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EFFECT OF ULTRASOUND PRE-TREATMENT IN THE ANAEROBIC CO-DIGESTION OF CATTLE MANURE WITH FOOD WASTE AND SLUDGE

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ABSTRACT

This paper present a study of the effect of applying ultrasound pre-treatment in the production of methane when co-digesting mixtures of cattle manure with food waste and sludge. A series of experiments were carried out under mesophilic and thermophilic conditions in continuously stirred-tank reactors containing 70% cattle manure, 20% food waste and 10% sewage sludge. Ultrasound pre-treatment allows operating at lower HRT, achieving higher volumetric methane yields: 0.85 L CH_4/L.day at 36ºC and 0.82 CH_4/L.day at 55ºC, when cattle manure and sewage sludge were sonicated. With respect to the non sonicated waste, these values represent increases of up to 31% and 67% for mesophilic and thermophilic digestion, respectively.

Keywords: ultrasound, anaerobic co-digestion, cattle manure, sewage sludge, food waste

1. INTRODUCTION

The use of agricultural material such as manure, slurry and other organic wastes for biogas production has significant environmental advantages in terms of heat and power production and its use as a biofuel. Biogas plants can contribute significantly to sustainable development in rural areas as well as providing farmers with new income opportunities (Directive 2009/28/EC). However, the low biogas yield of animal manure (Marañón et al., 2001, Castrillón et al., 2002, Amon et al., 2006) sometimes does not warrant the capital costs of farm-scale plants (Cavinato et al., 2010).

To enhance biogas production, pre-treatments (chemical, thermal, ultrasound, enzymatic) can be applied and/or the manure can be co-digested with other wastes to achieve synergetic effects that make the anaerobic digestion process profitable. By applying pre-treatments, it is possible to accelerate the hydrolysis of organic matter, thus increasing soluble chemical oxygen demand (COD) and, in many cases, also increasing methane yield. Pre-treatments such as alkaline or acid hydrolysis, enzymatic hydrolysis, thermal treatment or ultrasound may be applied, the last technique being considered one of the most versatile (Luste et al., 2012).

Until now, the ultrasound pre-treatment studies found in the literature have mainly focused on sewage sludge (Xie et al., 2009). Some authors, however, have applied ultrasonication to other substrates, such as dairy cattle slurry (Luste and Luostarinen, 2011) or dairy cattle slurry plus industrial meat processing by-products (Luste et al., 2012). Our research group applied ultrasound to dairy cattle slurry supplemented with raw glycerin (Castrillón et al., 2011; Castrillón et al., 2013a), achieving very good results. When adding 6% glycerin to cattle slurry and applying an specific energy of around 1,100 kJ/kg TS, a methane yield of 590 L/kg VS and a volumetric biogas production of 56.5 m^3/t wet waste (65% methane content) were obtained, operating in an induced bed reactor at 55ºC and at OLR of 6.4 kg COD/m^3.day (18 days HRT).

Different researchers have studied the co-digestion of manure with a wide variety of co-substrates such as fruit and vegetable wastes (Callaghan et al., 2002), the organic fraction of municipal solid waste (Capela et al., 2008), food waste (Neves et al., 2009; Banks et al., 2011) and raw glycerin (Astals et al., 2012; Castrillón et al., 2011; Castrillón et al., 2013b). Studying the co-digestion of cattle slurries with fruit and vegetable wastes (FVW) and with chicken manure, Callaghan et al. (2002), found that increasing the proportion of FVW from 20% to 50% improved the methane yield from 230 to 450 L CH_4/kg VS. Capela et al. (2008) evaluated the technical feasibility of anaerobic co-digestion with three types of organic solid waste under mesophilic conditions: the organic fraction of municipal solid waste (OFMSW), industrial sludge, and cattle manure. Increasing the OFMSW in the mixture generally resulted in higher methane production and volatile solids reduction. Banks et al. (2011) evaluated the feasibility of centralised pre-processing and pasteurisation of source-separated domestic food waste followed by transport to farms for anaerobic co-digestion with dairy cattle slurry. The results obtained showed that the addition of food waste improved energy yields per digester unit volume, with a corresponding increased potential for improving farm income by as much as 50%.

Biogas production potential of unscreened dairy manure and different mixtures of unscreened dairy manure and food waste using batch digesters at 35ºC were studied by El-Mashad and Zhang (2010). The methane yield of unscreened manure and two mixtures of unscreened manure and food waste (68/32 and 52/48), after 30 days of digestion, were 241, 282 and 311 L/kgVS, respectively.

In line with our previous study (Marañón et al., 2012), the goal of the present research work was to evaluate the effect of applying ultrasound pre-treatment on the co-digestion of dairy cattle manure with food waste and sewage sludge. A series of experiments were carried out under mesophilic and thermophilic conditions using continuously stirred-tank reactors.

2. EXPERIMENTAL STUDY

2.1. Materials

Mixtures of dairy cattle manure (CM), food waste (FW) and sewage sludge (SS) were used as co-substrates for anaerobic digestion.

The cattle manure was collected in 20 L plastic bottles from the cesspit of a dairy farm with 120 livestock units (LU), after agitation of the cesspit contents, then ground and stored at 4ºC (for no more than three weeks).

The food waste came from an old age residence, collected separately in 10 L plastic bags. The food waste was mixed, ground and subsequently frozen to -20ºC in plastic containers for storage in the laboratory.

The sewage sludge employed was a mixed of co-settled primary and secondary sludge, after dehydrated using a filter press, from a wastewater treatment plant designed for a population equivalent of 85,000. After collection, the sludge was frozen for storage at -20ºC in plastic containers.

The inocula used were mesophilic and thermophilic digestates from CSTR co-digesting mixtures of cattle manure, food waste and sewage sludge (Marañón et al., 2012). The digestate was allowed to stand a minimum of two days before being mixed with the substrates to ensure degasification.

2.2. Equipment

The manure was grounded using a domestic triturator and the food waste, using a STR-

2000 triturator.

The ultrasonic equipment used in this study was a Hielsher UPS 400S (power: 400 W, frequency: 24 kHz).

Digestions were performed in 5 L jacketed CSTR, made of glass and provided with automatic temperature control, and filled up to a volume of 3.75 L.
The biogas volume was measured daily using Bronkhorst Hi-Tech F-101D mass flow meters and the composition was checked every two days in an Agilent 7890 gas chromatograph.

2.3. Analytical methods

Samples from the reactors (digestates) were taken twice a week to analysing their composition. Chemical oxygen demand was determined following Method 5220 D (closed reflux, colorimetric method) of the Standard Methods for the Examination of Water and Wastewater (APHA, 1998) using a Perkin Elmer Lambda 35 Visible-UV system.
Total nitrogen and total phosphorus were determined by ion chromatography (861 Advanced Compact IC 2.861.0010) after transformation into nitrates and phosphates, respectively, by digestion under pressure with H_2O_2 and HCOOH in a microwave oven (Milestone Ethos 1 Advanced Microwave Digestion Labstation).
The methane and carbon dioxide content of the biogas was measured on an Agilent gas chromatograph using a TCD detector and a Porapack N packed column plus a molecular sieve, employing the following temperature ramp: starting temperature 35ºC (1.5 minutes) increasing up to 55ºC at a rate of 1.5ºC/minute.
Volatile acidity and total alkalinity were measured by volumetric analysis with H_2SO_4 and 0.1 N NaOH, using the method specified in Degremont (1979).

2.4. Experimental procedure

A series of experiments using CSTR containing 70% of CM, 20% of FW and 10% of SS (*721*) by weight, with and without pre-treatment by ultrasound, were carry out. The experimental set up is shown in Figure 1. The use of this mixture was based on results from previous research work (Marañón et al., 2012).

Mixtures were prepared daily from the stored wastes, maintaining the weight ratio of each residue in the mixture and completing the final volume with fresh water to obtain a total solid content of around 4%.

Ultrasound was applied to pre-treat only CM or CM and SS, but not to FW, due to the higher biodegradability of this waste. The specific energy used for sonication was 7,500 kJ/kg TS. The time required for sonication was calculated as a function of the volume of feed and total solids (Castrillón et al., 2011).

The experiments were carried out at mesophilic (36±1ºC) and thermophilic (55±1ºC) temperatures. Different hydraulic residence times (HRT) for each mixture were studied. The most important parameters were monitored throughout this period of continuous operation: Chemical oxygen demand (COD), total alkalinity (TAC), volatile acidity (VA), total solids (TS), volatile solids (VS) and biogas production. The biogas and methane yields are expressed at standard conditions of temperature and pressure (IUPAC).

3. RESULTS AND DISCUSSION

Table 1 shows the composition of the feed mixtures and of the final digestates from the different experiments. As can be seen, there is a wide variation in the different parameters determined, which is due to the variability in the composition of the samples of the different substrates taken along the period of the experiments. The feed mixtures presented high volatile acidity and alkalinity. C/N ratio oscillated between values of 16-18. Most of the researchers recommend an operating C/N ratio ranging from 20 to 30 for anaerobic bacterial growth in anaerobic digestion systems (Li et al., 2011), but the optimal C/N ratio varies with the type of feedstock to be digested. For example, Romano and Zhang (2008) recommended the C/N ratio to be maintained at 15 for the co-digestion of onion juice and digested sludge. When corn stover was inoculated with digested sewage sludge, the digestion process worked well with a C/N of 15 to 18 but failed with a C/N of 21 or higher (Zhu and Li, 2009).

With respect to the digestate, total alkalinity is high, however considerably lower than the initial mixture (Table 1). The volatile acidity values are below 0.5 g/kg, indicating that the reactors are stable. As a consequence of this, the pH from digestates was slightly basic (between 7.7 and 8.4).

Figure 2 shows the efficiency in the removal of volatile solids for the different operating conditions analysed, ranging between 49% and 62.5%. As can be seen, under mesophilic conditions, the removal was higher when operating at a HRT of 20 days, increasing when applying sonication prior to the digestion (59% without applying sonication, 62% when cattle manure was sonicated and 62.5% when both, cattle manure and sewage sludge, were previously sonicated).

Under thermophilic conditions, the volatile solid removal was, in general, slightly lower, ranging between 49% and 60.5%. Without previous sonication, the removal was, like under mesophilic conditions, higher operating at a HRT of 20 days (58%), but when applying sonication to cattle manure the maximum removal (60.5%) was found at shorter HRT (18 days) or even shorter when applying sonication to both, cattle manure and sewage sludge (59.8% for 14 days; 59.3% for 16 days).

Figures 3 to 6 show the evolution in specific and volumetric methane production obtained in the co-digestion of the mixture of 70% CM, 20% FW and 10% SS at 36ºC and 55ºC, with or without sonication, for the different HRT. Table 2 shows mean values of the specific methane production (SMP) and the volumetric methane production (VMP) depending on the operating conditions.

With respect to the SMP, the best results were obtained for the higher HRT (22 days) operating at 36ºC (603 L/kg VS) without sonication, decreasing when lowering the HRT. For the other cases studied, with previous sonication, SMP were lower, ranging between values of 329-463 L/kg VS.

But regarding the energy yields per digester unit volume, better results were found when applying sonication. The maximum value (0.85 L CH_4/L.day) was found for 14 days HRT at 36ºC when both wastes, cattle manure and sewage sludge, were sonicated. This value represents an increase of 15% or 31% with respect to the non sonicated waste depending on the HRT (20 or 18 days, respectively). Lower HRT when co-digesting the non sonicated waste led to instability of the reactor. Operating at 55ºC, higher increases were found when applying sonication. A maximum value of 0.82 L CH_4/L.day was obtained for 16 days HRT when applying sonication to cattle manure and sludge, representing an increase of 41% or 67% with respect to the non sonicated waste for HRT of 20 days or 18 days, respectively.

The differences found may be perfectly reasonable, different temperatures produce different reactor conditions. In this sense, Gannoun et al. (2007) examined the anaerobic digestion of combined olive mill and abattoir waste water at 37ºC and 55ºC, finding that the thermophilic reactor produced higher COD removal and biogas yield than the mesophilic reactor. On the other hand, experiments with proteinaceous wastewater using UASB laboratory scale reactors led to 84% COD removal when operating at 37ºC versus 69-83% COD removals at 55ºC (Fang and Chung, 1999). Independently of the biogas yield and organic matter removal, it is well known that mesophilic anaerobic digestion is more stable than thermophilic digestion, the inhibition phenomena due to the presence of ammonium and volatile acids being more unusual in mesophilic digestion (Fernández et al., 2008; Khalid et al., 2001).

Ammonium nitrogen was determined through the process, as a potential inhibitor in the anaerobic digestion process. Ammonium nitrogen in digestate oscillated between 630-970 mgN-NH_4^+/L at 55ºC and 500-1250 mg NH_4^+-N/L at 36ºC. When calculating the corresponding unionized ammonia (NH_3) concentration (Hansen et al., 1998) for 55ºC and pH 8.2, the maximum value obtained in the digestate was 368 mg NH_3-N/L. This value is quite low and it is not likely to lead to inhibition phenomena in the reactors. Experiments carried out by Hansen et al. (1998), when digesting swine manure demonstrated that a free ammonia concentration of 1.1g NH_3-N/L was needed to inhibit the process.

4. CONCLUSIONS

Application of ultrasound pre-treatment allows operating at shorter retention times in the anaerobic reactors. Considering the energy yields per digester unit volume, better results were found when applying sonication. The maximum value (0.85 L CH_4/L.day) was found for 14 days HRT at 36ºC, representing an increase of 31% with respect to the non sonicated waste (0.74 L CH_4/L.day) for 18 days HRT. Higher increases were found at 55ºC, a maximum value of 0.82 L CH_4/L.day was found for 16 days HRT, representing an increase of 67% with respect to the non sonicated waste (0.49 CH_4/L.day) for 18 days HRT.

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Caption for Tables

Table 1. Composition of feed mixtures and digestates obtained under the different operating conditions (all results in g/kg, with the exception of pH)

Table 2. Biogas and methane productions in the co-digestion of cattle manure, food waste and sludge (70/20/10) under the different operating conditions

Caption for Figures

Figure 1. Experimental set up for the co-digestion in continuously stirred tank reactor

Figure 2. Removal of volatile solids in the different co-digestion experiments.

Figure 3. Evolution of the specific and volumetric methane production in the mesophilic anaerobic co-digestion of mixtures of cattle manure, food waste and sewage sludge, for different operating conditions, with and without previous sonication of the cattle manure.

Figure 4. Evolution of the specific and volumetric methane production in the mesophilic anaerobic co-digestion of mixtures of cattle manure, food waste and sewage sludge, for different operating conditions, with and without previous sonication of the cattle manure and sewage sludge.

Figure 5. Evolution of the specific and volumetric methane production in the thermophilic anaerobic co-digestion of mixtures of cattle manure, food waste and sewage sludge, for different operating conditions, with and without previous sonication of the cattle manure.

Figure 6. Evolution of the specific and volumetric methane production in the thermophilic anaerobic co-digestion of mixtures of cattle manure, food waste and sewage sludge, for different operating conditions, with and without previous sonication of the cattle manure and sewage sludge.

Table 1. Composition of feed mixtures and digestates obtained under the different operating conditions (all results in g/kg, with the exception of pH)

	TS	VS	COD	pH	TAC	VA
			Feed mixture			
	30.5-49.5	11.8-38.4	31.2-58.8	6.5-7.9	7.6-8.8	2.5-3.7
HRT (days)			*Digestate*			
Mixture 721M (without sonication, mesophilic digestion)						
22	20.5-23.8	10.9-12.5	16.5-18.7	8.0-8.2	5.3-5.4	0.17-0.29
20	19.2-29.4	10.1-12.5	13.9-21.7	8.0-8.2	4.1-5.9	0.17-0.34
18	15.1-31.2	8.2-12.1	14.9-17.2	7.9-8.2	4.6-5.1	0.19-0.24
Mixture S721M (cattle manure sonicated, mesophilic digestion)						
22	15.1-17.8	8.6-10.1	16.2-22.8	8.2-8.3	3.0-3.7	0.15-0.19
20	20.9-24.7	10.7-14.2	17.5-20.8	7.9-8.1	2.6-3.0	0.17-0.32
18	21.0-23.1	10.6-11.8	20.7-25.8	8.1-8.2	2.1-2.4	0.17-0.20
16	25.7-26.6	11.6-12.1	14.7-20.8	7.8-7.9	2.5-3.1	0.14-0.17
14	20.1-24.3	11.5-12.4	13.9-14.0	7.9-8.1	2.6-2.9	0.17-0.20
Mixture S72S1M (cattle manure and sludge sonicated, mesophilic digestion)						
22	21.5-22.1	10.8-13.7	16.6-19.5	8.2-8.3	3.4-3.8	0.17-0.22
20	23.8-25.2	10.3-11.8	14.2-17.6	7.7-8.1	2.4-3.1	0.07-0.16
18	20.9-23.9	10.6-12.5	15.4-18.8	7.7-7.9	2.5-2.8	0.13-0.14
16	21.5-23.4	11.0-13.9	11.4-17.8	7.7-8.0	2.7-3.3	0.12-0.13
14	15.8-22.8	10.5-12.8	14.7-16.4	7.7-7.9	3.2-3.9	0.17-0.19
Mixture 721T (without sonication, thermophilic digestion)						
22	20.9-27.6	11.6-12.9	17.5-19.5	8.3-8.5	4.7-6.0	0.46-0.82
20	17.1-25.4	10.0-12.7	15.2-22.4	8.2-8.4	3.9-6.1	0.31-0.58
18	17.9-27.2	9.7-13.3	16.7-30.8	8.2-8.3	4.2-4.5	0.29-0.41
Mixture S721T (cattle manure sonicated, thermophilic digestion)						
22	19.0-21.3	11.0-11.1	17.5-22.7	8.2-8.4	3.1-3.4	0.20-0.43
20	28.8-32.5	10.3-13.8	15.0-23.5	8.1-8.4	2.2-3.6	0.18-0.30
18	24.2-30.9	10.3-13.8	15.0-23.5	8.0-8.3	2.2-3.6	0.18-0.30
16	20.0-21.6	9.5-11.2	12.5-18.7	7.9-8.3	2.4-3.6	0.23-0.30
14	29.2-33.9	10.8-22.6	13.4-16.6	8.1-8.3	3.0-3.9	0.24-0.40
Mixture S72S1T (cattle manure and sludge sonicated, thermophilic digestion)						
22	19.8-23.6	10.4-11.6	17.6-20.1	8.2-8.3	3.7-4.1	0.29-0.37
20	26.8-34.7	12.0-18.1	19.3-21.8	8.0-8.3	2.2-3.1	0.32-0.47
18	22.3-34.8	12.5-18.0	17.5-21.8	8.1-8.3	2.3-3.1	0.25-0.47
16	21.1-23.2	12.1-12.7	16.6-18.5	8.0-8.2	2.5-2.8	0.18-0.31
14	16.6-17.5	10.0-10.1	14.2-18.5	8.1-8.2	3.0-3.2	0.30-0.35

Table 2. Biogas and methane productions in the co-digestion of cattle manure, food waste and sludge (70/20/10) under the different operating conditions

HRT (days)	SMP ($L\ CH_4$/kg VS)	VMP ($L\ CH_4$/L.day)
Mixture 721M (non sonicated, mesophilic)		
22	603	0.74
20	546	0.74
18	431	0.65
Mixture S721M (cattle manure sonicated, mesophilic)		
22	376	0.42
20	390	0.50
18	424	0.62
16	452	0.83
14	427	0.80
Mixture S72S1M (cattle manure and sludge sonicated, mesophilic)		
22	428	0.56
20	460	0.60
18	407	0.65
16	450	0.73
14	432	0.85
Mixture 721T (non sonicated, thermophilic)		
22	424	0.53
20	423	0.58
18	329	0.49
Mixture S721T (cattle manure sonicated, thermophilic)		
22	418	0.53
20	392	0.58
18	463	0.75
16	378	0.70
14	355	0.65
Mixture S72S1T (cattle manure and sludge sonicated, thermophilic)		
22	350	0.46
20	380	0.48
18	398	0.65
16	467	0.82
14	375	0.75

Figure 1. Experimental set up for the co-digestion in continuously stirred tank reactor

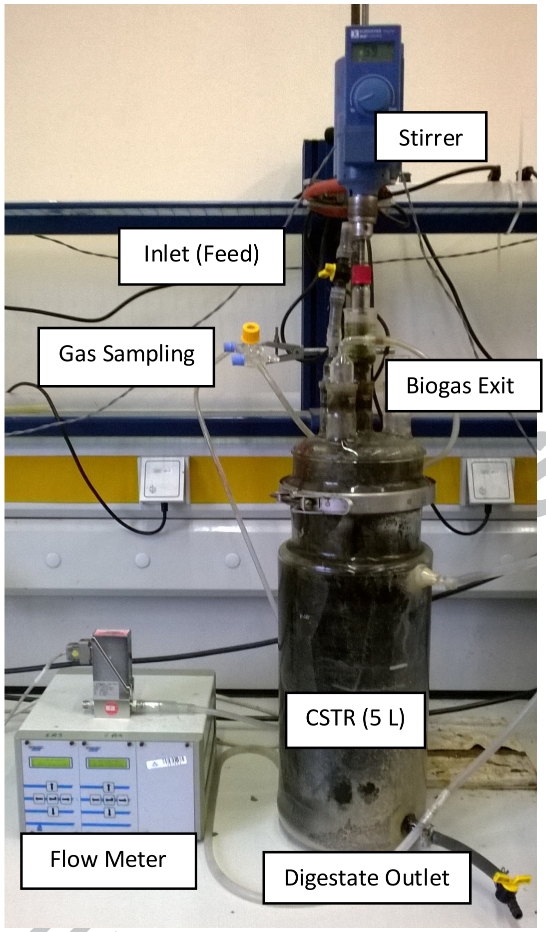


Figure 2. Removal of volatile solids in the different co-digestion experiments

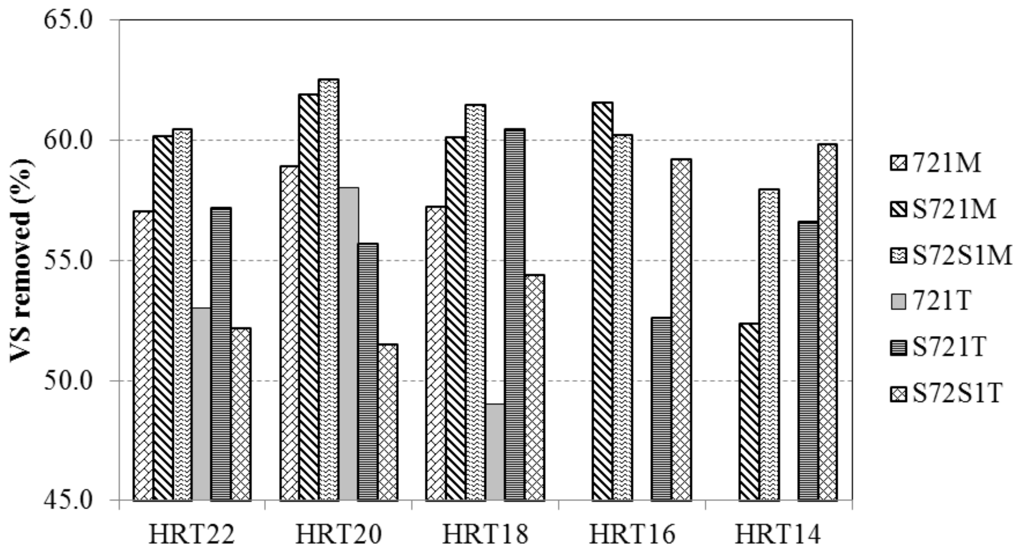


Figure 3. Evolution of the specific and volumetric methane production in the mesophilic anaerobic co-digestion of mixtures of cattle manure, food waste and sewage sludge, for different operating conditions, with and without previous sonication of the cattle manure (▲ $SMP_{sonicated}$ + $SMP_{not\ sonicated}$ ◆ $VMP_{sonicated}$ □ VMP_{not} sonicated)

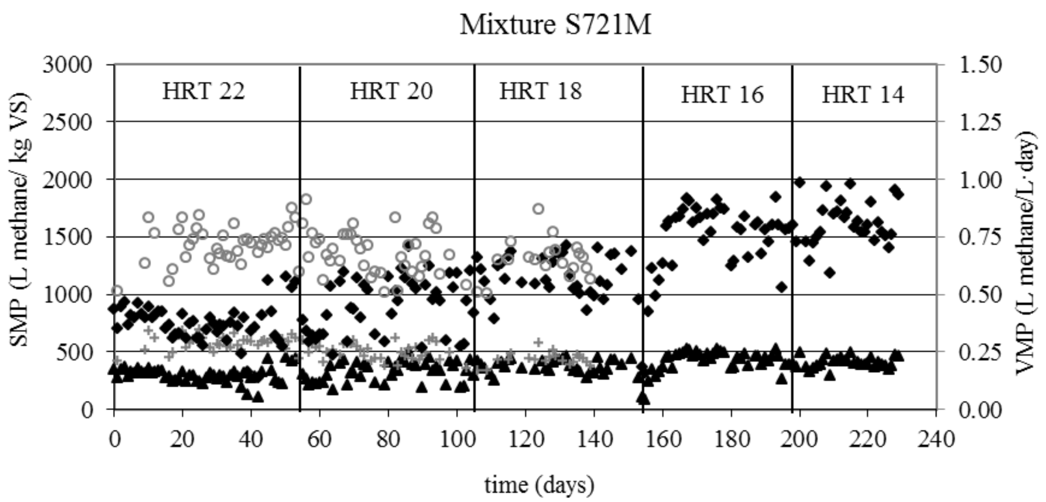


Figure 4. Evolution of the specific and volumetric methane production in the mesophilic anaerobic co-digestion of mixtures of cattle manure, food waste and sewage sludge, for different operating conditions, with and without previous sonication of the cattle manure and sewage sludge (▲$SMP_{sonicated}$ + $SMP_{not\ sonicated}$ ◆ $VMP_{sonicated}$ □ $VMP_{not\ sonicated}$)

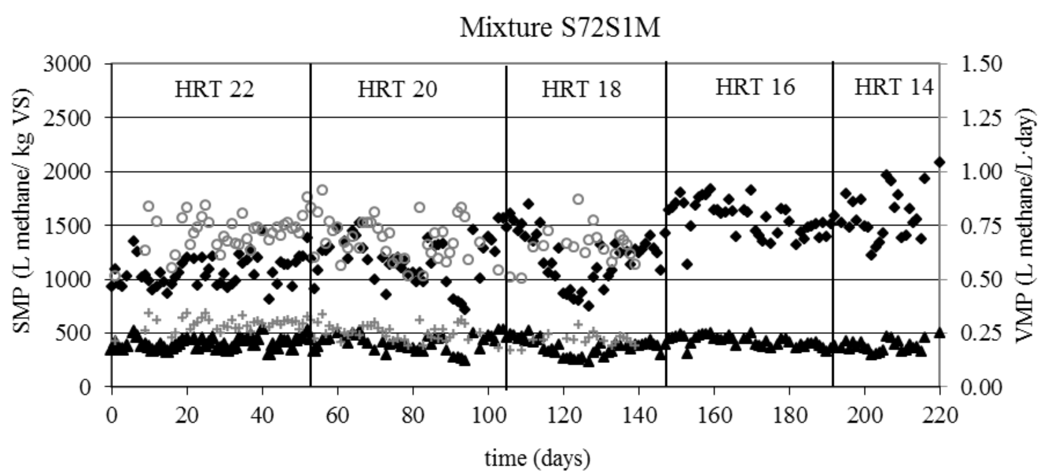


Figure 5. Evolution of the specific and volumetric methane production in the thermophilic anaerobic co-digestion of mixtures of cattle manure, food waste and sewage sludge, for different operating conditions, with and without previous sonication of the cattle manure (▲$SMP_{sonicated}$ + $SMP_{not\ sonicated}$ ◆ $VMP_{sonicated}$ ▯ $VMP_{not\ sonicated}$)

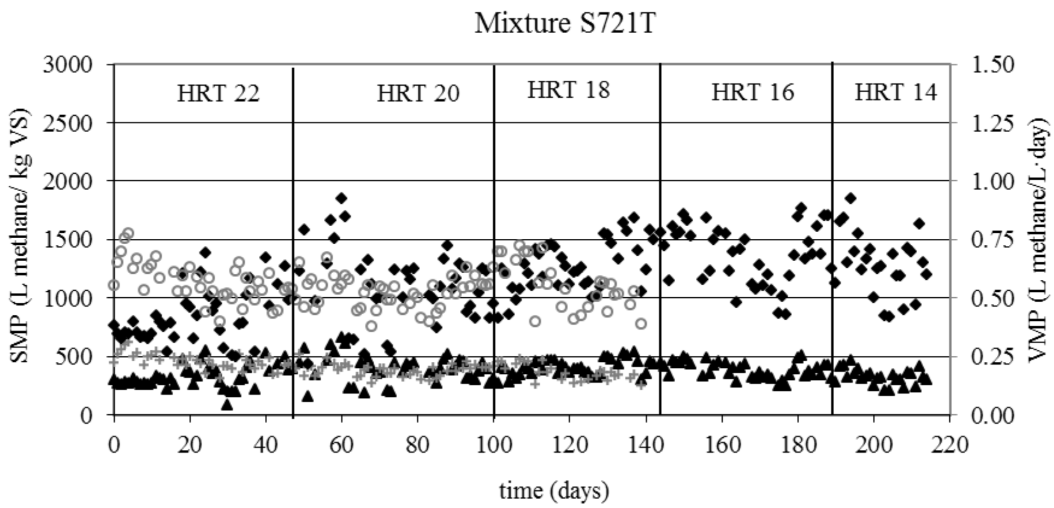
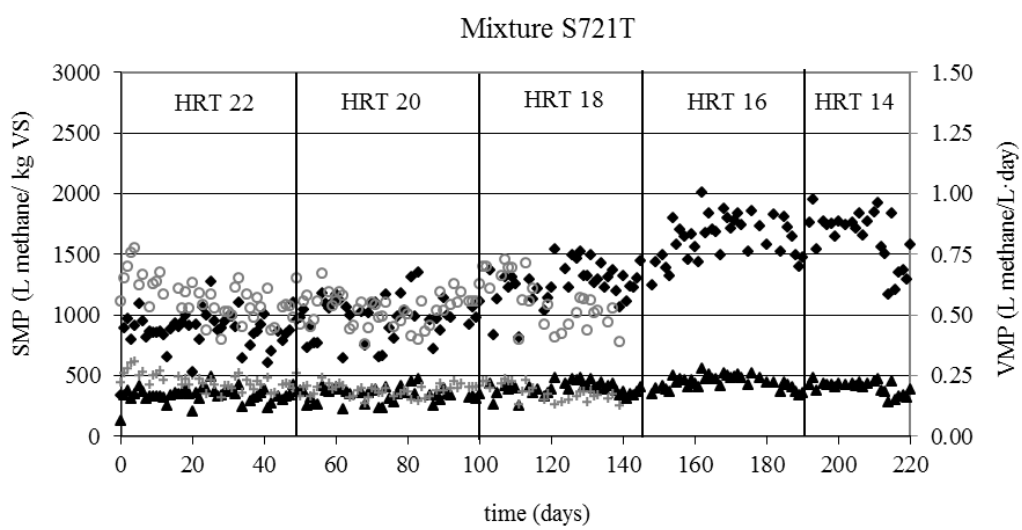


Figure 6. Evolution of the specific and volumetric methane production in the thermophilic anaerobic co-digestion of mixtures of cattle manure, food waste and sewage sludge, for different operating conditions, with and without previous sonication of the cattle manure and sewage sludge (▲$SMP_{sonicated}$ + $SMP_{not\ sonicated}$ ◆ $VMP_{sonicated}$ ▯ VMP_{not} sonicated)



Highlights

We studied the effect of ultrasound on methane yield in the co-digestion of waste
Ultrasound pre-treatment allow higher energy yields per digester volume
Up to 67% increase in methane yield at 55ºC and 31% at 37ºC were found