



Can hydrothermal pretreatment improve anaerobic digestion for biogas from lignocellulosic biomass?

Dou Wang^{a,b}, Fei Shen^{a,b,*}, Gang Yang^{a,b}, Yanzong Zhang^b, Shihuai Deng^{a,b}, Jing Zhang^{a,b}, Yongmei Zeng^{a,b}, Tao Luo^{c,d}, Zili Mei^{c,d}

^a Institute of Ecological and Environmental Sciences, Sichuan Agricultural University, Chengdu, Sichuan 611130, PR China

^b Rural Environment Protection Engineering & Technology Center of Sichuan Province, Sichuan Agricultural University, Chengdu, Sichuan 611130, PR China

^c Biogas Institute of Ministry of Agriculture, Chengdu, Sichuan 610041, PR China

^d Key Laboratory of Development and Application of Rural Renewable Energy, Ministry of Agriculture, Chengdu, Sichuan 610041, PR China

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ABSTRACT

Hydrothermally-pretreated rice straw (HPRS) from various pretreatment temperatures was anaerobically-digested in whole slurry. Results indicated promoting pretreatment temperature significantly deconstructed rice straw, and facilitated the conversion of insoluble fractions to soluble fractions. Although 306.6 mL/g TS biogas was maximally yielded in HPRS-90 and HPRS-180, respectively, via digestion in whole slurry, it was only 3% promotion compared to the untreated rice straw. HPRS-210 yielded 208.5 mL/g TS biogas, which was 30% reduction with longer lag period of 19.8 d, suggesting serious inhibitions happened. Through slightly increasing organic loading, more serious acidification and reduction on biogas yield, especially at higher pretreatment temperatures, indicated the soluble fractions controlled digestion performances. Pearson correlation analysis suggested negative relationship existed between methane yield and the soluble fractions including soluble carbohydrates, formic acid and furfural. Hydrothermal pretreatment, especially at higher temperature, did not improve anaerobic digestion, thereby, was not recommended, however, lower temperature can be considered potentially.

1. Introduction

Exhaustion of fossil fuels and the resultant massive greenhouse gas emissions have greatly motivated the rising interests in seeking alternative energy sources. Capturing energy from solar radiation, wind and water is currently facing some technical challenges, especially, the uncertain availability and insufficient storage options (Theuretzbacher et al., 2015b). In addition, energy security accelerates the technology development of energy refinery from cheap biomass, because biomass is available abundantly on the earth, as well as bioenergy utilization does not dramatically change the infrastructure and technologies of current energy consumption. By contrast, lignocellulosic feedstocks including agricultural residues, energy crops, woody wastes are some important candidates for refining various biofuels, such as bioethanol, biogas and bio-oil, to reduce the dependency on fossil fuels and improve energy security (Monlau et al., 2014; Monlau et al., 2015; Zhang et al., 2016).

Among various biofuels, the biogas production from lignocellulosic biomass via anaerobic digestion is widely accepted as one of the highly promising alternatives to fossil-derived energy due to several inherent

and significant merits (Monlau et al., 2015; Sawatdeenarunat et al., 2015). It is a simple and efficient way to dispose various feedstocks, containing the readily biodegradable organic matters of carbohydrates, proteins and fats, by converting them into simple derivatives and finally into CH₄ and CO₂ with the cooperation of prokaryotic microorganisms (Mao et al., 2015). No additional step for the product separation is needed during the methane production process because of its easy releasing from the digestate (Hendriks and Zeeman, 2009). However, major challenges for obtaining biogas from lignocellulosic feedstocks are the inherent recalcitrant structure and the complex chemical composition, conferring the resistances to hydrolysis and further conversion by anaerobic microorganisms (Li et al., 2014; Monlau et al., 2014). Therefore, efficiently breaking lignocellulosic recalcitrance is definitely considered as a dominating step in the whole streams of anaerobic digestion (Sambusiti et al., 2013; Sawatdeenarunat et al., 2015). Besides, the heterogeneity and low density of lignocellulosic biomass impede its deconstruction by microorganisms and form a floating layer on the surface of digestate (Tian et al., 2015). These will result in the poor accessibility between substrate and microorganisms, worsen heat and

* Corresponding author at: 211 Huimin Road, Wenjiang District, Chengdu, Sichuan 611130, PR China.
E-mail addresses: fishensjtu@gmail.com, fishen@sicau.edu.cn (F. Shen).

mass transfer, and lower biogas yield consequently. Therefore, a proper pretreatment step to disintegrate the natural physicochemical barriers is generally required to promote homogeneity and make lignocellulosic biomass efficiently available for subsequent biological conversion (Sawatdeenarunat et al., 2015).

Currently, various pretreatment technologies, including mechanical, thermal, chemical, and biological method or their potential combination, have been attempted on lignocellulosic feedstocks prior to anaerobic digestion (Sawatdeenarunat et al., 2015). Consequently, the anaerobic digestion performances can be improved more or less, however, the inherent disadvantages, such as high cost on the consumed energy or chemicals, potential pollutions, potential inhibition to microorganisms, lower efficiency, and substrate loss, could not be avoided to some degree (Hendriks and Zeeman, 2009). Hydrothermal pretreatment involves in lignocellulosic feedstock and water only, and has been widely accepted as a green technology without potential chemical consumption and potential pollution (Saha et al., 2013). Typically, it can remove most of hemicellulose and part of lignin in biomass via degrading them into soluble fractions, and loosen the recalcitrant structure as well. Therefore, hydrothermal pretreatment has been widely applied for facilitating biofuels production, especially bioethanol, from lignocellulosic feedstocks (Cybulska et al., 2013).

In this context, hydrothermal method was directly transplanted from pretreating lignocellulosic feedstocks for producing bioethanol to biogas, because considerable researches generally believed the achieved improvement on digestion performances was mainly attributed to the structure-breaking and the promoted accessibility of solid substrate after pretreatment (Chandra et al., 2012; Saha et al., 2013). However, most of these investigations were subjected to solid-liquid separation, and only the obtained insoluble fraction was generally utilized for digestion (Hesami et al., 2015; Zhou et al., 2017). Definitely, this will be bound to reduce the biogas yield once the derived soluble fraction from hydrothermal pretreatment could not be digested together, because acetic acid, furfural, 5-hydroxymethyl furfural (5-HMF), monomeric and oligomeric sugars, and the lignin-derivatives are in the soluble fraction abundantly (Sambusiti et al., 2013). Recently, the whole slurry, including the soluble and insoluble fractions after pretreatment, has been investigated for anaerobic digestion, exhibiting obvious biogas reduction at higher pretreatment temperature, although biogas releasing can be promoted at the other temperatures (Wang et al., 2017a,b). In addition, it was reported that the hydrogen yield displayed strongly correlation with the pretreatment temperature as the hydrothermally-pretreated rice straw was anaerobically digested in whole slurry, indicating the substrate had characteristic of easy acidification, which will be not beneficial to biogas generation in potential (He et al., 2014). Besides, the derivatives, such as furfural, HMF, and phenolic compounds in the soluble fraction may potentially induce inhibitions as the anaerobic digestion was performed in whole slurry (Monlau et al., 2014). Obviously, the anaerobic digestion performances of the whole slurry from hydrothermal pretreatment are controlled by the combined actions of water-insoluble fraction (WIF) and water-soluble fraction (WSF), however, their actual effects are still not explicit. Thus, whether hydrothermal pretreatment can improve biogas production from lignocellulosic biomass deserves to be investigated in-depth so that the technology can be understood better and applied properly.

In order to clarify this issue, rice straw was hydrothermally pretreated at broad temperature ranges to achieve the slurry with different degrees of structure-broken WIF and concentrations of WSF. The resultant slurries were anaerobically digested at a low organic loading to clarify the actual performances as affected by pretreatment temperature. In addition, it can be hypothesized that the effects of WIF deconstruction on digestion performances would be very weak as organic loading was promoted slightly, while the effects from WSF could be reflected. Thus, slightly increasing organic loading was designed to analyze and elucidate the roles of the deconstruction and the formed

Table 1
Basic characteristics of the employed rice straw and inoculum.

Parameter	Rice straw	Inoculum
Total solids (TS) (%)	89.9 ± 0.1 ^a	3.7 ± 0.0
Volatile solids (VS) (% TS)	85.0 ± 0.1	52.2 ± 0.2
Total carbon (% TS)	39.3 ± 0.3	18.9 ± 0.3
Total nitrogen (% TS)	0.4 ± 0.0	2.9 ± 0.1
Cellulose (% TS)	29.1 ± 0.8	9.3 ± 0.2
Hemicellulose (% TS)	16.3 ± 1.0	4.2 ± 0.1
Lignin (% TS)	24.2 ± 0.2	19.4 ± 0.3
pH	N.A.	7.4

^a Data in the table were exhibited in the form of “Mean ± Standard deviation”. N.A. refers to not applicable.

soluble fraction by hydrothermal pretreatment in anaerobic digestion.

2. Materials and methods

2.1. Feedstock and inoculum

The air-dried rice straw was harvested from the farm of Sichuan Agricultural University, Chongzhou, China. Afterwards, rice straw was chopped through 2-mesh sieve by a straw chopper and stored in an airtight plastic bag before pretreatment. Inoculum for anaerobic digestion was obtained from the Biogas Institute of Ministry of Agriculture, Chengdu, China. The inoculum was pre-incubated for 15 d at mesophilic temperature to deplete the residual biodegradable organic matters and activate the microorganisms. The basic characteristics of rice straw and inoculum were listed in Table 1.

2.2. Hydrothermal pretreatment

The chopped rice straw with solid loading of 10% (w/w) was immersed in the distilled water for 12 h. Afterwards, the rice straw with the distilled water was input in a 1.5 L reactor (GCF-1.5, Dalian Auto-control Equipment Co., China) for pretreatment. The reactor was heated to the designed temperatures (90 °C, 150 °C, 180 °C and 210 °C, respectively) with heating rate of 10 °C/min, and the designed temperature was maintained for 15 min. Meanwhile, a top-mounted motor was employed for stirring with 150 rev/min during pretreatment. The reactor was rapidly cooled down by an inside stainless-steel coil with tap water as the pretreatment was completed. The hydrothermally-pretreated rice straw (HPRS) was in slurry state, and was taken out as completely as possible. Distilled water was used for washing the reactor, and the washing water was merged into the slurry in total. According to the total weight of the input rice straw and the distilled water for pretreatment, the evaporated water was compensated to slurry with the distilled water. The pretreated rice straw at 90 °C, 150 °C, 180 °C, and 210 °C was defined as HPRS-90, HPRS-150, HPRS-180, and HPRS-210, respectively. All resultant slurries were stored at 4 °C in a refrigerator till for subsequent analysis and anaerobic digestion.

2.3. Anaerobic digestion in batch

In order to guarantee the homogeneity of each replicate for HPRS digestion, the slurry was separated into solid fraction and liquid fraction via a 40-mesh sieve. The solid fraction was mixed thoroughly in a sealed plastic bag, and liquid was also mixed thoroughly by stirring. Afterwards, the liquid and solid were weighted as 2 equivalent parts, and the weighted liquid and solid were mixed together to achieve 2 replicates for digestion. Anaerobic digestion in batch was carried out in 1.0 L flasks with the working volume of approximately 800 mL. Organic loading of 35 g TS/L was applied for anaerobic digestion in whole slurry to investigate actual performances as affected by pretreatment

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