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Thermal steam explosion pretreatment to enhance anaerobic biodegradability of the solid fraction of pig manure

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HIGHLIGHTS

- Thermal steam-explosion (TH) pretreatment of the solid fraction of pig manure.
- Influence of the TH pre-treatment conditions (T, t) on anaerobic degradation (AD).
- TH improved the AD of pig manure, in biodegradability, and also degradation rates.
- Optimum at 170 °C and 30 min (3.54 severity factor).
- Double methane productivity compared to untreated (from 159 to 329 mL CH_4/gVS_{fed}).

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ABSTRACT

The assessment of the biodegradability of thermal steam-exploded pig manure was performed compared to untreated samples. The pre-treatment was performed under different combinations of temperature and time, ranging 150–180 °C and 5–60 min, and used as substrate in a series of batch biochemical methane potential (BMP) tests. Results were analyzed in terms of methane yield, kinetic parameters and severity factor. In all the pre-treatment conditions, methane yield and degradation rates increased when compared to untreated pig slurry. An ANOVA study determined that temperature was the main factor, and the optimum combination of temperature–time of pretreatment was 170 °C −30 min, doubling methane production from 159 to 329 mL CH_4/gVS_{fed}. These operation conditions correspond to a severity factor of 3.54, which was considered an upper limit for the pretreatment due to the possible formation of inhibitory compounds, hindering the process if this limit is exceeded.

1. Introduction

In Spain, currently the second largest producer of pig manure in the European Union (PNIR, 2007–2015), manure management has become an environmental issue. The traditional application of manure is as land fertilizer (Schröder, 2005), contributing to maintaining the soil organic carbon and nutrients stocks. However, due to the intensification of livestocks production, in many areas the organic waste nutrients exceed the requirement for crop production (Wnetrzak et al., 2013), resulting in groundwater contamination, nitrates leaching and eutrophication. Therefore, any kind of treatment for pig wastes must be implemented according to the environmental legislation, such as the EU Nitrates Directive (Good Agricultural Practice for Protection of Wastes; Regulations 2010).

The valorization of the organic matter content in pig manure to biogas through anaerobic digestion (AD) is a very interesting option (Krishania et al., 2013; Mata-Alvarez et al., 2000), although ammonia inhibition issues have to be taken into account for considering manure as a sole substrate (Hansen et al., 1998). However, similar to other types of organic wastes, biogas production from pig manure is relatively low: from 290 to 550 L CH_4/kg of organic matter (Burton and Turner, 2003), due to the limiting hydrolysis step of the fiber content (Menardo et al., 2011). Some treatment options are reported to increase AD performance, such as acidification (Moset et al., 2012; Sutaryo et al., 2013) or separation of solid and liquid fractions (Fangueiro et al., 2012; Møller et al., 2007).

Thermal pre-treatment appears as a very interesting option and a potential solution for a large quantity of lignocellulosic biomass. The use of a thermal pre-treatment to enhance the anaerobic digestion of sewage sludge is reported in several references (Bougrier et al., 2008; Pérez-Elvira et al., 2011), and has been developed full scale. However, the application to other organic wastes such as agricultural wastes (Ferreira et al., 2013; Qiao et al., 2011) or manure is very recent, and still open to research. Some references can be found concerning thermal pretreatment to chicken manure (Ardic and Taner, 2005; Bujoczek et al., 2000), swine manure (Chae et al., 2008; Mladenovska et al., 2006), dairy manure (Rico et al.,

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2011,2012; Yoneyama et al., 2006) and pig manure (Bonmatí et al., 2001; Carrère et al., 2009; Cuetos et al., 2008; Menardo et al., 2011). In the references found on thermal pre-treatment of manure the study was performed considering only the effect of temperature (with or without chemicals), but no evaluation on the influence or optimization of temperature–time combinations are reported.

Finally, the application of any thermal technology requires to concentrate the waste (Pérez-Elvira and Fdz-Polanco, 2012). Several references can be found about separation of solid and liquid fractions of pig slurry in order to economize transport or increase the energy potential through biogas production (Møller et al., 2007; Nolan et al., 2012; Xie et al., 2012).

The aim of this study was to evaluate the methane yield of steam exploded pig slurry (separated solid fraction of pig manure) under different temperature–time combinations, compared to a control of untreated slurry. BMP tests were performed and kinetic parameters were evaluated.

2. Methods

2.1. Manure samples: thickening and characterization

Pig slurry was collected from a piggery in Segovia (Spain), and corresponds to the original excrements of pigs, not mixed with washing water. The original sample was characterized before and after centrifugation at 1680*g* for 5 min to get the solid fraction. Table 1 presents the results from the analytical characterization for both the original sample and the solid fraction.

As expected, the concentrated pig slurry (manure) presented a higher percentage of volatile solids, which means a higher content of organic matter. The C/N ratio increased when compared to the original sample, which allows better conditions for the treatment.

2.2. Thermal pretreatment pilot plant and operation conditions

The thermal hydrolysis plant consisted of a reactor (2L) connected to a boiler by a control valve (V1) and to a flash tank (5L) by a decompression valve (V2). The operation was batch: 250 g of pig manure were fed to the reactor, and then heated with steam from the boiler (180 °C, 10 bar) for the desired period of time. Once completed the operation time, the steam inlet (V1) is closed and the decompression valve (V2) is opened in a sudden decompression, releasing the hydrolyzed pig slurry to the atmospheric flash tank. Before starting the experiments, the reactor was pre-heated for approximately 5 min at the same temperature selected for the pretreatment, in order to minimize condensation.

Different pretreatment conditions were tested varying both temperature (ranging from 120 to 180 °C) and time (ranging from 5 to 60 min), in a total of 15 different operation conditions, as presented in Table 2. The combination temperature and time of pretreatment define the severity factor (log R_0, Eq. (1)), also included in Table 2. This parameter is most widely accepted for steam pretreatments (Hendriks and Zeeman, 2009) to express the severity of the pretreatment.

$$\log R_0 = \log\left(t.exp\left(\frac{T - 10_0}{14.75}\right)\right) \quad (1)$$

in which t is the time (min) and T the temperature (°C), 100 is the base temperature (100 °C), and 14.75 is the activation energy based on the assumption that the reaction is hydrolytic and the overall conversion is first order (Xu et al., 2011). This expression only takes into account time and temperature, and does not consider the effect of the flash. The study of this effect was not assessed in this paper.

Theoretically, the more severe the pretreatment, the more organic matter is made available for digestion. However, very severe pretreatments can lead to formation of inhibitory compounds from the macromolecules, driving to an indirect relationship between the severity factor and the biodegradability. Therefore, there should be an optimum value for the severity factor.

2.3. Anaerobic biodegradability

Biochemical methane potential (BMP) tests were carried out in triplicate to assess the pig slurry biodegradability before and after the different pretreatment conditions applied, following standardized methodology (Angelidaki et al., 2009). A control test without substrate was included in order to check the methanogenic activity of the inoculum. All the experiments were carried out at mesophilic conditions in a thermostatic room (35.1 ± 0.3 °C), with constant mixing in a shaker table.

The anaerobic inoculum used was taken from a pilot sludge digester treating activated sludge (containing 12 g vs. kg^{-1}) and pre-incubated for four days (35.1 ± 0.3 °C) in order to minimize its residual biodegradable organic matter content.

Borosilicate glasses of 300 mL volume were used in the BMP tests, with a reaction volume of 110 mL in order to have enough headspace for biogas accumulation. The substrate/inoculum (S/I) ratio selected was 0.5 g vs. $(\text{gVS})^{-1}$, as suggested by Neves et al. (2004) and Angelidaki et al. (2009).

Biogas production was measured manually by a pressure transmitter (ifm, PN5007, range 1 bar) in the headspace of each reactor. From the biogas production curves, the specific methane yield (mL CH_4/gVS_{fed}) was calculated at test-day 20 under standard temperature and pressure conditions (STP – 0 °C, 1 atm) defined by IUPAC (International Union of Pure Applied Chemistry), by dividing the methane production due to the substrate (once subtracted the production due to the inoculum) by the quantity of volatile solids of substrate fed to the test.

2.4. Analytical methods

Substrates, inoculum and digestates were characterized in all the experiments. Total and volatile solids (TS and VS, respectively),

Table 1
Average characteristics of the original sample and solid fraction of pig manure (before and after centrifugation, respectively).

Parameter		Original sample	Solid fraction	Units
Total solids	TS	46.6 ± 2.1	166.4 ± 0.2	g/kg
Volatile Solids	VS	36.8 ± 2.7	138.6 ± 0.2	g/kg
Percentage of VS	% VS	79	83	–
Chemical oxygen demand	COD	54.20 ± 0.8	197 ± 3	gO_2/kg
Total kjeldahl nitrogen	TKN	7.40	6.05	gN/kg
Total organic carbon	TOC	6.29	7.48	% weight
Ratio C/N	C/N	8.5	12.4	–
Ammonium	NH_4^+	4671	–	gNH_4^+–N/g
Ratio COD/VS	COD/VS	1.47	1.42	–

Table 2
Thermal conditions applied, experimental results and kinetic parameters for the different experiments performed.

TH_i	T (°C)	t (min)	Severity Factor (log R_0)	FN	R_m (mL CH_4/gVS$_{fed}$ d)	λ (d)	P (mL CH_4/gVS$_{fed}$)	R^2
TH0	0	0	0.00	1.00	32.1	0.22	159	0.9852
TH1	120	5	1.29	1.26	31.6	0.10	200	0.9547
TH2	120	15	1.76	1.59	41.2	0.15	253	0.9737
TH3	120	30	2.07	1.63	48.2	0.12	259	0.9684
TH4	120	60	2.37	1.67	70.8	0.22	265	0.9821
TH5	150	5	2.17	1.70	47.9	0.21	271	0.9825
TH6	150	15	2.65	1.75	55.0	0.25	278	0.9876
TH7	150	30	2.95	1.72	78.3	0.32	273	0.9893
TH8	150	60	3.25	1.72	116.3	0.38	274	0.9875
TH9	170	5	2.76	1.84	63.1	0.45	292	0.9973
TH10	170	15	3.24	1.93	72.3	0.47	308	0.9950
TH11	170	30	3.54	2.07	106.1	0.45	329	0.9868
TH12	170	60	3.84	1.53	88.3	0.45	244	0.9844
TH13	180	5	3.05	1.99	70.6	0.39	317	0.9863
TH14	180	15	3.53	1.77	62.4	0.45	282	0.9971
TH15	180	30	3.83	1.70	87.7	0.47	271	0.9839

total kjeldahl nitrogen (TKN) and total organic carbon (TOC) were measured following the procedures given in the Standard Methods for Examination of Water and Wastewater (APHA et al., 2005). For the TOC determination, the infrared method was used, in a SHIMADZU TOC-SM5000A equipment. Chemical oxygen demand (COD) was determined according to standard UNE 77004:2002 based in the dichromate method. Biogas composition (CO_2, H_2S, O_2, N_2 and CH_4) was measured by gas chromatography in a Varian equipment CP-3800 CG TCD, being helium the carrier gas.

2.5. Data analysis and kinetic approach

Apart from obtaining the specific methane yields (mL CH_4/gVS$_{fed}$), a kinetic approach was also employed to analyze the obtained data. The kinetics of methane production were calculated using a reaction curve-type model (Eq. (2)), applied successfully in other studies regarding anaerobic biodegradability tests (Ferreira et al., 2013).

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

in which B is the methane production (mL CH_4/gVS), P is the maximum methane production (mL CH_4/gVS), R_m is the maximum methane 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®.

Finally, to evaluate the influence of temperature and time of pretreatment on methane production, the results obtained were compared by the analysis of variance methodology (ANOVA), using Microsoft Excel®, with a confidence level of 95%.

3. Results and discussion

3.1. Batch tests and methane yield

Fig. 1 presents the evolution of methane productivity (mL CH_4/gVS$_{fed}$) in all the BMP assays performed, including always the untreated substrate control (TH0). For better comparison, Table 2 presents the BMP test results as a factor (FN) that corrects the final specific methane yield with respect to the corresponding for the untreated substrate, at day 20.

3.2. Evaluation of temperature–time combinations

An initial evaluation of the results of the BMP (FN) presented in Table 2 show that all the pre-treatments performed (TH1–TH15) enhanced the initial biodegradability of the pig slurry (TH0). The smaller improvement was 26% (TH1: 120 °C, 5 min) while the best pre-treatment condition evaluated was TH11 (170 °C, 30 min), with an improvement respect untreated of 107%. When comparing the influence of temperature and time, a different behavior can be observed depending on the temperature level. For low temperatures (120 °C), the influence of pre-treatment time was the most noticeable, increasing the biodegradability of pig slurry from 26% for 5 min treatment, to 67% for 60 min. For moderate temperatures (150 °C), the influence of the pre-treatment time was minor, and the biodegradability improvement was always around 70% for any treatment time. For 170 and 180 °C, the optimum pre-treatment duration was 15–30 min and 5 min, respectively. However, longer pre-treatment caused a reduction in methane yield, probably due to the formation of inhibitory compounds, typically melanoidins. These recalcitrant compounds are produced by polymerization of low molecular weight intermediates, such as carbohydrates and amino compounds at elevated temperature (Maillard reaction).

Statistical analysis was carried out based on this qualitative analysis. The data concerning pre-treatment time of 60 min were not considered for this study, as methane yield was mostly inhibited. The results of the ANOVA study showed that in all cases temperature caused significant statistical difference between data ($F_{ratio} = 6.29$, $F_{critical} = 4.76$), while pre-treatment time did not ($F_{ratio} = 2.81$, $F_{critical} = 5.14$). This indicates that, when dealing with both variables, temperatures should be considered as the main parameter as the basis to define the best pre-treatment time.

Fig. 2a presents a graphical approach of the effect of both temperature and time on FN. It can be observed that the best improvements in methane yield are centered in the combination 170 °C, 30 min, which is in accordance to the globally accepted values regarding thermal hydrolysis of sludge (Fdz-Polanco et al., 2008). This suggests that these thermal hydrolysis conditions are the optimum for enhancing the methane yield of concentrated pig slurry.

3.3. Evaluation of kinetic parameters

The kinetic parameters obtained by fitting Eq. (2) to the experimental data on methane production are presented in Table 2. The results obtained concerning the maximum methane production (P) match with those previously discussed: increase for temperature and time increase, but decreasing trend for the longest times at temperatures above 170 °C. However, maximum methane production rates (R_m), seemed to differ slightly from this trend. The highest value of R_m was obtained for 150 °C, 60 min, and lower R_m values were obtained for higher temperatures. And, again, pre-

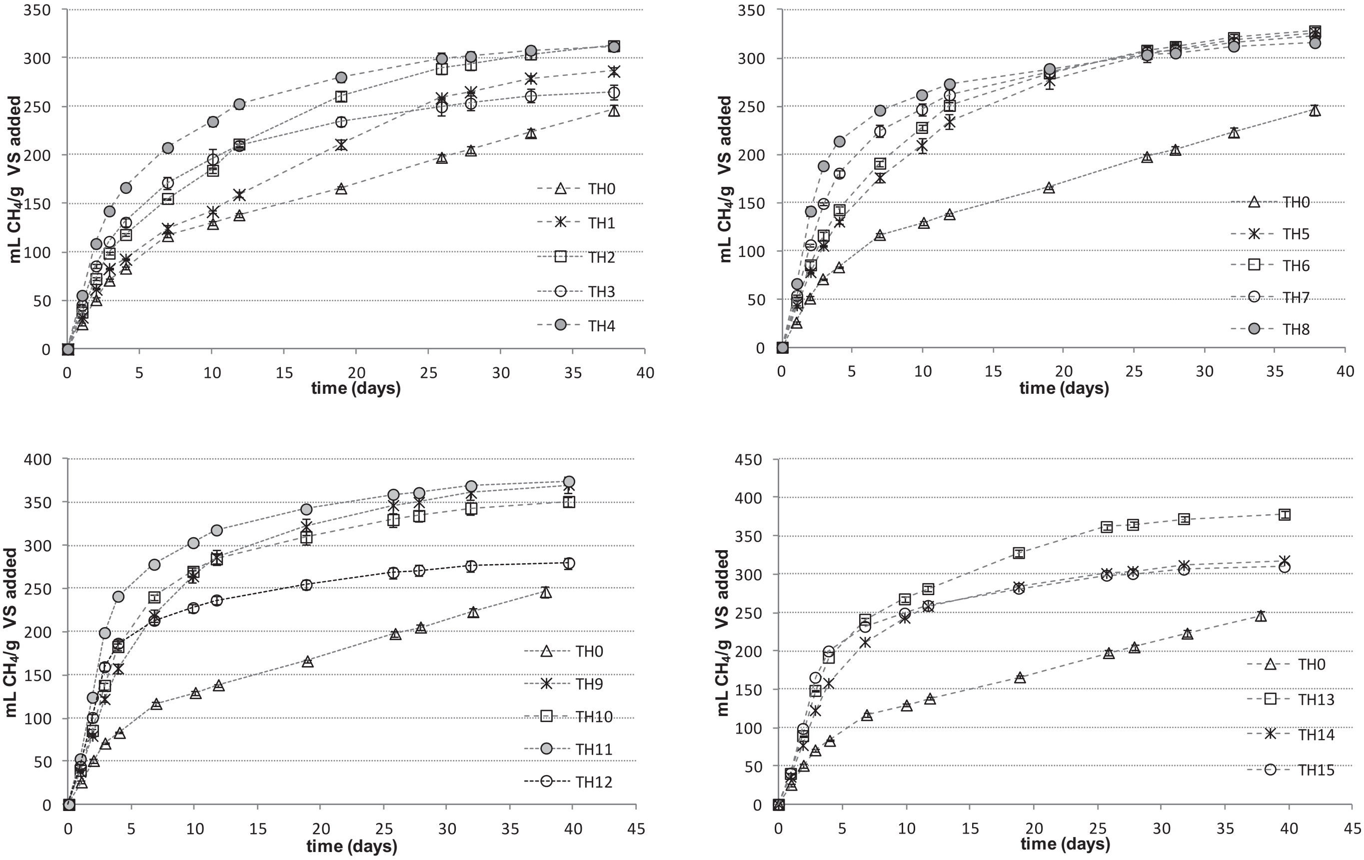

Fig. 1. Evolution of methane productivity (mL CH_4/g VS_{fed}) in the different BMP assays performed (TH0–TH15).

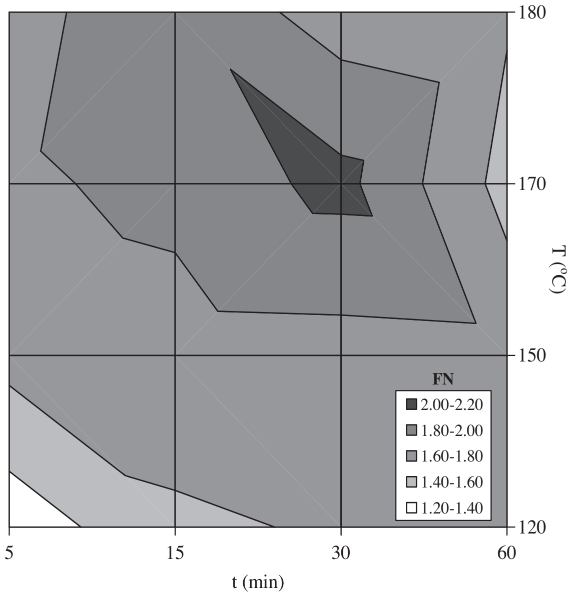

Fig. 2a. Methane productivity enhancement (presented as FN = (mL $CH_4)_{THi}$/(mL $CH_4)_{TH0}$) with respect to temperature and time of pretreatment. Darker areas correspond to higher FN values.

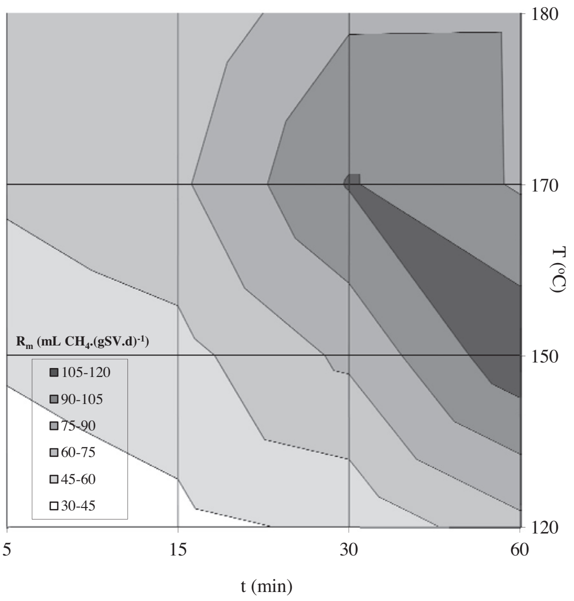

Fig. 2b. Methane production rates (R_m = mL CH_4/g vs. d)) with respect to temperature and time of pretreatment. Darker areas mean regions in which values of R_m are higher.

treatment times over 30 min for 170 °C and over 15 min for 180 °C caused a reduction in methane production rates.

Fig. 2b present the effect of temperature and time on R_m. Although similar to Fig. 2a with respect to the optimum (170 °C, 30 min), the trend for higher temperature–time values is different. These results suggest that inhibition of the maximum methane production is not necessarily coupled to lower degradation rates in the same levels. Even with the formation of refractory compounds due to high temperatures and pretreatment times, those conditions might still be able to improve rates, leading to reduced

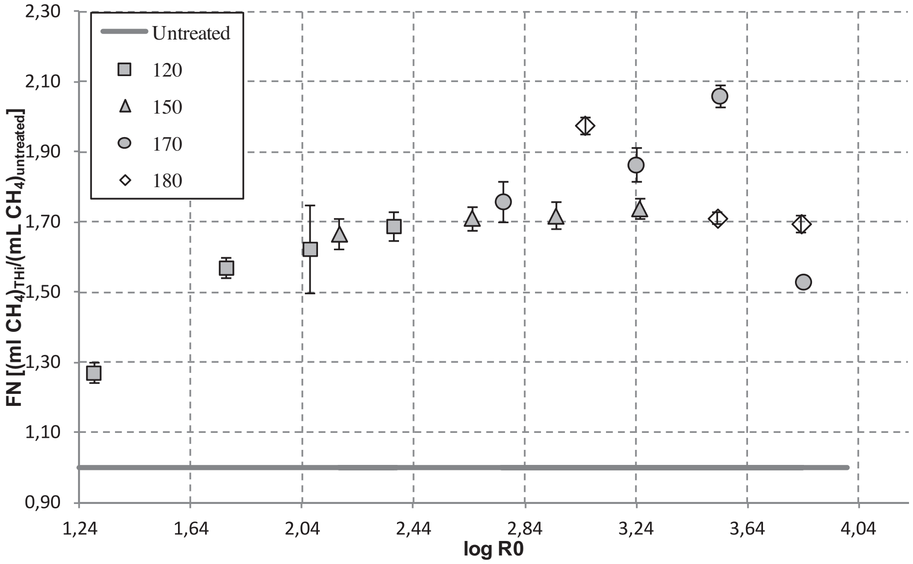

Fig. 3. Relationship between methane productivity enhancement and the severity factor (log R_0).

size of digesters as one possible advantage. However, a precise choice would depend on economic studies that take into account several variables and the process as a whole. A second explanation could be the degradation of organic matter for high intensity treatment, therefore decreasing the yield due to the lower organic matter available. However, no reduction in the values of COD was observed after the TH treatment, and therefore no experimental data support this hypothesis.

3.4. Evaluation of the severity factor

Fig. 3 presents the relationship between the severity factor and the normalized production of methane (FN). The influence of the severity factor on the response of the methane yield presents some differences when temperature and time change. For temperatures of 120 and 150 °C, it is clear by Fig. 3 that FN always increases with the severity factor. However, for the higher temperatures of 170 and 180 °C, there is a limit in the severity factor that, once surpassed, a reduction in FN occurred. According to Fig. 3 and Table 2, the optimum value for the severity factor would be 3.54 for a temperature of 170 °C. For a temperature of 180 °C, however, this severity factor led to lower values of FN. For higher values of the severity factor, inhibition in methane production was observed. This suggests that a severity factor of 3.54 could be considered an upper limit in which some instability in performance may be expected depending on operational conditions.

4. Conclusions

The influence of thermal pretreatment parameters (temperature and time) on the anaerobic biodegradation of the solid fraction of pig slurry was evaluated. All the conditions tested were better than the untreated condition, which means that the thermal pretreatment improved the anaerobic digestion of this type of waste, not only concerning biodegradability, but also degradation rates. Results suggested that temperature has a greater effect on methane yield than pretreatment time, and that the best combination of parameters would be 170 °C and 30 min, which was able to maximize methane production up to 200% when compared to untreated samples.

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References

American Public Health Association/American Water Works Association/Water Environment Federation (APHA/AWWA/WEF), 2005. Standard Methods for the Examination of Water and Wastewater, 21st ed. American Public Health Association/American Water Works Association/Water Environment Federation, Washington DC, USA.

Angelidaki, I., Alves, M., Bolzonella, D., Borzacconi, L., Campos, J.L., Guwy, A.J., Kalyuzhnyi, S., Jenicek, P., Van Lier, J.B., 2009. Defining the biomethane potential (BMP) of solid organic wastes and energy crops: a proposed protocol for batch assays. Water Sci. Technol. 59 (5), 927–934.

Ardic, I., Taner, F., 2005. Effects of thermal, chemical and thermochemical pretreatments to increase biogas production yield of chicken manure. Fresenius Environ. Bull. 14, 1–8.

Bonmatí, A., Flotats, X., Mateu, L., Campos, E., 2001. Study of thermal hydrolysis as a pretreatment to mesophilic anaerobic digestion of pig slurry. Water Sci. Technol. 44 (4), 109–116.

Bougrier, C., Delgenès, J.P., Carrère, H., 2008. Effects of thermal treatments on five different waste activated sludge samples solubilisation, physical properties and anaerobic digestion. Chem. Eng. J. 139, 236–244.

Bujoczek, G., Oleszkiewicz, J., Sparling, R., Cenkowskic, S., 2000. High Solid Anaerobic Digestion of Chicken Manure. J. Agric. Eng. Res. 76, 51–60.

Burton, C.H., Turner, C., 2003. Manure Management, Treatment Strategies for Sustainable Agriculture. Silsoe Research Institute, Wrest Park, Silsoe, Bedford, UK.

Carrère, H., Sialve, B., Bernet, N., 2009. Improving pig manure conversion into biogas by thermal and thermo-chemical pretreatments. Bioresour. Technol. 100, 3690–3694.

Chae, K.J., Jang, A., Yim, S.K., Kim, S., 2008. The effects of digestion temperature and temperature shock on the biogas yields from the mesophilic anaerobic digestion of swine manure. Bioresour. Technol. 99, 1–6.

Cuetos, M.J., Gómez, X., Otero, M., Morán, A., 2008. Anaerobic digestion of solid slaughterhouse waste (SHW) at laboratory scale: influence of co-digestion with the organic fraction of municipal solid waste (OFMSW). Biochem. Eng. J. 40, 99–106.

Fangueiro, D., Lopes, C., Surgy, S., Vasconcelos, E., 2012. Effect of the pig slurry separation techniques on the characteristics and potential availability on N to plants in the resulting liquid and solid fractions. Biosyst. Eng. 113, 187–194.

Fdz-Polanco, F., Velazquez, R., Perez-Elvira, S.I., Casas, C., del Barrio, D., Cantero, F.J., Fdz-Polanco, M., Rodriguez, P., Panizo, L., Serrat, J., Rouge, P., 2008. Continuous thermal hydrolysis and energy integration in sludge anaerobic digestion plants. Water Sci. Technol. 57 (8), 1221–1226.

Ferreira, L.C., Donoso-Bravo, A., Nilsen, P.J., Fdz-Polanco, F., Pérez-Elvira, S.I., 2013. Influence of thermal pretreatment on the biochemical methane potential of wheat straw. Bioresour. Technol. 143, 251–257.

Hansen, K.H., Angelidaki, I., Ahring, B.K., 1998. Anaerobic digestion of swine manure: inhibition by ammonia. Water Res. 32 (1), 5–12.

Hendriks, A.T.W.M., Zeeman, G., 2009. Pretreatments to enhance the digestibility of lignocellulosic biomass. Bioresour. Technol. 100, 10–18.

Krishania, M., Kumar, V., Vijay, V.K., Malik, A., 2013. Analysis of different techniques used for improvement of biomethanation process: a review. Fuel 106, 1–9.

Mata-Alvarez, J., Macé, S., Llabrés, P., 2000. Anaerobic digestion of organic solid wastes. An overview of research achievements and perspectives. Bioresour. Technol. 74 (1), 3–16.

Menardo, S., Balsari, P., Dinuccio, E., Gioelli, F., 2011. Thermal pre-treatment of solid fraction from mechanically-separated raw and digested slurry to increase methane yield. Bioresour. Technol. 102, 2026–2032.

Mladenovska, Z., Hartman, H., Kvist, T., Sales-Cruz, M., Gani, R., Ahring, B.K., 2006. Thermal pretreatment of the solid fraction of manure: impact on the biogas reactor performance and microbial community. Water Sci. Technol. 53 (8), 59–67.

Møller, H.B., Hansen, J.D., Sørensen, C.A.G., 2007. Nutrient recovery by solid-liquid separation and methane productivity of solids. Am. Soc. Agric. Biol. Eng. 50 (1), 193–200.

Moset, V., Cerisuelo, A., Sutaryo, S., Møller, H.B., 2012. Process performance of anaerobic co-digestion of raw and acidified pig slurry. Water Res. 46, 5019–5027.

Neves, L., Oliveira, R., Alves, M.M., 2004. Influence of inoculum activity on the bio-methanization of a kitchen waste under different waste/inoculum ratios. Process Biochem. 39, 2019–2024.

Nolan, T., Troy, S.M., Gilkinson, S., Frost, P., Xie, S., Zhan, X., Harrington, C., Healy, M.G., Lawlor, P.G., 2012. Economic analysis of pig manure treatment options in Ireland. Bioresour. Technol. 105, 15–23.

Pérez-Elvira, S.I., Fdz-Polanco, M., Fdz-Polanco, F., 2011. Enhancement of the conventional anaerobic digestion of sludge: comparison of four different strategies. Water Sci. Technol. 64 (2), 375–383.

PNIR 2007–2015, Plan Nacional Integrado de Residuos, Anexo 15. Documento Estratégico. Conclusiones y propuestas relativas a la gestión de los purines del grupo de trabajo interministerial para el análisis de las instalaciones de tratamiento y reducción de purines de explotaciones de porcino en las que se produce energía eléctrica.

Qiao, W., Yan, X., Ye, J., Sun, Y., Wang, W., Zhang, Z., 2011. Evaluation of biogas production from different biomass wastes with/without hydrothermal pretreatment. Renewable Energy 36, 3313–3318.

Rico, C., Rico, J.L., Tejero, I., Muñoz, N., Gómez, B., 2011. Anaerobic digestion of the liquid fraction of dairy manure in pilot plant for biogas production: residual methane yield of digestate. Waste Manage. (Oxford) 31 (9–10), 2167–2173.

Rico, C., Rico, J.L., García, H., García, P.A., 2012. Solid-liquid separation of dairy manure: distribution of components and methane production. Biomass Bioenergy 39, 370–377.

Pérez-Elvira, S.I., Fdz-Polanco, F., 2012. Continuous thermal hydrolysis and anaerobic digestion of sludge. Energy integration study. Water Sci. Technol. 65 (10), 1839–1846.

Schröder, J., 2005. Revisiting the agronomic benefits of manure: a correct assessment and exploitation of its fertilizer value spares the environment. Bioresour. Technol. 96, 253–261.

Sutaryo, S., Ward, A.J., Møller, H.B., 2013. Anaerobic digestion of acidified slurry fractions derived from different solid-liquid separation methods. Bioresour. Technol. 130, 495–501.

Wnetrzak, R., Kwapinski, W., Peters, K., Sommer, S.G., Jensen, L.S., Leahy, J.J., 2013. The influence of the pig manure separation system on the energy production potentials. Bioresour. Technol. 136, 502–508.

Xie, S., Wu, G., Lawlor, P.G., Frost, J.P., Zhan, X., 2012. Methane production from anaerobic co-digestion of the separated solid fraction of pig manure with dried grass silage. Bioresour. Technol. 104, 289–297.

Xu, J., Chen, Y., Cheng, J.J., Sharma-Shivappa, R.R., Burns, J.C., 2011. Delignification of switchgrass cultivars for bioethanol production. Bioresources 6 (1), 707–720.

Yoneyama, Y., Nishii, A., Nishimoto, M., Yamada, N., Suzuki, T., 2006. Upflow anaerobic sludge blanket (UASB) treatment of supernatant of cow manure by thermal pre-treatment. Water Sci. Technol. 54, 221–227.