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Biomethane potential of wheat straw: Influence of particle size, water impregnation and thermal hydrolysis

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HIGHLIGHTS

- Assessment of the influence of thermal pretreatment on the anaerobic biodegradation of wheat straw.
- Optimum severity factor at 200 °C and 5 min (3.6 severity factor).
- Evaluation through BMP tests: 27% increase in methane productivity of steam exploded straw respect untreated straw.
- Cutting (3–5 cm) wheat straw showed to be better than milling (<1 mm).

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ABSTRACT

The anaerobic digestion of organic wastes such as wheat straw represents a very interesting means of generating biogas while reducing the amount of waste to disposal. An enhancement in the hydrolysis limited digestion of straw can be achieved by optimizing operation and performing pre-treatments. In this study, the influence of particle size, water impregnation and thermal pre-treatment was investigated through biochemical methane potential tests (BMP). The maximum methane yield was obtained by heating the straw at 200 °C for 5 min followed by steam explosion, obtaining a 27% increase in methane productivity compared to non-treated straw (from 233 to 296 mL CH_4/gVS_{fed}). Cutting (3–5 cm) showed to be better than milling (<1 mm), and the impregnation of the straw with water helped to enhance BMP test results by 4–10% (supposed better mixing due to a 10 times reduction of solids concentration) but had no effect on thermal pre-treatment. On the contrary, the economic impact of milling and water addition on a thermal pre-treatment would be absolutely negative, increasing the operation cost necessary to reduce the size and to heat water, respectively.

1. Introduction

Wheat straw is the largest agricultural residue in Europe, and the second largest in the world, after rice straw [1,2]. Nowadays straw is either used as bedding material for livestock, applied to the soil as natural fertilizer or as biomass for energetic valorization. The search for renewable energy sources together with the concern on greenhouse gas emissions have increased the interest on lignocellulosic materials as a source of energy [3,4], which is particularly well suited for energy applications because of its large-scale availability, low cost and large production.

Anaerobic digestion of biomass is a more economical and environmentally beneficial way of biomass utilization compared to typical pathways to biodiesel or bioethanol [5].

However, the main obstacle impeding a more widespread application of straw as feedstock for anaerobic digestion is its low digestibility due to its refractory structure. Like other lignocellulosic biomass, wheat straw is a complex mixture of cellulose, hemicellulose and lignin. Bioconversion of wheat straw is favored because of its relatively low lignin content (15–20%) and high carbohydrate content (30–40 and 20–30%w/w cellulose and hemicellulose, respectively) [6]. Lignin surrounds and seals the cellulose structure while hemicellulose serves as a connection between both of them [7]. Therefore, hydrolysis is a slow and difficult process [8,9].

In order to improve the biodegradability of wheat straw, several methods have been investigated, such as mechanical size reduction from the organic particulate matter [10–13], or the introduction of a lysis pre-treatment, such as physico-chemical alkaline dilution [14], microwave pre-treatment [15,16] or thermal steam explosion [17–19]. This last option has proven to be very interesting, as it can be cost effective if a proper energy recovery is performed.

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Hydrothermal steam explosion is performed without the addition of chemicals or oxygen, representing a potential solution for the pre-treatment of large quantities of lignocellulosic biomass [1,20].

In a steam explosion, biomass is heated and rapidly discharged to atmospheric pressure causing the explosion of the macromolecules, with the aim of upgrading the digestibility of lignocellulosic materials, by increasing porosity, removing lignin content and promote hemicellulose hydrolysis [18,21]. Although the successful use of steam explosion has been proved from the point of view of fermentation and ethanol production [1,22–25], the evaluation of the methane potential of steam-exploded straw is more recent [19,21,26], and open to many other wastes (corn stover, maizecrop waste, rice straw, herbaceous waste, manure, sewage sludge).

The studies performed specifically with wheat straw [1,18,21] have shown that the effect of the thermal pre-treatment depends on several factors, such as chip size, temperature and time. It is generally accepted that there is an optimum temperature in the range of 150–220 °C and 5–20 min, but care must be paid for too severe treatments due to the formation of inhibiting phenolic and heterocyclic compounds, such as furfural [10].

Chip size is a crucial parameter, as any sort of cutting or milling is necessary to avoid clogging and heat transfer problems during thermal treatment (overcooking the outside of large chips and formation of inhibitory compounds). However, a promising finding is that enzymatic hydrolysis is improved for larger biomass particle sizes [27], as milling is an energy intensive and expensive process. Regarding the anaerobic digestion process, the effect of particle size reduction on methane yield of agricultural wastes is contradictory: positive in some studies [12,28,29], while negative in others [30,31]. Therefore, the influence of particle size on wheat straw digestibility is still open to research.

The effect of water impregnation on thermal hydrolysis is a novel study in this paper. The impregnation of straw with acid or alkali has been successfully applied in enzymatic hydrolysis, while the influence of humidity on steam explosion effect is unexplored from the point of view of later methanization. From another point of view, dilution can be imperative to avoid overload or inhibition during the anaerobic digestion [32].

The aim of the present study is to evaluate the effect of particle size, dilution and thermal pre-treatment variables (temperature, time and water impregnation) on the biodegradability of wheat straw. For this purpose, batch anaerobic biodegradability tests were performed in order to check the biochemical methane potential (BMP) under different milling, washing and thermal hydrolysis conditions to determine individual and combined effects. Furthermore, a kinetic model has been used to obtain the specific rate constants to assess the relationship of the parameters evaluated.

2. Materials and methods

2.1. Raw material and experimental set-up

Wheat straw was grown in Valladolid (Spain), harvested in 2012 and characterized (Table 1). The original straw was ground (3–5 cm) or milled (<1 mm), according to the experimental set-up in Table 2.

Three series of experiments (A, B and C) were performed (Table 2) to cover the three scenarios to study: (A) influence of particle size and water dilution on wheat straw digestibility; (B) influence of steam explosion pre-treatment; and (C) influence of water impregnation time on thermal hydrolysis and digestion.

Table 1
Average characteristics of the original wheat straw.

	TS (g/kg)	VS (g/kg)	TCOD (g/kg)	TKN (g N/kg)	TOC (% weigh)	C/N
Series A–B	895 ± 11	821 ± 9	1075 ± 8	4.723	43.2 ± 0.3	92
Series C	924 ± 9	846 ± 5	1089 ± 6	4.578	43.4 ± 0.2	92

Table 2
Experimental set-up.

Series A			Series B				Series C			
Test	Particle size	Dilution	Test	T (°C)	t (min)	log R_0	Test	T (°C)	t (min)	Washing time (h)
A1	3–5 cm	No	B0	Untreated			C1			0
A2	3–5 cm	Yes	B1	170	15	3.2	C2	200	5	3
A3	<1 mm	No	B2	200	5	3.6	C3			12
A4	<1 mm	Yes	B3	220	1	3.5	C4			24

2.2. Particle size reduction and water impregnation

Based on bibliography (where references to particle sizes ranging from 0.2 mm to 10 cm can be found), two particle sizes were selected for series A: 3–5 cm pieces and powder <1 mm. In most of the references, the particle size influence is not assessed but established in the range of 1–5 cm [12,18,21,33].

The larger particle size was chopped with a cramp to get the desired interval 3–5 cm. A laboratory mill (Philips, HR7775) was used to grind the straw into a minor particle size (<1 mm) controlled with a sieve (CISA™). In the studies of series B and C only the major particle size were used.

Water addition in series A and C was performed by mixing the straw with water. In series A, the water was added when preparing the BMP tests (1:10 dilution), while in series C the straw was soaked for a desired washing time.

2.3. Thermal steam explosion pre-treatment unit plant

The pre-treatment was performed at the steam explosion pilot plant facility designed by Cambi AS and located at the wastewater treatment plant of Salamanca, Spain.

The steam explosion unit consists of a 30 L reactor vessel and a flash tank with a removable bucket to collect the pretreated material (Fig. 1). The steam is generated by a 25 kW electric steam boiler (200 L capacity) which can supply steam up to a maximum pressure of 34 bar (240 °C). Wheat straw is loaded into the reactor using a motorized ball valve (V1) at the top of the reactor. Steam is added to the reactor from the bottom, through an air-actuated valve (V2), heating the waste during the time established. The desired operation pressure (corresponding to a certain temperature) is set on the control panel unit, controlled automatically by the air-actuated valve (V2). For security reasons also a manual valve (V3) has to be opened to add steam to the pressure reactor. An air-actuated ball valve at the bottom of the vessel (V4) is responsible for the rapid pressure drop (explosion) and release of the pretreated biomass to the flash tank. The pretreated biomass is collected in a removable bucket at the bottom of the flash tank. Any steam that is not condensed leaves the unit via a carbon filter to remove smell.

In all the experiments, one kilogram of wheat straw was used. The reactor was fed to the unit and the reactor was pre-heated for 15 min before starting the experiments.

The effects of temperature and time were evaluated based on the severity factor ($\log R_0$, Eq. (1)), which is the common term used in steam pre-treatments [11]:

$$\log R_0 = \log\left(t \cdot \exp\left(\frac{T-100}{14.75}\right)\right) \quad (1)$$

where t is the time (min) and T the temperature (°C).

Different pre-treatment conditions were tested varying temperature (ranging 170–220 °C) and time (ranging 1–15 min), based on

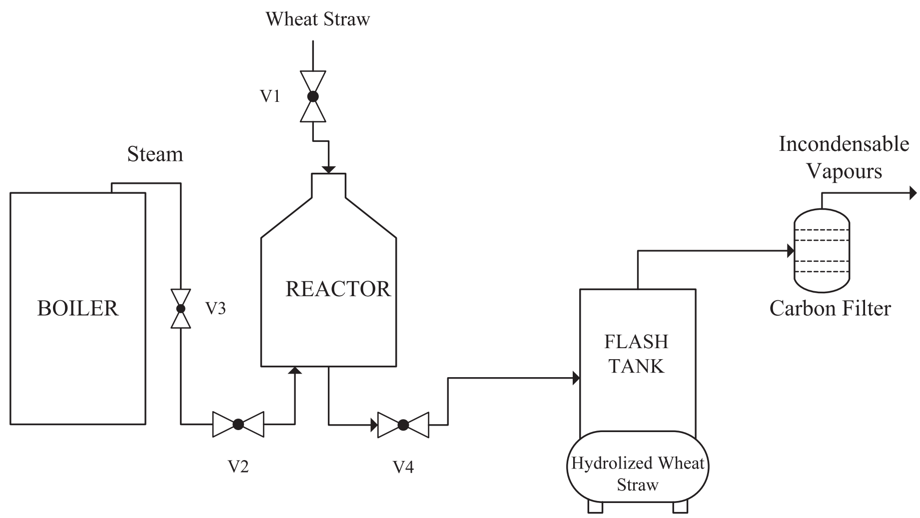

Fig. 1. Cambi SA steam explosion unit. V, valves; VM, motorized valves; VA,B, one way valves; P, manometers to measure pressure; RD, safety valves; CF, carbon filter; TB-T, treated biomass tank; and WT, water tank.

previous studies [14,21,26] concluding that the optimum severity factor ($\log R_0$–Eq. (1)) is in the range 3.3–3.6, and this criteria was considered to establish the combinations of temperature and time for series B.

2.4. Biochemical methane potential tests (BMP)

Batch anaerobic digestion tests were carried out in order to assess the wheat straw biodegradability after the different pre-treatment conditions applied. All tests were in triplicate in a 2 L borosilicate glass (260 mm height, 160 mm diameter and a 40 mm bottleneck) with 400 mL of a mixture of wheat straw and inoculum (with 12 g VS/kg and collected from a pilot digester treating waste activated sludge at 35 °C). The substrate to inoculum ratio (S/I) selected was 0.5 g VS/g VS as suggested in a previous researchers [32,34]. A control test without substrate was included in order to check the methanogenic activity of the inoculum.

Before starting the test, the bottles were closed with rubber stoppers and aluminum crimps and degassed. Helium gas was circulated in the gas chamber for 5 min, and the test started after releasing the pressure. All the experiments were carried out at mesophilic conditions in a thermostatic room (35.1 ± 0.3 °C), with constant mixing in a rotary desk. All the assays were finished when the methane production was below 5% of the total cumulative production.

The biogas volume was monitored by period measurements of the headspace pressure by a manually pressure transmitter (ifm, PN5007, range 1 bar).

The methane production of a control test performed with only inoculum was subtracted to obtain the real methane production from the straw. This value was finally expressed as specific methane yields (mL CH_4/g VS_{fed}), presented under standard temperature and pressure conditions (STP-0 °C, 1 atm) defined by IUPAC (International Union of Pure and Applied Chemistry), and divided by the mass of volatile solids of substrate fed into to the assay.

The kinetics of methane production was calculated using a first-order model (Eq. (2)), applied successfully in other reports on anaerobic biodegradability tests [26,35].

$$B = P \cdot \left(1 - exp\left(\frac{-Rm(t-\lambda)}{P}\right)\right) \quad (2)$$

where B is the methane production (mL CH_4/gVS), P is the maximum methane production (mL CH_4/gVS), Rm is the maximum biogas production rate (mL CH_4/gVS·d), λ is the lag time (d) and t is the time of the assay (d). The data were analyzed with Statgraphics® [36].

2.5. Analytical methods

Total and volatile solids (TS and VS) and total Kjeldahl nitrogen (TKN) were measured following the procedures given in Standard Methods for Examination of Water and Wastewater [37]. Total chemical oxygen demand (TCOD) was determined according to standard UNE 77004:2002 based in dichromate method [38]. A combustion infrared method, with SHIMADZU TOC-SM5000A equipment, was used to determine the total organic carbon (TOC). The biogas composition (CO_2, H_2S, O_2, N_2, CH_4) was measured by gas chromatography in a Varian equipment CP-3800 CG TCD, being helium the carrier gas.

3. Results and discussion

3.1. Series A: influence of particle size and dilution on wheat straw digestibility

Fig. 2 and Table 3 present the results for the methane production curves (from BMP tests) and kinetic parameters for the series of tests A.

The results show that when adding water (tests A2–A4 compared to A1–A3) methane production slightly increased (4% increase for test A2 compared to A1, at 3–5 cm straw size, and 10% increase for test A4 compared to A3, at 1 mm straw size), and also did the production rate (5% increase for test A2 compared to A1, and 15% increase for test A4 compared to A3). The reason is probably related with a better mixing in the BMP tests performed with water (A2 and A4), as the solids concentration in the test decreased from 200 g/kg to 20 g/kg.

Analyzing the influence of the particle size by comparing tests A1–A2 with A3–A4, it can be observed that the methane production was 5–13% higher for the larger particle size, and the kinetics were also faster. These results are in agreement with those obtained by De la Rubia et al. [30] for sunflower oil cake, and Izumi et al. [31] for food waste, but disagree with the results obtained by other authors.

Sharma et al. [39] found a significant increase in methane productivity of wheat straw by size reduction from 30 mm

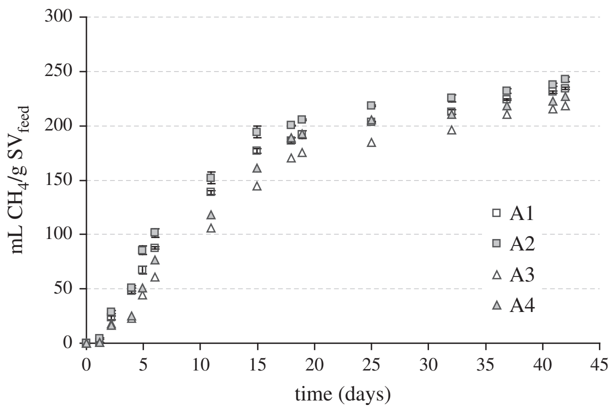

Fig. 2. Methane yield for series A.

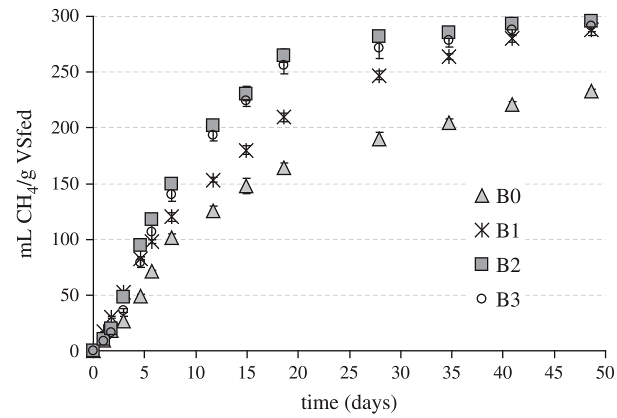

Fig. 3. Methane yield for series B.

Table 3
Results for methane yield and kinetic parameters for series A.

Parameter	A1	A2	A3	A4
P (mL CH_4/gVS)[a]	239	245	232	239
Rm (mL CH_4/gVS.d)[b]	20.2	23.2	15.9	18.3
λ (day)[c]	0.9	0.8	1.2	1.2
R^2	0.996	0.994	0.991	0.988

[a] P, maximum methane production.
[b] Rm, maximum biogas production rate.
[c] λ, lag time (according to Eq. (2)).

Table 4
Results for methane yield and kinetic parameters for series B.

Parameter	B0	B1	B2	B3
P (mL CH_4/gVS)[a]	245	291	304	301
Rm (mL CH_4/gVS.d)[b]	16.1	20.6	28.5	26.2
λ (day)[c]	0.8	0.4	0.9	1
R^2	0.985	0.997	0.995	0.991

[a] P, maximum methane production.
[b] Rm, maximum biogas production rate.
[c] λ, lag time (according to Eq. (2)).

(192 mL/gVS) to 1 mm (241 mL/gVS) but only a small effect for further size reduction to 0.1 mm. Hjorth et al. [29] obtained 70% increase in methane production for extruded straw respect non-extruded straw (150 mL/gVS), although no particle size values for treated straw are given. Friction heat and shear forces in the extruder could play an important role additional pre-treatment to the cutting. Palmowski and Müller [12] obtained 57% and 86% increase in methane yield for particles of 5 cm and 0.2 cm respectively, as compared to the untreated sample (182 mL/gVS).

Putting together these results with those obtained in the present research, it can be concluded that the composition of the bio-waste should be assessed to compare small and large chip sizes, as the content of carbohydrates, proteins and lipids is not uniform in the different particle size fractions but have a clear different methane potential. If no organic matter is removed during the mechanical treatment cutting seems to be better to milling or grinding in order to minimize the energy input, as milling is not considered economically feasible due to the high energy requirements,

Based on these results, wheat straw with 3–5 cm was selected to be used in series B and C.

3.2. Series B: influence of steam explosion pre-treatment

The results of the BMP tests performed for series B and the kinetic parameters obtained are presented in Fig. 3 and Table 4, where B0 corresponds to non-treated wheat straw, cut to 3–5 mm.

In all steam-explosion experiments (B1–B3), the methane yield was higher than B0 (233 mL/dVS) in the range of 24–27% (288–296 mL/gVS). The kinetic study shows that the methane production rate was also 19–24% higher. Although similar results for tests B1–B2–B3, the highest productivity and production rate were for test B2, performed at 200 °C for 5 min (increase of 27% in the methane yield), corresponding to a severity factor ($\log R_0$) of 3.64.

These results prove the effectiveness of the steam explosion pre-treatment, and concur with those obtained by other authors. The study published on methane production from steam-exploded wheat straw [21] agrees with the results obtained here. Bauer et al. [21] increased methane production from 275 to 331 mL/gVS when treating the straw at 180 °C for 15 min [26] have previously optimized the pre-treatment conditions to a severity factor in the range 3.25–3.53, finding the optimum at 200 °C and 5 min. This optimal agrees with the one here obtained.

3.3. Series C: influence of water impregnation time on thermal hydrolysis

This series was conducted with 3–5 cm wheat straw thermally pretreated at 200 °C for 5 min (according to the optimum results for series A and B), with the objective of evaluating if water impregnation of wheat straw had any positive effect on the pre-treatment. The hypothesis that supports the theoretical possibility of increasing the steam explosion effect by washing the straw is the chance of the water to penetrate in the biomass structure,

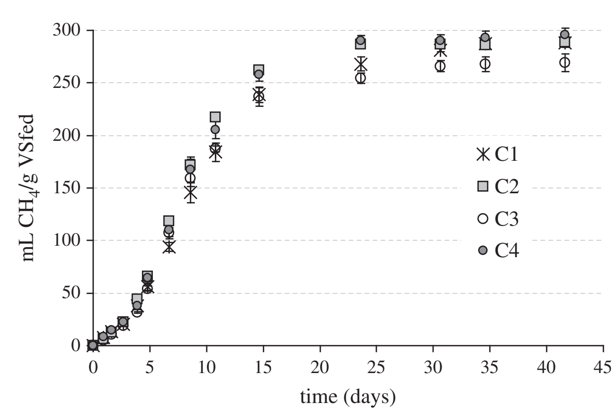

Fig. 4. Methane yield for series C.

Table 5
Results for methane yield and kinetic parameters for series C.

Parameter	C1	C2	C3	C4
P (mL CH_4/gVS)[a]	316	315	293	323
Rm (mL CH_4/gVS.d)[b]	23.4	28.2	24.4	26.7
λ (day)[c]	1.3	1.3	1.4	1.3
R^2	0.971	0.963	0.962	0.964

[a] *P*, maximum methane production.
[b] *Rm*, maximum biogas production rate.
[c] λ, lag time (according to Eq. (2)).

leading to a possible stronger lysis effect by steam explosion during the pressure drop.

Fig. 4 and Table 5 present the results for the methane yield and kinetic parameters for series of tests C. The results show a negligible influence of the washing time on the thermal steam explosion pre-treatment, as the results obtained were very similar in the different tests. Only the kinetics showed to be slightly faster, but negligible. Therefore, the water added probably did not penetrate in the wheat straw structure, and did not help to disrupt the fibrils in the decompression step.

Although according to Jakoviak et al. [16], the humidity of the biomass may cause differences on the contents of cellulose and hemicellulose of wheat straw, the evaluation of the chemical composition of the straw in terms of lipids, carbohydrates and proteins would again be determining to confirm any hypothesis.

4. Conclusions

The methane production of 30–50 mm wheat straw pieces was 10.4% higher than the powder <1 mm. Therefore, for process performance and economics, cutting is desirable to milling. Thermal pre-treatment enhanced methane production by 19–24% for temperatures in the range 170–220 °C and 1–15 min heating time, being the optimum for 200 °C and 5 min, which increased methane yield by 27% (from 233 to 296 mL CH_4/gVS_{fed}). The impregnation of straw with water showed a poor positive effect in the BMP tests, but no effect on thermal steam explosion, apart from a clear worsening of the process economics if water was added.

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References

[1] S.J. Horn, Q.D. Nguyen, B. Westereng, P.J. Nilsen, V.G.H. Eijsink, Screening of steam explosion conditions for glucose production from non-impregnated wheat straw, Biomass and Bioenergy 35 (2011) 4879–4886.
[2] F. Talebnia, D. Karakashev, I. Angelidaki, Production of bioethanol from wheat straw: an overview on pre-treatment, hydrolysis and fermentation, Bioresource Technology 101 (2010) 4744–4753.
[3] P. Alvira, E. Tomás-Pejó, M. Ballesteros, M.J. Negro, Pretreatment technologies for an efficient bioethanol production process based on enzymatic hydrolysis: a review, Bioresource Technology 101 (2010) 4851–4861.
[4] C. Zhong, Y.X. Cao, B.Z. Li, Y.J. Yuan, Biofuels in China: past, present and future, Biofuels Bioprod. Biorefining 4 (2010) 326–342.
[5] R. Chandra, H. Takeuchi, T. Hasegawa, Methane production from lignocellulosic agricultural crop wastes: a review in context to second generation of biofuel production, Renewable and Sustainable Energy Reviews 16 (2012) 1462–1476.
[6] S. Prasad, A. Singh, H.C. Joshi, Ethanol as an alternative fuel from agricultural, industrial and urban residues, Resources Conservation and Recycling 50 (2007) 1–39.
[7] K. Ross, G. Mazza, Comparative analysis of pyrolysis products from a variety of herbaceous canadian crop residues, World Journal of Agricultural Sciences 7 (6) (2011) 763–776.
[8] G. Radeva, I. Valchev, S. Petrin, E. Valcheva, P. Tsekova, Kinetic model of enzymatic hydrolysis of steam-exploded whaet straw, Carbohydrate Polymers 87 (2012) 1280–1285.
[9] J. Puls, J. Schuseil, Chemistry of hemicelluloses – relationship between hemicellulose structure and enzymes required for hydrolysis, in: M.P. Coughlan, G.P. Hazlewood (Eds.), Hemicellulose and Hemicellulases, vol. 4, Portland Press, London, 1993, pp. 1–27.
[10] A.T.W.M. Hendriks, G. Zeeman, Pretreatments to enhance the digestibility of lignocellulosic biomass, Bioresource Technology 100 (2009) 10–18.
[11] S. Menardo, G. Airoldi, P. Balsari, The effect of particle size and thermal pre-treatment on the methane yield of four agricultural by-products, Bioresource Technology 104 (2012) 708–714.
[12] L.M. Palmowski, J.A. Müller, Influence of the size reduction of organic waste on their anaerobic digestion, Water Science and Technology 41 (3) (2000) 155–162.
[13] H.A. Ruiz, D.S. Ruzene, D.P. Silva, M.A.C. Quintas, A.A. Vicente, J.A. Teixeira, Evaluation of a hydrothermal process for pretreatment of wheat straw – effect of particle size and process conditions, Journal of Chemical Technology and Biotechnology 86 (2011) 88–94.
[14] S. McIntosh, T. Vancov, Optimisation of dilute alkaline pretreatment for enzymatic saccharification of wheat straw, Biomass and Bioenergy 35 (2011) 3094–3103.
[15] Z. Sapci, The effect of microwave pretreatment on biogas production from agricultural straws, Bioresource Technology 128 (2013) 487–494.
[16] D. Jackowiak, D. Bassard, A. Pauss, T. Ribeiro, Optimisation of a microwave pretreatment of wheat straw for methane production, Bioresource Technology 102 (2011) 6750–6756.
[17] S. Zahibi, R. Alinia, F. Esmaeilzadeh, J.F. Kalajahi, Pretreatment of wheat straw using steam, steam/acetic acid and steam/ethanol and its enzymatic hydrolysis for sugar production, Biosystems Engineering 105 (2010) 288–297.
[18] G. Han, J. Deng, S. Zhang, P. Bicho, Q. Wu, Effect of steam explosion treatment on characteristics of wheat straw, Industrial Crops and Products 31 (1) (2010) 28–33.
[19] L.H. Zhang, D. Li, L.J. Wang, T.P. Wang, L. Zhang, X.D. Chen, Z.H. Mao, Effect of steam explosion on biodegradation of lignin in wheat straw, Bioresource Technology 99 (2008) 8512–8515.
[20] K. Ohgren, A. Rudolf, M. Galbe, G. Zacchi, Fuel ethanol production from steam-pretreated corn stover using SSF at higher dry matter content, Biomass and Bioenergy 30 (2006) 863–869.
[21] A. Bauer, P. Bosch, A. Friedl, T. Amon, Analysis of methane potentials of steam-exploded wheat straw and estimation of energy yields of combined ethanol and methane production, Journal of Biotechnology 142 (2009) 50–55.
[22] X.F. Sun, F. Xu, R.C. Sun, Z.C. Geng, P. Fowler, M.S. Baird, Characteristics of degraded hemicellulosic polymers obtained from steam exploded wheat straw, Carbohydrates Polymers 60 (2005) 15–26.
[23] I. Ballesteros, M.J. Negro, J.M. Oliva, A. Cabanas, P. Manzanares, M. Ballesteros, Ethanol production from steam-explosion pretreatment wheat straw, Applied Biochemistry and Biotechnology 129–132 (2006) 496–508.
[24] C. Hongzhang, L. Liying, Unpolluted fractionation of wheat straw by steam explosion and ethanol extraction, Bioresource Technology 98 (2007) 666–676.
[25] G. Panagiotou, L. Olsson, Effect of compounds released during pretreatment of wheat straw on microbial growth and enzymatic hydrolysis rates, Biotechnology Bioengineering 96 (2007) 250–258.
[26] L.C. Ferreira, A. Donoso-Bravo, S.I. Pérez-Elvira, P.J. Nilsen, F. Fdz-Polanco, Influence of thermal pretreatment on biomethane potential of wheat straw, Bioresource Technology 143 (2013) 251–257.
[27] Z.-H. Liu, L. Qin, F. Pang, M.-J. Jin, B.-Z. Li, Y. Kang, Effects of biomass particle size on steam explosion pretreatment performance for improving the enzyme digestibility of corn stover, Industrial Crops and Products 44 (2013) 176–184.
[28] J.P. Delgenès, V. Penaud, R. Moletta, Pretreatments for the enhancement of anaerobic digestion of solid wastes, in: J. Mata-Alvarez (Ed.), Biomethanization of the Organic Fraction of Municipal Solid Wastes, IWA Publishing, London, 2003, pp. 201–228.
[29] M. Hjorth, K. Gränitz, A.P.S. Adamsen, H.B. Møller, Extrusion as pretreatment to increase biogas production, Bioresouce Technology 102 (2011) 4989–4994.
[30] M.A. De la Rubia, V. Fernández-Cegrí, F. Raposo, R. Borja, Influence of particle size and chemical composition on the performance and kinetics of anaerobic digestión process of sunflower oil cake in batch mode, Biochemical Engineering Journal 58–9 (2011) 162–167.
[31] K. Izumi, Y. Okishio, N. Nagao, C. Niwa, S. Yamamoto, T. Toda, Effects of particle size on anaerobic digestion of food waste, International Biodeterioration and Biodegradation 64 (2010) 601–608.
[32] I. Angelidaki, M. Alves, D. Bolzonella, L. Borzacconi, J.L. Campos, A.J. Guwy, S. Kalyuzhnyi, P. Jenicek, J.B. Van Lier, Defining the biomethane potential (BMP) of solid organic wastes and energy crops: a proposed protocol for batch assays, Water Science and Technology 59 (5) (2009) 927–934.
[33] W. Qiu, H. Chen, Enhanced the enzymatic hydrolysis efficiency of wheat straw after combined steam explosion and laccase pretreatment, Bioresource Technology 118 (2012) 8–12.
[34] L. Neves, R. Oliveira, M.M. Alves, Influence of inoculum activity on the bio-methanization of a kitchen waste under different waste/inoculum ratios, Process Biochemistry 39 (2004) 2019–2024.
[35] S.I. Pérez-Elvira, L.C. Ferreira, A. Donoso-Bravo, M. Fdz-Polanco, F. Fdz-Polanco, Full-stream and part-stream ultrasound treatment effect on sludge anaerobic digestion, Water Science and Technology 61 (6) (2010) 1363–1372.

[36] Statgraphics Centurion 15.2.14 (XV), STATPOINT Technologies, INC, Warrenton, Virginia, 2010.
[37] APHA, AWWA, WPDF, Standard Methods for the Examination of Water and Wastewater, 21st ed., American Public Health Association, Washington DC, 2005.
[38] Water Quality, Determination of the chemical oxygen demand (COD), Dichromate Method, UNE 77004:2002.
[39] S.K. Sharma, I.M. Misha, M.P. Sharma, J.S. Saini, Effect of particle size on biogas generation from biomass residues, Biomass 17 (1988) 251–263.