreatments, three factors were adjusted: the final temperature (factor A), holding time at target temperature (factor B), and heating rate (factor C).

**Table 1.** Assignment of Factors and Levels in Experimental Design using an Orthogonal Matrix $L_9(3^4)$

| Factor * | A (°C) | B (min) | C (°C/min) | D (vacancy) |
|---|---|---|---|---|
| Level I | 100 | 0 | 5 | - |
| Level II | 150 | 10 | 7.5 | - |
| Level III | 180 | 20 | 10 | - |

* Columns A, B, and C represent microwave final temperature, holding time at target temperature, and microwave heating rate, respectively. Column D stands for vacancy, to account for statistical error.

According to the $L_9(3^4)$ orthogonal array, each of the three microwave factors were assigned three levels (Table 1), and nine treatment combinations with different parameters were established (Table 2). The vacant column was used to account for the statistical error of the orthogonal method. Raw untreated material served as the control (treatment 10). All of the experiments were conducted with three replicates.

**Table 2.** The Orthogonal Matrix $L_9(3^4)$

| Treatments * | A (°C) | B (min) | C (°C/min) | D (vacancy) |
|---|---|---|---|---|
| 1 | 1 (100) | 1 (0) | 3 (10) | 2 |
| 2 | 2 (150) | 1 | 1 (5) | 1 |
| 3 | 3 (180) | 1 | 2 (7.5) | 3 |
| 4 | 1 | 2 (10) | 2 | 1 |
| 5 | 2 | 2 | 3 | 3 |
| 6 | 3 | 2 | 1 | 2 |
| 7 | 1 | 3 (20) | 1 | 3 |
| 8 | 2 | 3 | 2 | 2 |
| 9 | 3 | 3 | 3 | 1 |
| 10 | AT ** | 0 | 0 | - |
| 11 | AT | 10 | 0 | - |
| 12 | AT | 20 | 0 | - |

* Columns A, B, and C represent microwave final temperature, holding time at target temperature, and microwave heating rate, respectively. Column D stands for vacancy, to account for statistical error.
** AT: Ambient Temperature

## Microwave Pretreatments

A closed-vessel microwave accelerated reaction system (MARS 5, CEM Corporation, Matthews, NC, USA) was used to pretreat the samples. It was equipped with a turning carousel and a maximum of 40 vessels (XPRESS TFM 55 mL). The switchgrass samples consisted of 0.5 g leaf or stem material in 15 mL of distilled water for each vessel. The samples were microwave-treated according to the experimental design.

## Biochemical Methane Potential (BMP) Assays

The inoculum for anaerobic digestion of the biomass was composed of municipal sludge, with a pH value of 7.2. The total solids (TS) and volatile solids (VS) of the inoculum were 2.45% and 1.55% (w:v), respectively.

The experiments were conducted in 200 mL serum glass bottles at 37 °C. Each bottle contained 32 mL of inoculum, 2 g of pretreated switchgrass leaf or stem material, and distilled water, making a total volume of 122 mL. Urea was used to adjust the carbon-to-nitrogen ratio (C/N) to a level appropriate for the growth of anaerobic microorganisms (Zhang and Zhang 1999). The pH was adjusted to be between 7.0 and 7.5 before anaerobic digestion (Mendez *et al*. 2014). The digesters were flushed with $N_2$-gas to establish anaerobic conditions. Each batch reactor was mixed manually twice a day to degas the sample and prevent the formation of a dry layer (Kandel *et al*. 2013). Bottles containing the inoculum and distilled water served as blank controls. The BMP tests were carried out until the methane yield became negligible (<3 mL $CH_4$/day) (Lehtomaki *et al*. 2008).

## Analytical Methods

TS and VS were determined using the weight loss method (Pei *et al.* 2014). The hemicellulose, cellulose, and lignin contents were measured as described by Van Soest *et al*. (1991). Biogas production was monitored using the saturated brine displacement method. The gas component was analyzed using a gas chromatograph (GC-8600, Beijing Beifen Tianpu Instrument Tech. Co., Ltd., Beijing, China).

Biomass solubilization was determined by calculating the ratio of soluble volatile solids (sVS) to total volatile solids (sVS/VS). The increase in the sVS/VS ratio with respect to the control (no microwave treatment) was calculated according to Eq. 1, as described by Passos *et al*. (2013), shown below,

$$\text{sVS/VS increase (\%)} = \frac{(\text{sVS/VS})_p - (\text{sVS/VS})_o}{(\text{sVS/VS})_o} \times 100 \qquad (1)$$

where $(\text{sVS/VS})_o$ and $(\text{sVS/VS})_p$ represent the biomass solubilization before (*o*) and after pretreatment (*p*).

## First Order Kinetic Model

A first order kinetic model (Eq. 2) was used to correlate the methane production with the digestion time in the anaerobic digestion system, as follows,

$$B = B_0 \cdot (1-\exp(-kt)) \qquad (2)$$

where $B$ (mL/g VS) is the cumulative methane production as a function of time $t$, $B_0$ (mL/g VS) is the ultimate (or maximum) methane production, $k$ (day$^{-1}$) is the kinetic constant, and $t$ (day) is time (Hashimoto 1986; Lei *et al*. 2010).

# RESULTS AND DISCUSSION

## Solubilization of Switchgrass Leaf and Stem Fractions

The solubilization (sVS/VS) was higher for the leaf than for the stem after microwave pretreatments and under ambient temperature (AT) (Table 3). Microwave pretreatment enhanced the solubilization of the leaf and stem fractions. The sVS/VS increase ranged from 1.5% to 154.0% in the leaf fraction, and from 2.1% to 130.3% in the stem fraction (Table 3), depending on the microwave final temperature, holding time, and heating rate. Jackowiak *et al*. (2011b) also reported that microwave pretreatment improved the solubilization of switchgrass, but whole plants were used in that study. In our study, the solubilization of both the leaf and stem switchgrass fractions were significantly affected by the microwave final temperature ($P$<0.0001), holding time (leaf: $P$=0.0111; stem: $P$=0.0002), and heating rate (leaf: $P$=0.0010; stem: $P$=0.0002). However, the exposure time had a smaller influence on the solubilization (sCOD/tCOD) of whole fresh plants for switchgrass (Jackowiak *et al*. 2011b). Interestingly enough, a study on whole wheat straw showed that the solubilization (sCOD/tCOD) increased with longer holding times at the target temperature (Jackowiak *et al*. 2011a). This phenomenon may be related to the chemical composition of the biomass. Therefore, we evaluated the contents of the hemicellulose, cellulose, and lignin in the solid fraction of the leaf and stem materials after the microwave pretreatments.

**Table 3.** Matter Solubilization of Switchgrass Leaf and Stem Fractions

| Treatments | sVS/VS ratio * | | sVS/VS increase (%) | |
|---|---|---|---|---|
| | Leaf | Stem | Leaf | Stem |
| 1 | 0.160±0.008[de] | 0.122±0.001[de] | 17.7 | 10.6 |
| 2 | 0.174±0.003[c] | 0.131±0.002[cd] | 28.1 | 19.0 |
| 3 | 0.245±0.005[b] | 0.161±0.005[b] | 80.8 | 46.6 |
| 4 | 0.153±0.008[e] | 0.112±0.015[e] | 12.7 | 2.1 |
| 5 | 0.173±0.004[cd] | 0.135±0.002[c] | 27.2 | 22.9 |
| 6 | 0.245±0.012[b] | 0.172±0.004[b] | 80.4 | 56.8 |
| 7 | 0.138±0.012[f] | 0.126±0.003[cd] | 1.5 | 14.1 |
| 8 | 0.177±0.007[c] | 0.133±0.007[cd] | 30.3 | 20.7 |
| 9 | 0.345±0.003[a] | 0.253±0.011[a] | 154.0 | 130.3 |
| 10 | - | - | - | - |
| 11 | 0.136±0.009[f] | 0.110±0.006[e] | - | - |
| 12 | 0.135±0.008[f] | 0.110±0.003[e] | - | - |

* Means (±standard deviation) within columns that have the same superscript letters are not significantly different ($P$<0.05). Variance analyses of each experimental factor for sVS/VS ratio were as follows: microwave final temperature (leaf: $P$<0.0001; stem: $P$<0.0001), holding time (leaf: $P$=0.0111; stem: $P$=0.0002), and heating rate (leaf: $P$=0.0010; stem: $P$=0.0002)

The hemicellulose, cellulose, and lignin contents in untreated samples were, respectively, 29.5%, 31.09%, and 3.24% in the leaf fraction, and 29.58%, 34.67%, and 6.51% in the stem fraction. The hemicellulose content changed markedly as a result of the microwave pretreatments (Fig. 1).

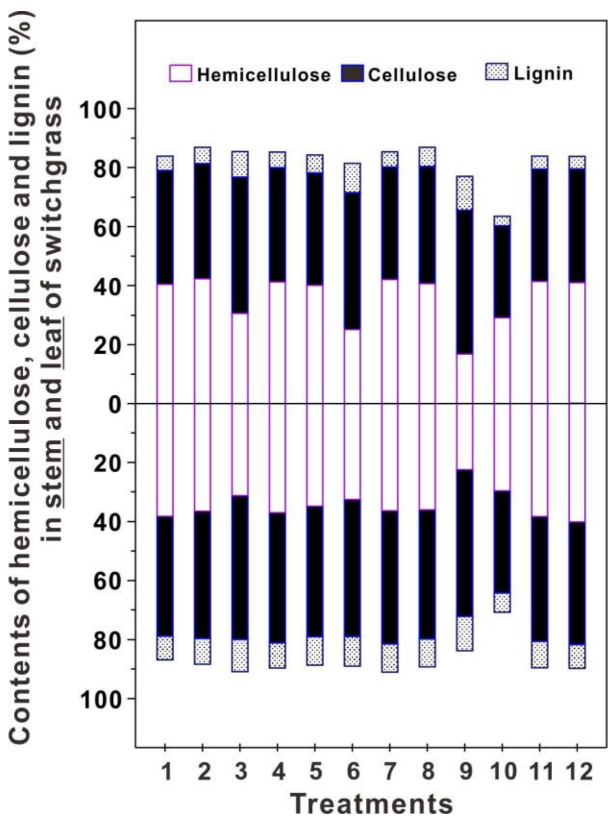


**Fig. 1.** Contents of hemicellulose, cellulose, and lignin in switchgrass leaf and stem fractions after microwave pretreatments.

The cellulose contents in the leaf and stem fractions of switchgrass were significantly affected by the microwave final temperature (Table 4 and 5); thus, the highest cellulose contents were found in both leaf and stem fractions treated at 180 °C (treatments 3, 6, and 9). Therefore, the advantage of the microwave pretreatments was that the material added to the reactor for digestion was more readily available (*i.e*., higher cellulose content) than the biomass pretreated at AT (Fernandez-Cegri *et al*. 2012).

The lignin content in the leaf fraction was affected by the combined effects of the microwave final temperature, holding time, and heating rate (Table 4), while that the lignin in the stem fraction was not significantly affected by the microwave heating rate (Table 5). During the microwave pretreatments, some organic matter was solubilized and lignin became more concentrated (Fernandez-Cegri *et al*. 2012). Therefore, the lignin contents in leaf and stem fractions were higher after severe pretreatments (treatments 3, 6, and 9) than in untreated materials or in materials immersed in distilled water at AT (Fig. 1).

**Table 4.** The Variance Analyses of Hemicellulose, Cellulose, and Lignin Contents of Switchgrass Leaf Fraction for Each Experimental Factor

| Factors* | Hemicellulose (%) | | Cellulose (%) | | Lignin (%) | |
|---|---|---|---|---|---|---|
| | F Value | Pr>F | F Value | Pr>F | F Value | Pr>F |
| A | 180.17 | <.0001 | 195.89 | <.0001 | 172.91 | <.0001 |
| B | 9.91 | 0.0010 | 3.20 | 0.0624 | 11.81 | 0.0004 |
| C | 13.48 | 0.0002 | 0.74 | 0.4884 | 3.78 | 0.0404 |

* Factors A, B, and C represent microwave final temperature, holding time at target temperature, and microwave heating rate, respectively.

**Table 5.** The Variance Analyses of Hemicellulose, Cellulose, and Lignin Contents of Switchgrass Stem Fraction for Each Experimental Factor

| Factors* | Hemicellulose (%) | | Cellulose (%) | | Lignin (%) | |
|---|---|---|---|---|---|---|
| | F Value | Pr>F | F Value | Pr>F | F Value | Pr>F |
| A | 42.99 | <.0001 | 30.25 | <.0001 | 28.97 | <.0001 |
| B | 8.37 | 0.0023 | 4.22 | 0.0295 | 5.96 | 0.0093 |
| C | 6.85 | 0.0054 | 0.62 | 0.5503 | 0.89 | 0.4245 |

* Factors A, B, and C represent microwave final temperature, holding time at target temperature, and microwave heating rate, respectively.

## Methane Production in BMP Assays

There was greater methane production from the leaf fraction than from the stem fraction under both untreated and pretreated conditions (Fig. 2 and 3). This was likely due to the higher content of soluble matter in the leaf in comparison to the stem. In BMP assays, the highest methane production was 134.81 mL/g VS in the leaf for treatment 4 (100 °C, 10 min, 7.5 °C/min), and 99.35 mL/g VS in the stem for treatment 5 (150 °C, 10 min, 10 °C/min). These values were lower than those obtained by Jackowiak *et al.* (2011b) for microwave pretreated whole fresh switchgrass plants (290 mL/g VS). Furthermore, the methane yield from the leaf fraction was affected only by the microwave final temperature (Fig. 4). The methane yield from the stem fraction was affected by the combined effects of the microwave final temperature and heating rate, as illustrated by the response surface plot (methane yield vs. temperature and heating rate; Fig. 5).

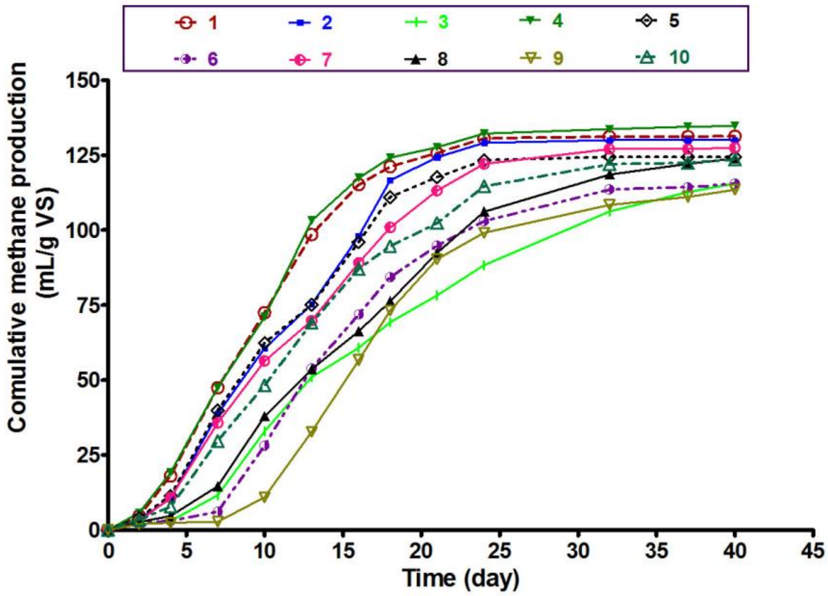


**Fig. 2.** Cumulative methane production from switchgrass leaf fraction under untreated and microwave pretreated conditions. Control, untreated sample. Variance analyses of each experimental factor for cumulative methane production were as follows: microwave final temperature (*P*<0.0001), holding time (*P*=0.3335), and heating rate (*P*=0.8819).

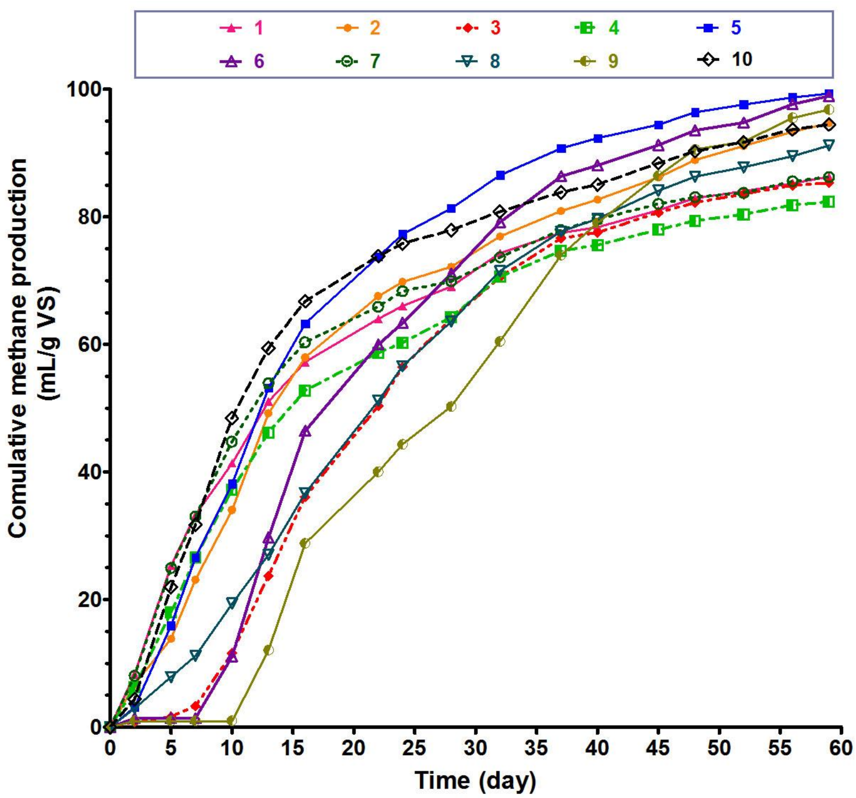


**Fig. 3.** Cumulative methane production from switchgrass stem fraction under untreated and microwave pretreated conditions. Control, untreated sample. Variance analyses of each experimental factor for cumulative methane production were as follows: microwave final temperature (*P*=0.0090), holding time (*P*=0.3408), and heating rate (*P*=0.0423).

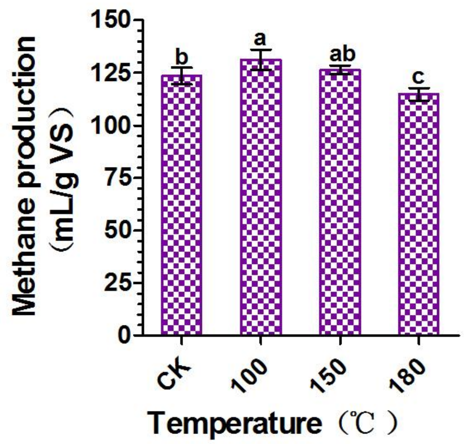


**Fig. 4.** Methane production from switchgrass leaf fraction after microwave pretreatments with various final temperatures. CK, untreated leaf sample. Different lower letters indicate significant difference at *P*<0.05.

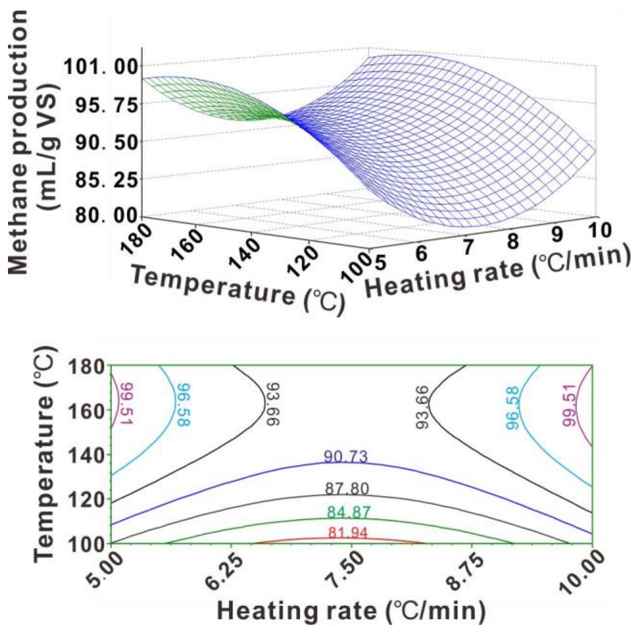


**Fig. 5.** Response surface plot showing the effect of microwave final temperature and heating rate on methane production from switchgrass stem fraction.

In other studies, microwave pretreatments resulted in the formation of phenolic acids, as well as furfural and 5-hydroxymethyl furfural (HMF). The formation of these compounds not only represented a loss of fermentable sugars, but also resulted in inhibitory or toxic effects on the anaerobic microorganisms (Chen *et al.* 2008; Hendriks and Zeeman 2009). Additionally, phenolic acids were released during the microwave pretreatments (0.32 mg per gram switchgrass at 90 °C and 0.64 mg per gram at 150 °C) in the study of Jackowiak *et al*. (2011b). Furfural and 5-hydroxymethyl furfural, which are the thermal decomposition products of xylose (Jing and Lue 2007), formed in the supernatant of the switchgrass mixture when the microwave temperature exceeded 130 °C (Jackowiak *et al*. 2011b). Maybe this was one of the reasons that the methane yield after the microwave pretreatment was only enhanced by 9.1% in the leaf and 5.2% in the stem. It needed to be proved by further experiments.

## First-order Kinetic Model

The high $R^2$ values indicated that the experimental data fit well with the proposed model at the 95% confidence level (Table 6 and 7). For the leaf fraction, compared with the kinetic constant (*k*) in the control (treatment 10), *k* values were 74.4% and 72.1% higher in treatments 1 (100 °C, 0 min, 10 °C/min) and 4 (100 °C, 10 min, 7.5 °C/min), respectively. For the stem fraction, only treatment 7 (100 °C, 20 min, 5 °C/min) resulted in an increased *k* value (4.5% increase). In a study on whole switchgrass, the *k* values were increased by 44% and 68% at 150 °C and 180 °C, respectively, compared with the control (Jackowiak *et al*. 2011b).

To assess whether microwave pretreatments accelerated the methane production process, the time (days) required to obtain 80% of ultimate methane production of untreated samples was evaluated. Compared with untreated leaf samples, those in treatments 1 (100 °C, 0 min, 10 °C/min) and 4 (100 °C, 10 min, 7.5 °C/min) reached the 80% point 11 and 12 days sooner, respectively (Table 6). For whole switchgrass, a reduction of 4.5 days was observed after a microwave pretreatment at 150 °C (Jackowiak *et al*. 2011b). For the stem fraction, however, none of the microwave pretreatments reduced the time required to reach 80% of ultimate methane production (Table 7).

**Table 6.** Ultimate Methane Yield ($B_0$) and Kinetic Constant (k) with 95% Confidence Limits for Pretreated Switchgrass Leaf Fraction

| Treatments | $B_0$ (mL/g VS) | k (day$^{-1}$) | $R^2$ | $t_{80\%}$ * (days) |
|---|---|---|---|---|
| 1 | 148±7 | 0.075±0.009 | 0.9186 | 26.5 |
| 2 | 157±10 | 0.056±0.007 | 0.9306 | 30 |
| 3 | 252±89 | 0.016±0.007 | 0.9002 | 44.5 |
| 4 | 151±6 | 0.074±0.008 | 0.9361 | 25.5 |
| 5 | 147±11 | 0.060±0.010 | 0.9247 | 34 |
| 6 | 197±39 | 0.026±0.007 | 0.9116 | 40.5 |
| 7 | 160±10 | 0.048±0.006 | 0.9519 | 33.5 |
| 8 | 235±55 | 0.021±0.007 | 0.9207 | 37.5 |
| 9 | - | - | - | - |
| 10 | 160±17 | 0.043±0.009 | 0.8921 | 37.5 |

* $t_{80\%}$ = Time to obtain 80% of ultimate methane yield from untreated leaf (128 mL/g VS)

**Table 7.** Ultimate Methane Yield ($B_0$) and Kinetic Constant (k) with 95% Confidence Limits for Pretreated Switchgrass Stem Fraction

| Treatments | $B_0$ (mL/g VS) | k (day$^{-1}$) | $R^2$ | $t_{80\%}$ * (days) |
|---|---|---|---|---|
| 1 | 86±1 | 0.065±0.003 | 0.9745 | 31.5 |
| 2 | 100±2 | 0.047±0.002 | 0.9843 | 29.5 |
| 3 | 129±16 | 0.021±0.004 | 0.9285 | 41.5 |
| 4 | 85±2 | 0.055±0.004 | 0.9474 | 39 |
| 5 | 106±3 | 0.050±0.003 | 0.9617 | 24.5 |
| 6 | 147±20 | 0.021±0.005 | 0.9141 | 34 |
| 7 | 86±1 | 0.070±0.002 | 0.9823 | 29.5 |
| 8 | 132±13 | 0.022±0.003 | 0.9445 | 38 |
| 9 | - | - | - | - |
| 10 | 94±3 | 0.067±0.006 | 0.9044 | 24 |

* $t_{80\%}$ = Time to obtain 80% of ultimate methane yield from untreated stem (75 mL/g VS)

## CONCLUSIONS

1. The microwave pretreatments enhanced the solubilization of switchgrass leaf and stem fractions to different degrees, because the increase in sVS/VS differed between the leaf and stem fractions.
2. After microwave pretreatment, the methane yield was enhanced by 9.1% in the leaf at 100 °C, and by 5.2% in the stem at 150 °C with the heating rate of 10 °C/min. The kinetics of methane production increased as a result of microwave pretreatments of the leaf fraction, and the time required to reach 80% of ultimate methane production was reduced by 12 days. The microwave pretreatments of the stem fraction increased the time required to reach 80% of ultimate methane production.

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