Modelling, Measurement and Control C
Vol. 79, No. 3, September, 2018, pp. 127-132
Journal homepage: http://iieta.org/Journals/MMC/MMC_C

Enhancing anaerobic digestion of wheat straw through multistage milling

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https://doi.org/10.18280/mmc_c.790310

Received: 2 May 2018
Accepted: 8 June 2018

Keywords:
biogas, biomethane, pretreatment, straw

ABSTRACT

The effects of a mechanical pretreatment, carried out by a two stages mill followed by fractionation into two different products, were determined on the biodegradability and methane yield of wheat straw. Straw was chopped in a knife mill to an average length of about 30 mm, and a portion was used as reference material. Pretreatment reduced the size of the processed straw, whose median particle size was 300μm and 1200μm for the fine and the coarse fractions, respectively. Untreated and pretreated materials were anaerobically digested in batch reactors under mesophilic conditions for 28 days. The methane yield of raw material was 167.8 $Nm^3\ t_{VS}^{-1}$, whereas the pretreated material reached an average methane yield of 250.6 $Nm^3\ t_{VS}^{-1}$, showing a 49.3 % gain on VS basis compared to the feedstock. The finest fraction of the processed material reached a methane production of 264.7 $Nm^3\ t_{VS}^{-1}$. The specific electric energy consumption was 66.6 kWh per ton of processed material. The energy efficiency was high, since consumption was only 23.7 % of the gross electric energy gain available after pretreatment. These results proved that the investigated pretreatment could be successfully implemented to improve methane production efficiency in anaerobic digesters.

1. INTRODUCTION

Within the European Union, 14,572 biogas plants reached an installed electrical capacity of 7.85 GW in 2013, and 47.5 TWh of electricity were delivered to the network. A 62 % of these installations (9,035) was concentrated in Germany, followed by Italy with 1,391 plants, 80 % of which (1,121) were fed with substrates from agriculture. The number of biomethane plants on the same year was far short, 282 units with a strong domination of Germany (154 plants). Biomethane industry in Italy was still at a very early stage (2 plants) [1].

In October 2014, the Italian Ministry of Economic Development introduced the obligation of placing primarily on the Italian fuel market the so-called "advanced biofuels", that are those, including biomethane, being produced exclusively from specific feedstock, such as straw, corn stover, forest residues, and dedicated lignocellulosic crops. The mandatory share of advanced biofuels in the transport sector is set at 1.7 % of the energy demand for the year 2018, to grow up to 2 % in 2022 [2]. Using biomethane significantly reduces pollutant emissions compared to gasoline and diesel powered engines, and is also well below the levels of biodiesel [3].

The residues of the agricultural activities in the European Union exceed 200 million tons every year and they are mostly constituted by cereal straw, which represents the most abundant biomass available in the Union for bioenergy production [4]. It is mostly left for mulching or burned on the field, and it is thus available in considerable quantity and at low-cost.

Despite its abundance and its strongly encouraged use, straw, and more generally lignocellulosic residues, is of very limited use for methane production because of its low biodegradability. In the anaerobic digestion of lignocellulosic materials, the difficulty of hydrolysis is the limiting factor of the process. In this first step, different bacterial strains operate the degradation of complex organic substrates - proteins, fats and carbohydrates - producing simpler compounds such as amino acids, fatty acids and soluble monosaccharides, which are thus made available for transport into the cells of acidogenic microorganisms. Lignin, resistant to anaerobic degradation, acts as a binder in the lignocellulosic matter, and, therefore, constitutes a physical barrier to the enzymatic action on the hydrolysable carbohydrates. To overcome this obstacle, physical, chemical and biological pretreatments, and their combinations, have been developed to disrupt the lignocellulosic structure to favour cellulose and hemicellulose accessibility to enzymes.

Chemical or physic-chemical pretreatments are often not economically attractive due to the high cost of chemicals and the need for their recovery and/or treatment of the liquid effluent [5]. They also have several drawbacks with regard to anaerobic digestion processes. The diluted sulfuric acid pretreatment, probably the most common process, results in the production of hydrogen sulphide (H_2S) which seriously deteriorates the quality of gas. Moreover, inhibitors are formed during pretreatment of lignocellulosic biomass at high temperatures and acidic conditions, i.e. furfural, 5-hydroxymethylfurfural and phenolic compounds that usually inhibit the microbial growth and fermentation, resulting in low biogas production [6, 7]. Maleic acid pretreatment, which showed promising results for bio-ethanol, resulted in a strong reduction in methane production from straw and hay [8].

Alkali pretreatment is more compatible with subsequent anaerobic digestion than acidic treatment, since any alkali remaining with the treated solids is useful in the anaerobic digestion, which requires alkalinity addiction for pH control. A mild alkaline pretreatment of wheat straw with NaOH – 4 % and very long residence time (120 h) at 37 °C - produced 111.6% more methane than the untreated substrate, reaching a yield of 165.9 Nm^3 t_{VS}^{-1} over 35 days of digestion [9].

Another mild alkaline pretreatment of wheat straw, carried out at 40°C for 24 h with the addition of 10 % NaOH, increased the methane yield by 43 %, reaching a production of 293 Nm^3 t_{VS}^{-1} after 31 days of digestion. The same NaOH concentration and 30 minutes residence time at 100°C, produced a methane yield of 341 Nm^3 t_{VS}^{-1}, corresponding to a 67 % gain as compared to the untreated substrate [10].

Wheat straw biodegradability during anaerobic digestion was also improved by treatment with potassium hydroxide (KOH). A biomethane yield of 258 Nm^3 t_{VS}^{-1} was obtained over 40 days of digestion, resulting in a yield gain of 41% as compared to untreated straw; pretreatment required 6% KOH (w/w_{TS}) and residence time of 72 h at room temperature [11].

Among physic-chemical processes, wet explosion of wheat straw (heating at 180°C and 10 bar pressure with H_2O_2 as oxidizing agent) resulted in a slight reduction of methane production [12], and this was most probably due to the formation of inhibitory compounds [13].

In the field of physical pretreatments, steam explosion has received substantial attention for both ethanol and biogas production. Steam exploded wheat straw reached a methane production of 273 Nm^3 t_{VS}^{-1} after treatment at 220 °C, 23 bar and 1 minute residence time, resulting in a 20 % increase in methane production compared to untreated straw. For more severe conditions, the biodegradability decreased due to a possible formation of inhibitory compounds [14]. In another steam explosion experiment, the biological methane potential of the non steam exploded, ground wheat straw did not significantly differ from the best steam explosion treated sample (286 Nm^3 t_{VS}^{-1}), which was achieved at a pretreatment temperature of 140 °C and a retention time of 60 min [15].

Thermal-expansionary pretreatment combines the boiling of an aqueous biomass suspension under high pressure followed by rapid decompression. Following 20 minutes residence time at 170 °C, a methane yield of 360 Nm^3 t_{VS}^{-1} was achieved after 60 days of digestion, gaining 41 % respect the untreated raw material [16].

Microwave heating was also used to improve the degradability of cereal straw. Despite the strong increase in methane yield of the substrates, the process resulted not sustainable, because of the high demand of electric energy [17].

The cellulolytic anaerobic bacteria Clostridium cellulolyticum were adopted to improve the biochemical methane potential of wheat straw, which reached 342.5 and 326.3 Nm^3 t_{VS}^{-1} after 36 days of digestion and 24 and 60 hours of incubation respectively, with an increase of 13.0 % and 7.6 % compared to the raw material [18].

Biomass size reduction, or comminution, is a mechanical pretreatment whose objective is to increase the specific surface area available, thus facilitating the exchange of mass and heat and the action of anaerobic microorganisms. The comminution also causes a reduction of the crystallinity of the cellulose and its degree of polymerization. In order to achieve a greater hydrolysis capacity of the biomass and reduced times of digestion, it is necessary to produce fragments not larger than 1-2 mm [19]. The great advantage of this pretreatment is the absence of any effluent, while the high demand of electric energy is the main drawback.

Koullas [20] reported that the efficiency of conversion of carbohydrates increased from 17 % to 68 % after two hours of grinding of wheat straw in a ball mill. However, such a device is unsuitable for commercial installations, because of long processing times and the excessive energy consumption: up to 30,000 kWh t^{-1} (108 MJ kg^{-1}) [21].

Sharma et al. reported the effects of particle size of agricultural and forest residues on biogas generation through anaerobic digestion under mesophilic conditions [22]. Out of five particle sizes of wheat straw (0.088, 0.40, 1.0, 6.0, and 30.0 mm), the maximum quantity of methane, 250 Nm^3 t_{VS}^{-1}, was produced with both the smallest particles, 0.088 and 0.4 mm. Moreover, biomethane yield stepped from 160 to 230 Nm^3 t_{VS}^{-1} as particle size decreased from 30 to 6 mm. No data are available concerning the energy efficiency of the process.

A recent work [23] studied the effects of successive grinding steps on anaerobic digestion of wheat straw, concluding that micronization did not improve methane yield, which ranged from 281 to 306 Nm^3 t_{VS}^{-1}, but had a positive effect on the biodegradation kinetics. In addition, no significant increase of kinetics was observed below a size threshold value around 200 µm. Milling was carried out at a laboratory scale and the evaluation of the energy demand was not performed.

Mechanical treatment of wheat straw improved methane yields from 285 to 334 Nm^3 t_{VS}^{-1} as particle size was reduced from 50 mm to 2 mm, after 65 days of digestion at 40°C [24]. The authors did not perform any direct measure of the electric energy demand, and the size of the equipment and the mass flow rate during the experiments were not declared.

These data are necessary for an accurate evaluation of the energy balance, since they have a strong influence on the energy consumption. Several researchers measured an energy demand of about 43 kWh t^{-1} (0,15 MJ kg^{-1}) of processed straw for hammer mills of very low power (1.5 kW) equipped with a screen size of 1.6 mm [25, 26]. Whereas, using a device of greater power (18 kW), such energy demand was already reached for a screen size of 3.2 mm [27]. For mills of even greater size, suitable to obtain straw fragments of 1-2 mm in medium or large scale digestion plants, energy consumption was in the order of 90-130 kWh t^{-1} (0.32-0.47 MJ kg^{-1}) [18].

It was not possible to found in the literature a detailed analysis of cost-benefit concerning mechanical pretreatments of wheat straw for anaerobic digestion.

The objective of the present study was to evaluate the effectiveness of an industrial scale, double stage mill used to pretreat wheat straw. Through a set of anaerobic digestion experiments, it was defined the methane yield of both untreated and pretreated materials, including the biogas composition. A detailed analysis of chemical composition and particle size on both raw and processed materials was performed, in order to help in interpreting the results. Lastly, an evaluation of the energy balance of the pretreatment was carried out.

2. MATERIALS AND METHODS

2.1 Pretreatment

The milling device has been designed to process feedstock with a high dry matter content (>70%). It comprises two milling stages: the first acts predominantly by impact, maximizing the number of shocks to obtain a significant breakage due to fatigue, whereas the second exerts strong shear actions on the processed material [28]. The device was equipped with a 75 kW electric engine. A centrifugal classifier, placed downstream the mill, divided the material into two different streams. A centrifugal fan equipped with an 18 kW electric motor produced an air flow of about 1000 $m^3 h^{-1}$, used to convey the processed material through the device.

Naturally dried straw from a soft wheat grown in central Italy was chopped in a knife mill to an average length of about 30 mm, which is a typical dimension used in anaerobic digestion plants. A portion of this material was used as a reference (Wheat straw, WS).

Fifty kilograms of the chopped material were ground in the above described device, producing two different streams of material, named WSM and WSF. A screw conveyer fed the mill at a constant mass flow rate of 750 kg h^{-1}. The power drawn by the device was measured using a wattmeter (MTME-485, ABB-SACE, Italy); power, supply voltage, current and time were logged into a PC card at one-second intervals. The specific energy (MJ kg^{-1}) required for milling was determined by integrating the area under the power demand curve for the total time required to grind the 50 kg sample. In order to assess the energy demand, the test was performed in two replicates.

2.2 Chemical composition analysis

The two products and the raw material were analyzed for total and volatile solids (TS and VS, respectively) according to the APHA standard methods [29]. The content of NDF, ADF and ADL was analyzed according to the procedure of Van Soest [30]. Total nitrogen (TKN) was analyzed by the Kjeldal method and lipids (EE) by extraction with diethyl ether [31].

2.3 Particle size analysis

The particle size distribution of WSM and WSF was analyzed according to the ASABE standard S319.3 [32]. Through this test, mass percentages are measured as a function of their particle size by passing through sieves of specified mesh sizes. A sieve analyzer used twelve ISO sieves (3.000, 2.000, 1.400, 1.180, 1.000, 0.700, 0.600, 0.500, 0.425, 0.300, 0.212 and 0.150). The total samples mass for the particle size analysis was about 250 g, and each time the sieve was operated for 15 minutes.

2.3 Anaerobic digestion

Biogas production experiments were carried out on the raw material (WS) and the two products (WSM and WSF), to assess the biogas and methane yields. The experiments were performed in batch anaerobic reactors with a working volume of 2 litres and equipped with mixing and thermostating systems; the reactors were operated in mesophilic conditions (35°C). Anaerobic sludge from a mesophilic digester, containing 9,17% total solids (TS) and 5,48% volatile solids (VS), was used as inoculum to start the biological process. The loading rate of 60 g/L was applied for both raw material and processed straw, resulting in a substrate loading in the range 53-54 g_{TS}/L (47,7-50 g_{VS}/L); the substrate/inoculum ratio was in the range 0,58-0,59 on a TS basis. The experiments lasted 28 days and each of them was performed in triplicate, including two controls with inoculum sludge only; the gas produced by controls was subtracted from the actual gas produced through digestion of the media.

Biogas production was measured daily in averaged samples following standard methods [29]. The composition of the biogas with reference to methane content was measured using a SG06IOMX6 portable automatic analyser (B.A.G.G.I. srl, Milan, Italy).

3. RESULTS

3.1 Chemical composition analysis

WSM and WSF accounted for 73.0 % and 26.3 % of the raw material, respectively. The processing lost was 0.7 %, due to the reduction of the moisture content in the products respect the raw material. Grinding reduced the size of the processed straw that reached 300 μm as median particle size (d_{50}) for the finest fraction (WSF), whereas it was of about 1200 μm for WSM.

The results of the chemical analysis are shown in Table 1. WSF showed a significant decrease in the concentration of NDF, ADF and ADL with respect to raw material. In particular, NDF decreased from 82 % to 72.9 % on dry weight basis. The ash content increased from 8.7 % to 11.5 % and the nitrogen content enhanced by 35 % respect to the untreated straw, as shown by the CP values. NDF, ADF and ADL in WSM increased significantly respect to the raw material, whereas the ash content decreased.

Table 1. Main chemical characteristics of untreated and pretreated straw

	TS	VS	CP	EE	NDF	ADF	ADL
	[%]				[$\%_{TS}$]		
WS	92.1*a*	83.7*a*	3.4*a*	1.2*a*	82.0*a*	55.9*a*	7.6*a*
WSM	92.9*b*	86.3*b*	3.0*b*	0.7*b*	84.4*b*	58.1*a*	8.1*b*
WSF	92.3*a*	80.8*c*	4.6*c*	1.8*c*	72.9*c*	50.3*b*	6.4*c*

Notes: 1. Values followed by the same letters in the same column are not statistically different with a p-value <0.05 (Tukey test).

3.2 Biogas yield and quality

The untreated straw reached a methane yield of 167.8 Nm^3 t_{VS}^{-1}, whereas for WSM and WSF the production was 245.6 and 264.7 Nm^3 t_{VS}^{-1}, respectively (Table 2). On average, the methane yield gain was 49.2 % on VS basis and 50.1 % on wet basis. Methane concentration in biogas was significant higher in WSF (52.3 %) respect the raw material (49.3 %).

As expected, the methane production was quite faster in the processed material than in the unprocessed straw. As depicted in Figure 1, untreated straw reached 50 % of the cumulated production during the 11th day from the beginning of the assay, whereas it took about 7 days for WSM and WSF. The methane production rates for WSM and WSF were high, reaching their respective maximum of about 25.2 and 33.5 Nm^3 $t_{VS}^{-1}day^{-1}$ during the 3rd and 2nd day of digestion. The maximum production rate for the untreated material was only 10.2 Nm^3 $t_{VS}^{-1}day^{-1}$ and was reached at the beginning of the 4th day.

3.3 Specific energy requirement

The average power drawn by the pretreatment device, including the need for the feeding conveyer and the pneumatic transport of the processed material, was 49.9 kW, resulting in a specific energy requirement of 66.6 ±3.0 kWh t^{-1} (239.7 ±10.8 kJ kg^{-1}).

Table 2. Methane yield and biogas composition

	CH_4 yield		CH_4
	[Nm^3 t_{VS}^{-1}]	[Nm^3 t^{-1}]	[%vol]
WS	167.8 *a*	140.4 *a*	49.3 *a*
WSM	245.6 *b*	211.9 *b*	51.1 *a b*
WSF	264.7 *c*	213.8 *b*	52.1 *b*

Notes: 1. Values followed by the same letters in the same column are not statistically different with a p-value <0.05 (Tukey test).

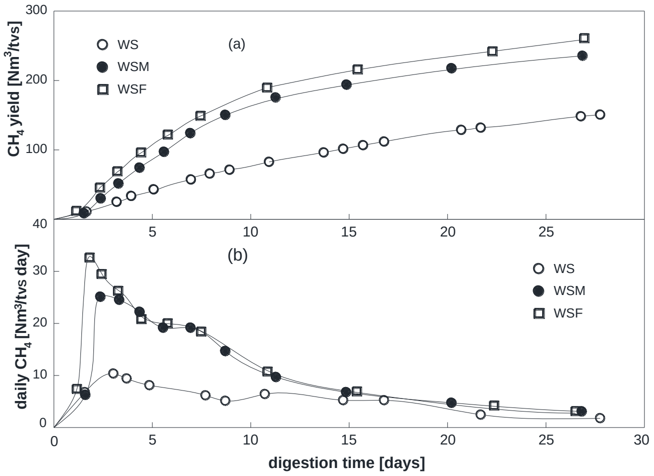

Figure 1. Cumulative (a) and daily (b) methane yield

3.4 Energy data analysis

Electrical energy (E_e) produced by untreated and pretreated biomasses, expressed as kWh t^{-1} of raw material (WS), was calculated using Eq (1):

$$E_e = Y \cdot LHV \cdot \eta_e \cdot (m_P / m_U) \quad (1)$$

where Y was the methane yield of the samples, expressed in Nm^3 t^{-1}, LHV was assumed to be 9.94 kWh Nm^{-3} (35.7 MJ Nm^{-3}), η_e was the co-generator electrical efficiency, estimated as 40 %, m_U and m_P were the masses of the processed sample before and after pretreatment, respectively.

The electric energy gain was obtained by subtracting from the energy produced by the pretreated material the sum of the energy obtained from the raw material and the energy consumption of the process. The results are shown in Table 3.

As regards biofuels production, primary energy (E_P) produced by the samples, expressed as kWh t^{-1} of raw material, was calculated using Eq (2):

$$E_P = Y \cdot LHV \cdot (m_P / m_U) \quad (2)$$

whereas the primary energy consumed to pretreat the raw material was calculated using Eq (3):

$$EP = EC \cdot f \quad (3)$$

where E_C was the electric energy used for pretreatment, expressed in kWh t^{-1} and *f* was the coefficient which enables to transform electric energy into primary energy, set equal to 1.86 kWh kWh^{-1} for the Italian electric grid [33]. The primary energy gain was obtained by subtracting from the energy produced by the pretreated material the sum of the primary energy obtained from the raw material and the primary energy consumed for pretreatment. The results are shown in Table 4.

Table 3. Energy performance for CHP applications

		ELECTRIC ENERGY			
	CH_4	output	consum	net gain	
	[Nm^3 t^{-1}]	[kWh t^{-1}]	[kWh t^{-1}]	[kWh t^{-1}]	[%]
WS	140.4	558.4			
WSM	212.0	615.2	66.6	213.8	38.3
WSF	213.9	223.6			

Specific energies refer to the mass of the material before processing (WS).

Table 4. Energy performance for biofuel applications

		PRIMARY ENERGY			
	CH_4	output	consum	net gain	
	[Nm^3 t^{-1}]	[kWh t^{-1}]	[kWh t^{-1}]	[kWh t^{-1}]	[%]
WS	140.4	1396.1			
WSM	212.0	1538.0	123.9	577.2	41.3
WSF	213.9	559.1			

Specific energies refer to the mass of the material before processing.

4. DISCUSSION

As discussed in the introduction, data concerning the energy efficiency of mechanical, industrial scale devices used to pretreat lignocellulosic feedstocks are scarce.

The investigated pretreatment enhanced by 50.1 % the methane production by anaerobic digestion of wheat straw, from 140.4 to an average of 210.9 Nm^3 per ton of processed raw material. The energy consumption (66.6 kWh t^{-1}) was low compared to the data mentioned in the introduction, given the final dimension of the processed material and the industrial size of the investigated mill.

With reference to the use of biogas in CHP plants, the electric energy production from pretreated straw was 838.8 kWh t^{-1} of processed raw material, achieving a 38.2 % net gain over the untreated material, whose energy yield was 558.4 kWh t^{-1}. Electric energy consumption for pretreatment was 23.7 % of the gross electric energy gain and 7.9 % of the overall output.

With reference to the use of biomethane as a biofuel, the investigated pretreatment produced a net extra output of primary energy equal to 577.2 kWh t^{-1} of processed feedstock, achieving a 41.3 % net gain over the raw material. Primary energy consumption for pretreatment was 17.6 % of the gross primary energy gain and 5.9 % of the overall output.

A recent work [34] studied a mechanical pretreatment of wheat straw, which was able to improve methane production by 13 % from 245 to 278 Nm^3 t_{VS}^{-1}. A very low specific energy consumption (29 kWh t^{-1}) was measured to produce final straw sizes lower than 10 mm; however, the size of the equipment was not declared. The net electric energy gain after pretreatment was about 80.8 kWh t^{-1}, thus far below the results of the present study.

With reference to previous studies carried out on industrial scale, a mixture of ensiled rice straw together with corn and triticale silage was pretreated by an industrial extruder, whose working capacity reached up to 5 t h^{-1} [35]. The electric energy required to pretreat the samples increased from 10.1 to 12.4 kWh t^{-1} as the mass of rice straw in the mixture was enhanced from 10 to 30 %. The electrical yield enhanced about by 10 %

at the lower concentration of straw, decreasing to almost nothing as the quantity of straw reached 30 % of the feed. In comparison to these results, the energy consumption of the device investigated in the present study was higher, but the electrical energy gain was far better.

5. CONCLUSIONS

A mechanical, industrial scale process was investigated for pretreating straw through multiple milling stages and fractionation. The two products obtained from the process achieved a methane yield of 245.6 and 264.7 Nm^3 t_{VS}^{-1}. On average, the methane yield was 210.9 Nm^3 per ton of processed material (wet basis), achieving a yield gain of 50.1 % over the untreated material.

Mechanical pretreatments have several strengths, such as very short residence times and the absence of any effluent, but the high demand of electric energy is their main drawback.

The energy consumption of the investigated process was 66.6 kWh t^{-1} and its energy balance was largely positive. The net energy gain for CHP applications was equal to 213.4 kWh t^{-1} of processed raw material, achieving a 38.2 % net gain over the untreated material.

With reference to the use of biomethane as a biofuel, the net gain of primary energy was equal to 577.2 kWh t^{-1} of processed feedstock, achieving a 41.3 % gain over the unprocessed straw. material.

These gains outperformed the performances of the previously described, mechanical, industrial scale processes.

As a consequence, the results of the present study suggest that the investigated pretreatment can be successfully implemented to improve methane production efficiency in anaerobic digesters, also reducing production costs. In fact, at current prices of agricultural products in Italy, corn silage delivered to the digestion plant costs about 45 € t^{-1}, resulting in a specific raw material cost of about 0.4 € $Nm^{-3}CH_4$. Whereas the cost of straw is about 60 € t^{-1}, resulting in a specific raw material cost of about 0.29 € $Nm^{-3}CH_4$, taking also into account the electric energy required by the pretreatment device, whose cost was assumed equal to 0.18 € kWh^{-1}.

Moreover, straw is listed among the feedstock allowed for the production of "advanced biofuels", and the pretreated material can be successfully used in the newborn biomethane industry in Italy.

ACKNOWLEDGMENT

The authors thank Scienza per Amore Association (National Research Register, n. 61097BTH) for having funded this research, Mr. Danilo Speranza and all those who gave their assistance during the field work.

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NOMENCLATURE

ADF	Acid detergent fiber
ADL	acid detergent lignin
CH_4	methane
CP	crude protein
CHP	combined heat and power
d_{50}	median particle size, μm
EE	ether extract
kWh	kilowatt hour
NDF	neutral detergent fiber
Nm^3	cubic meters at normal conditions
TS	total solids
VS	volatile solids