

The use of co-digested solid fraction as feedstock for biogas plants

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Abstract

A comparative study was set up in order to assess the technical feasibility of the long-term reuse of the mechanically separated co-digested solid fraction as a feedstock for anaerobic digestion plants (ADP). The biogas yields of two feedstock mixtures (A and B) were assessed in mesophilic conditions ($40\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$) using 8 lab-scale continuous stirred-tank reactors (CSRT). Feedstock mixture A (control) consisted of pig slurry (70%), farmyard manure (4%), sorghum silage (12%) and maize silage (14%). Feedstock mixture B was the same as the control plus the solid fraction derived from the mechanical separation of the output raw co-digestate collected on daily basis from the reactors. All reactors were fed simultaneously, three times a week, over a period of nine months. According to the study results, the reuse of the co-digested solid fraction as feedstock for ADP could increase the methane yield by approximately 4%. However, ADP efficiency evaluation (e.g., daily yield of methane per m³ of digester) suggest to limit this practice to a maximum time period of 120 days.

Introduction

Anaerobic digestion of organic substrates for the production and transformation of biogas into electric and thermal energy is experiencing a period of strong growth in Italy. According to a recent survey (Fabbrì et al., 2013), approximately 1000 agricultural anaerobic digestion plants (ADP) are currently running on the national territory with an installed electrical capacity of 756 MW. These ADP are generally installed at livestock farms and are mostly fed with animal manure, energy crops and agricultural by-products. Co-digestate is one of the final products of ADP. It contains mostly water, undigested organic matter and readily available inorganic compounds (e.g., nitrogen, phosphorus, potash) to crops. Due to the construction of the ADP often inside intensive livestock production

units with insufficient arable land for nutrient recycling, export of nutrients to outside farm areas may be necessary to avoid excess load of nutrients, with special regards to nitrogen (N). For such a reason, in many Italian anaerobic digestion plants, co-digestate is mechanically separated in order to obtain a liquid and a solid fraction (Dinuccio et al., 2010). In the liquid phase the greater amount of potassium and inorganic nitrogen is concentrated, whereas the solid fraction mainly contains organic compounds and phosphorus (Dinuccio et al., 2010). The liquid fraction is generally land applied near the ADP while the solid fraction is exported to outside farm areas or sold to other farmers. Nevertheless, the co-digested solid fraction can still contain a high biogas and methane (CH₄) potential (Balsari et al., 2010), due to the presence of residual and undigested volatile solids (VS). Thus, it can be reused as ADP feedstock. Balsari et al. (2010), in a work carried out at a national level through batch trials, found specific CH₄ yields of co-digested solid fraction ranging between 71.4 and 156.9 NL/kgVS. According to these figures they estimated that the reuse of the mechanically separated co-digested solid fraction into the digester has the potential to improve the total CH₄ production of the ADP by between 4% and 8%, depending on ADP operating parameters (e.g., feedstock type and quality, organic loading rate - OLR, hydraulic retention time - HRT) and the type of separator (e.g., screw press, one stage rotating separator) used to separate the raw co-digested slurry. Moreover, utilizing the co-digested solid fraction in this manner could reduce greenhouse gases (GHG) and ammonia (NH₃) normally released (Dinuccio et al., 2013) during its storage. However, specific studies assessing the applicability of such an option in a continuous fed anaerobic digestion system are lacking. This paper presents the results of a laboratory scale experiment carried out with the objective to assess the technical feasibility of the long-term reuse of the mechanically separated co-digested solid fraction as a feedstock for ADP.

Material and methods

Biomasses collection and characterization

Fresh samples of pig slurry, farmyard manure, sorghum silage and maize silage were collected at a selected full scale ADP (Table 1) operating in the Piemonte region (north western Italy), on the first working day of each month for the duration of the experimental period (270 days). Collected fresh biomass samples were then stored at 5°C for a period of 30 days and used to the anaerobic digestion tests. All biomasses were analysed for pH, total solids (TS), VS, total nitrogen (TN), total ammoniacal nitrogen (TAN), hemicelluloses (HC), celluloses (CE) and lignin (ADL). The pH was measured by a portable pH meter (Hanna Instruments HI 9026) using a glass electrode combined with a thermal automatic compensation system. TS were determined after 24 h at 105 °C. VS were determined according to AOAC (2000), after incineration in a muffle furnace at 550 °C for 4 hours. TN was analysed by the Kjeldahl standard method, after acidification with H₂SO₄ and mineralisation of the sample; TAN was analysed in accordance with AOAC (2000). HC, CE and ADL were determined by the Van Soest methods (Van Soest et al., 1991).

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Figure 1. The lab-scale continuous fed stirred-tank reactors (CSTR) used for the trial

Experimental setup

The biogas yields of two different feedstock mixtures were compared:

- mixture A (control – the same of the selected full scale ADP, Table 1): pig slurry (70%) farmyard manure (4%), sorghum silage (12%), maize silage (14%) - mixture B: the same mixture as the control plus all (100%) the solid fraction obtained after mechanical separation of the output co-digestate collected on daily basis from the digester.

The experiment was carried out under mesophilic conditions ($40 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$), within a temperature-controlled chamber, by using 8 identical lab-scale continuous fed stirred-tank reactors (CSTR). Each reactor (Figure 1), cylindrical in shape, is made up of plexiglass, with a total volume of 6.5L. The biomass within the reactor is continuously mixed at a constant rate of about 4 rpm by a vertical mixer connected to a geared motor installed on the top of reactor. The reactors are equipped with inlet and outlet ports for feeding and effluent discharge. A pipe situated at the top of the reactors is connected to Tedlar® gas bags by means of Tygon® tubing to collect the produced biogas. The experi-

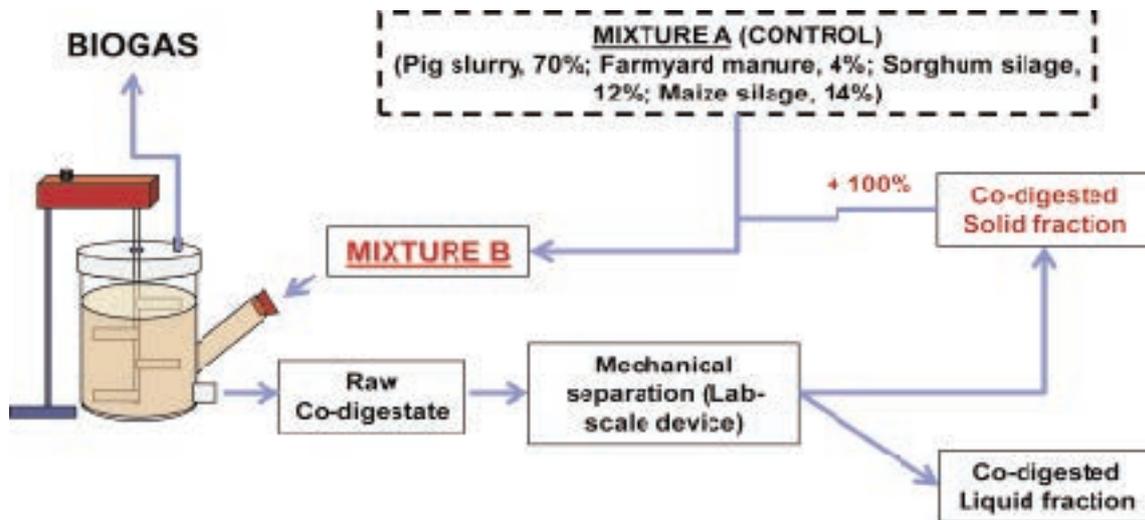


Figure 2. Feeding scheme of the reactors R4-R6.

Table 1: Main operating parameters of the selected anaerobic digestion plant

Feedstock composition	Installed electric power (kWhel)	Temperature (°C)	OLR (kgVS m ⁻³ digester day ⁻¹)	HRT (days)
Pig slurry (70%) Farmyard manure (4%) Sorghum silage (12%) Maize silage (14%)	500	~ 40	~ 2.20	~ 40

Table 2. Main chemical and physical characteristics of the fresh biomasses (standard deviation in parentheses, n=3) used in the trial

	Maize silage	Sorghum silage	Farmyard manure	Pig slurry
PpH	3.75 (0.22)	3.96 (0.19)	8.49 (0.17)	7.26 (0.25)
TS (%)	30.5 (2.96)	27.6 (2.46)	22.3 (2.37)	1.13 (0.52)
VS (%TS)	95.5 (0.96)	91.2 (1.05)	78.7 (4.01)	67.6 (4.38)
TN (%)	0.34 (0.10)	0.37 (0.09)	0.48 (0.09)	0.15 (0.07)
TAN (%)	0.03 (0.02)	0.03 (0.01)	0.38 (0.06)	0.11 (0.04)
HC (%)	7.82 (0.74)	6.24 (0.12)	4.20 (0.94)	n.d.
CE (%)	8.16 (0.63)	9.15 (0.72)	7.01 (0.41)	n.d.
ADL (%)	0.99	1.41	2.67	n.d.

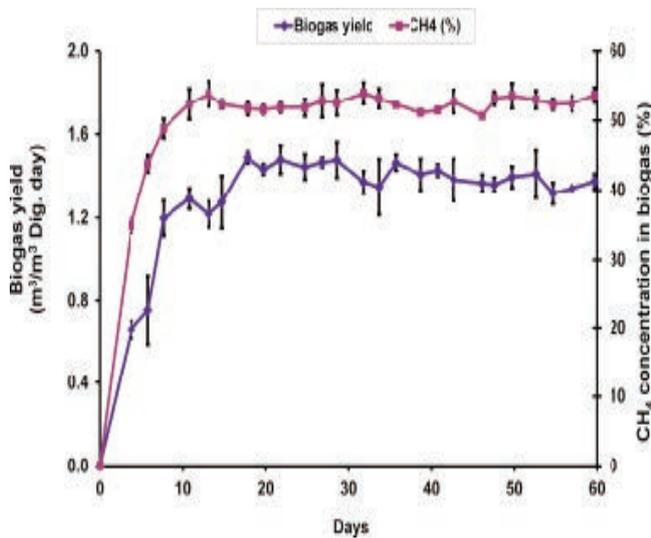


Figure 3. Specific biogas yield and methane concentration recorded from reactors R1-R8 320 during the startup phase (days 0 – 60). Error bars indicate standard deviation (N = 8).

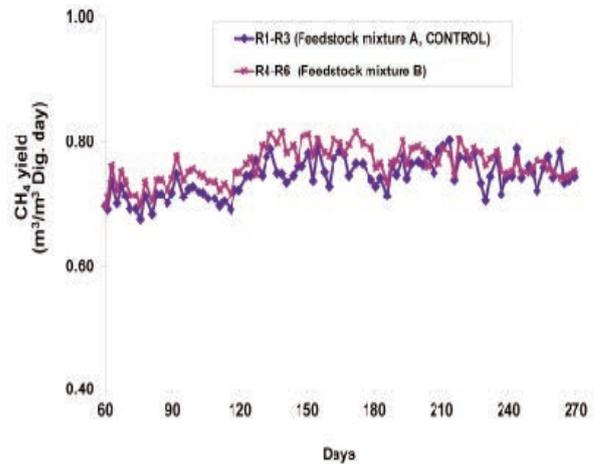


Figure 4. Average volumetric methane yields recorded from day 60 (end of the startup 322 phase) to day 270 (end of the trial) from reactors R1-R3 (feedstock mixture A, control) and 323 from reactors R4-R6 (feedstock mixture B). N = 3; standard deviation removed for clarity. 324

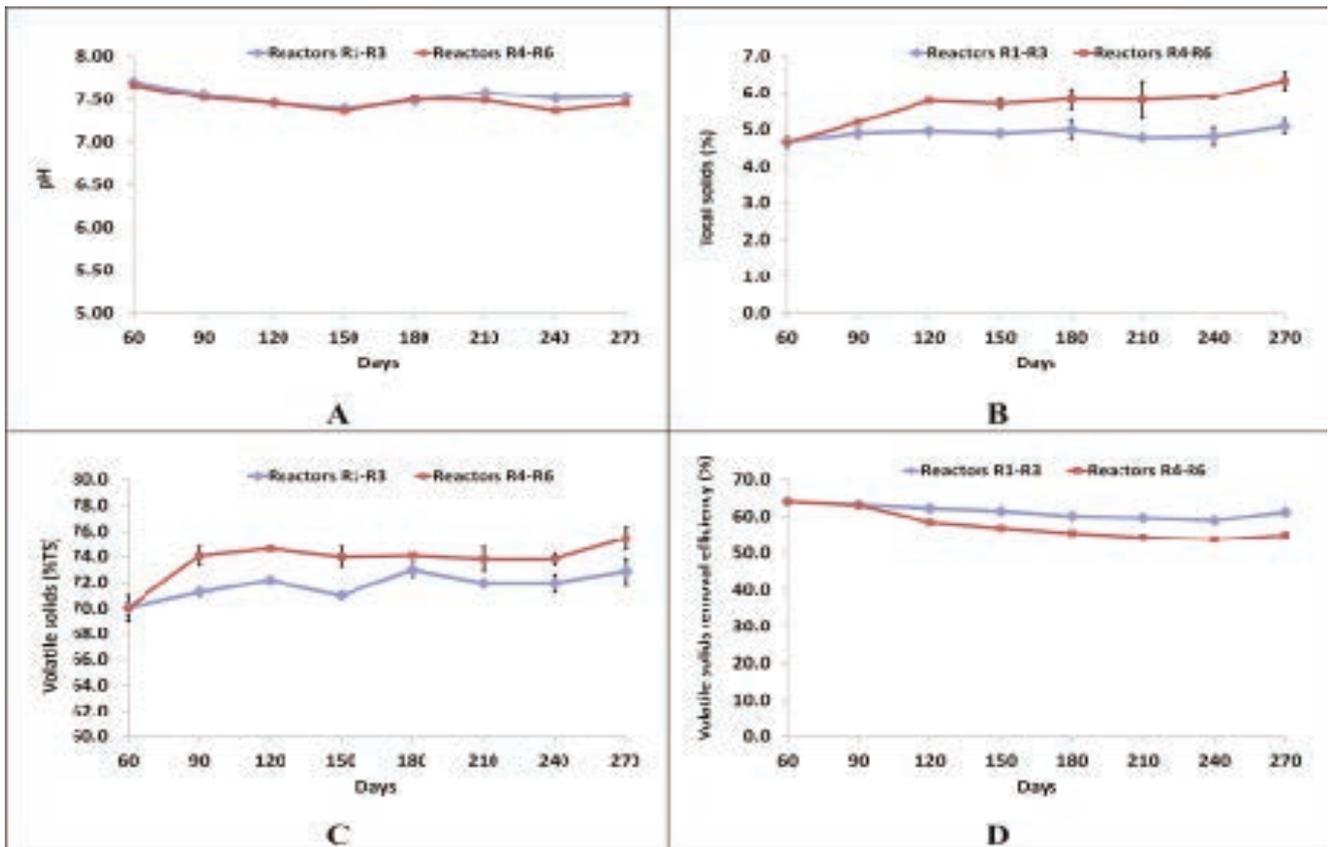


Figure 5. Evolution of pH (A), total solids (B) and volatile solids (C) content in raw co-325 digestate and volatile solids removal efficiencies (D) measured from reactors R1-R3 and 326 reactors R4-R6.

Table 3. Main chemical and physical characteristics of the co-digested solid fraction obtained by mechanical separation of raw co-digestate from reactors R4-R6

Days from the beginning of the experiment	pH	TS (%)	VS (%TS)	TN (%)	TAN (%)	HC (%)	CE (%)	ADL (%)
60 (end of start up phase)	8.26	17.2	85.7	0.43	0.14	5.86	8.51	1.43
90	8.30	18.1	86.6	–	–	–	–	–
120	8.18	17.6	88.8	–	–	–	–	–
150	8.30	16.5	86.9	–	–	–	–	–
180	8.21	16.6	87.6	–	–	–	–	–
210	8.14	16.4	86.9	–	–	–	–	–
240	8.23	17.2	87.5	–	–	–	–	–
270 (end of the experiment)	8.18	17.9	86.5	0.53	0.15	3.75	5.65	5.03

ment lasted 270 days. At the beginning of the experiment (day 0), the reactors were inoculated with 5.5 L of co-digested slurry coming from the selected full scale ADP. Thereafter all reactors (named R1-R8) were fed simultaneously, three times a week, with a determined amount of tested biomasses, throughout the experimental period (270 days). Prior to feeding, an equivalent volume of digester content (raw co-digestate) was discharged. The study was divided into two parts. In the first part of the experiment (startup phase) all reactors were run with feedstock mixture A for 60 days in order to establish a stable digestion process and to ensure steady state conditions. During this period the reactors were operated with an OLR of 2.2 kgVS/ m³ dig. day and a HRT of 40 days, in order to reproduce the same conditions of the

selected full scale ADP (Table 1). At the end of the start-up (day 60), two reactors (named R7-R8) were stopped and raw co-digestate was collected for chemical analysis. The results obtained from these analysis have been used as a baseline to assess the effect of co-digested solid fraction reuse in the digester. In the second part of the experiment (days 61 - 270), a set of three reactors (named R1-R3) continued to be fed with feedstock mixture A (control) and operated as during the startup period (i.e., OLR= 2.2 kgVS/m³ dig. day; HRT= 40 days) while the others three reactors (named R4-R6) were fed using feedstock mixture B (i.e., the same mixture as the control plus all the solid fraction obtained by mechanical separation of the output raw co-digestate collected on daily basis from reactors R4-R6; Figure 2).

Table 4. Average methane yield recorded during the experiment from reactors R1-R3 (feedstock mixture A, control) and from reactors R4-R6 (feedstock mixture B). Standard deviation in parentheses (n=3).

Experimental period (days)	Reactors	Average CH ₄ yield		Variation compared to control (%)	
		Nm ³ /kgVS	Nm ³ /m ³ Dig. day	Nm ³ /kgVS	Nm ³ /m ³ Dig. day
61-90	R1-R3	0.320 (0.008)	0.704 (0.018)	-12.2	+3.35
	R4-R6	0.281 (0.007)	0.728 (0.019)		
91-120	R1-R3	0.325 (0.007)	0.715 (0.015)	-11.7	+3.92
	R4-R6	0.287 (0.006)	0.743 (0.016)		
121-150	R1-R3	0.344 (0.008)	0.756 (0.017)	-11.6	+4.42
	R4-R6	0.304 (0.008)	0.789 (0.021)		
151-180	R1-R3	0.345 (0.010)	0.758 (0.022)	-12.0	+4.36
	R4-R6	0.303 (0.006)	0.791 (0.016)		
181-210	R1-R3	0.343 (0.009)	0.755 (0.020)	-13.9	+2.17
	R4-R6	0.295 (0.007)	0.771 (0.018)		
211-240	R1-R3	0.344 (0.014)	0.757 (0.030)	-14.8	+2.19
	R4-R6	0.293 (0.007)	0.774 (0.018)		
241-270	R1-R3	0.342 (0.009)	0.752 (0.020)	-17.1	+0.28
	R4-R6	0.284 (0.003)	0.754 (0.009)		

Table 5. Main chemical and physical characteristics of the raw co-digestate recorded at day 60 (end of the startup phase) and at day 270 (end of the trial) from reactors R1-R3 (feedstock mixture A, control) and from reactors R4-R6 (feedstock mixture B). Standard deviation in parentheses (n=3).

Days from the start of the experiment	Reactors	pH	TS (%)	VS	TN (%TS)	TAN (%)	HC (%)	CE (%)	ADL (%)
60 (end of start up phase)	R1-R6	7.68 (0.07)	4.66 (0.10)	70.0 (0.76)	0.23	0.14	0.47	0.26	0.21
270 (end of experiment)	R1-R3	7.53 (0.07)	5.09 (0.19)	72.8 (1.03)	0.24 (0.03)	0.15 (0.01)	0.84 (0.14)	0.73 (0.12)	1.13 (0.34)
	R4-R6	7.45	6.33	75.4	0.24	0.20	1.90	0.93	2.23

Mechanical separation of the raw co-digestate was performed by using a lab scale mechanical separator as described by Dinuccio et al. (2008). The total amount of separated raw co-digestate, as well as the amount of solid fraction recovered, were weighed and recorded. Biogas and CH₄ yields were monitored daily throughout the experimental period. Biogas volume was determined connecting the Tedlar® bags to a Ritter drum-type gas meter type TG05/5 instrument, while CH₄ concentration in the biogas was determined using a Draeger XAM 7000 analyzer with infrared sensors. The recorded data were normalized at standard temperature and pressure (0 °C and 1013 hPa) according to German Standard Procedure (VDI 46 30, 2006). The specific yields of biogas and CH₄ were subsequently expressed as normal m³ per m³ digester and day (Nm³/m³ dig. day) or as normal m³ per kg of volatile solids daily fed into the digester (Nm³/kgVS day). During the experimental period the pH, TS and VS of raw co-digestate and co-digested solid fraction were monitored monthly, while TN, TAN and fibres (HC, CE, ADL) were analysed two times: at the end of startup phase (day 60), and at the end of the trial (day 270). All parameters were analysed in triplicate using the same procedures as described in section 2.1. Data were analysed by analysis of variance procedure (ANOVA) followed by Tukey's means grouping tests.

Results and discussion

Characterisation of fresh biomasses and co-digested solid fraction

The main characteristics of fresh biomasses used for the trial are summarized in Table 2. The TS content ranged from 1.13% in pig slurry to about 30% in maize silage, whereas the VS/TS ratio ranged from 0.68 to 0.96. The TAN/TN ratio ranged from 7.43% (sorghum silage) to 78.8% (Farmyard manure). Maize silage had the lowest ADL content, whereas that of farmyard manure was the highest. In Table 3 are shown the main chemical and physical characteristics of the co-digested solid fraction obtained by mechanical separation of raw co-digestate from reactors R4-R6, and used as feedstock for the reactors during the test. Total solids content of co-digested solid fraction ranged from 16.4 to 18.1; VS and TS ratio resulted to be always higher than 0.85 suggesting a residual availability of undigested organic matter. However, the concentrations of HC and CE in co-digested solid fraction tended to decrease over time (Table 3), while, in contrast, the concentration of ADL increased, resulting 1.43% at day 60 (end of startup phase) and 5.03% at day 270 (end of the experiment). The separation efficiency in terms of mass (i.e., the relative amount of co-digested solid fraction obtained by mechanical separation of the raw co-digestate) of the used lab-scale mechanical separator resulted, on average, 9.70% (range 8.70-10.6%).

Continuous anaerobic digestion experiment

Startup phase

During the startup phase (60 days) the average percentage of CH₄ in biogas (Figure 3) gradually increased up to the greatest value (53.8%) at day 13; then it stabilized around an average value of 52.4% (range 50.6 - 53.9%). The average biogas yield followed a similar trend; this trend showed a peak (1.49 Nm³/m³ dig. day) at day 18 followed by a steady state period (days 19-60) during which the biogas yield averaged 1.40 (range 1.32 - 1.48) Nm³/m³ dig. day. During the 41 days steady state period, the average daily CH₄ produced by reactors R1-R8 ranged between 0.313 and 0.353 Nm³/kgVS, comparable to values measured by Gioelli et al. (2012) during a 12 months period of monitoring of the selected full scale ADP; the degree of VS degraded during the anaerobic digestion process resulted 64%. Investigations of 41 biogas plants in Austria by Hopfner-Sixt and Amon (2007) found CH₄ yields from co-fermentation of animal manure and energy crops up to 0.39 Nm³/kgVS, with VS degradation rates of 78–84%. The lower degree of degradation found in this study can be explained by the shorter HRT (40 days) of the reactors, which is similar to that of the selected full scale ADP (Table 1) but lesser than the minimum HRT of 45 – 60 days recommended in the literature (e.g., Öchsner and Helffrich, 2005) for an optimal degradation of VS content in energy crops.

Effect of reuse of the co-digested solid fraction as a feedstock on the performances of anaerobic digesters

Figure 4 depicts the average daily CH₄ yield for each feedstock mixture (A and B) recorded during the second part (days 61-270) of the experiment. During this 210 days period, the average volumetric CH₄ produced by mixture A (control, reactors R1-R3) ranged between 0.674 and 0.802 Nm³/m³ dig. day, reflecting the variability of the characteristics of fresh biomasses (Table 2) collected at the selected ADP during the experiment. The specific CH₄ yields, expressed as Nm³/kgVS (Table 4), obtained over the experimental period by feedstock mixture B (reactors R4-R6) were, on average, 17% lower than those recorded from the control (feedstock mixture A reactors R1-R3). However, the average daily volumetric CH₄ yields by mixture B (reactors R4-R6) (Figure 4) were generally higher than those obtained by mixture A for most of the experimental period, although such differences were not statistically significant (p>0.05). The reuse of the co-digested solid fraction in reactors R4-R6 gradually increased the average volumetric CH₄ production rate from 0.728 (days 61-90) to 0.791 (days 151-180) Nm³/m³ dig. day (Table 4). The latter value corresponds to an increase of 4.36% when compared to the average volumetric CH₄ production rate (0.758 Nm³/m³ Dig. Day) recorded from reactors R1-R3 (control). After this period such differences did, however, start to decrease, dropping to a value of +0.28% during the last 30 days of trial (Table 4).

The pH values of the raw co-digestate recorded over time (Figure 5A) suggests a regular course of the anaerobic digestion process within all the reactors. The average pH values of co-digestate from reactors R4-R6 ranged between 7.4 and 7.7, within the optimum range (6.5–7.8) for the

adequate growth of anaerobic microorganisms (Liu et al., 2008). This observation indicates that the process adapted well to the introduction of the co-digested solid fraction as co-substrate, as pH fluctuation is a widely used indicator of process stress in anaerobic reactors (Ward et al., 2008). However, the concentration of TAN (Table 5) in raw co-digestate from reactors R4-R6 has shown the tendency to increase, resulting 0.14% at day 60 (end of start up phase) and 0.20% at day 270 (end of the experiment), indicating the possibility of inhibition on the activity of microorganisms. Free ammonia has been suggested to be the main cause of inhibition in anaerobic digesters due to its high membrane permeability (Kroeker et al., 1979; de Baere et al., 1984). Ammonia inhibition was reported to occur above pH 7.4 in the range of 1500–3000 mgTAN/L, whereas at concentrations in excess of 3000 mgTAN/L, ammonia was claimed to be toxic irrespective of pH (Van Velsen, 1979; Koster and Lettinga, 1984). A remarkable increase over time of hemicelluloses, celluloses and lignin content of raw co-digestate from reactors R4-R6 was also observed (Table 5). Lignin is not degradable under anaerobic conditions and may prevent microbial access to hemicelluloses and celluloses (Mussatto et al., 2008). On average, the concentration of TS (Figure 5B) and VS (Figure 5C) in raw co-digestate from reactors R4-R6 resulted, respectively, 15.5% and 18.9% higher than the concentration in raw co-digestate from reactors (R1-R3). An average VS removal efficiency (Figure 5D) of 66.0% and 63.6%, respectively, for reactors R1-R3 and reactors R4-R6 was calculated.

Conclusions

The results obtained in this laboratory-scale study confirm that the co-digested solid fraction can still contain a high biogas and methane potential. The reuse of the co-digested solid fraction as feedstock for ADP seems to be an interesting option. Under the specific laboratory conditions adopted in this study, the long-term reuse of the co-digested solid fraction into the digester improved the total CH₄ production by approximately 4%. However, after 120 days of continuous recirculation of the co-digested solid fraction the volumetric CH₄ yield of the reactors started to decline, mainly due to the accumulation of recalcitrant organic fibres (e.g., lignin) which are compounds minimally digestible by anaerobic microorganisms. Therefore it is suggested to restrict this practice for limited periods of time, monitoring regularly the productivity of the ADP (e.g., daily yield of biogas and methane per m³ of digester) and the key process parameters (e.g., pH and TAN concentration in raw co-digestate) in order to maintain such variables steady and within the optimal ranges for the adequate growth of anaerobic microorganisms. Nevertheless, full scale ADP conditions may differ from the laboratory. Thus, to confirm the results obtained in this study, experiments should be made in full scale ADP.

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