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Effect of substrate concentration on dry mesophilic anaerobic digestion of organic fraction of municipal solid waste (OFMSW)

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Abstract

The influence of total solid contents during anaerobic mesophilic treatment of the organic fraction of municipal solid waste (MSW) has been studied in this work. The work was performed in batch reactors of 1.7 L capacity, during a period of 85–95 days. Two different organic substrate concentrations were studied: 931.1 mgDOC/L (20% TS) and 1423.4 mgDOC/L (30% TS). Experimental results showed that the reactor with 20% total solids content had significantly higher performance. Thus, the startup phase ended at 14 days and the total DOC removal was 67.53%. The startup in reactor R30 ended at 28 days obtaining 49.18% DOC removal. Also, the initial substrate concentration contributed substantially to the amount of methane in the biogas. Hence, the total methane production in the methanogenic phase was 7.01 L and 5.53 L at the end of the experiments for R20 and R30, respectively.
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Keywords: Anaerobic digestion; Organic fraction of municipal solid waste (OFMSW); Mesophilic; Biogas; Methane

1. Introduction

The production of municipal solid waste in Spain has increased from 18,783,442 (year 1998) to 24,163,199 tons (year 2003), that which supposes an increment of 28.64% in this period (INE, 2006). Of these, 40–45% corresponds to the fraction of organic, it is known as OFMSW (organic fraction of municipal solid waste). This OFMSW is susceptible of energy appraisement by the application of biological treatments.

The anaerobic digestion has been considered as the main commercial option for the treatment of the OFMSW. It produces methane and generates a digested residue that is similar to the compost produced aerobically. The process is conditioned by initial performance parameters such as: the feeding regime, the total solid contents in OFMSW, the solids retention time (SRT) and the temperature. The microorganisms are classified according to the good range of temperature in which they are developed (Romero et al., 1993; Angenent et al., 2002): psicrophilic ($T < 15$ °C), mesophilic (15 °C $< T <$ 45 °C) and thermophilic ($T >$ 45 °C). The operation in mesophilic range (33–37 °C) is more stable and requires a smaller energy expense. Also, the inhibition for ammonium (Angelidaki and Ahring, 1994; Hansen et al., 1998) and for volatile acids of long chain (Fields, 2001) is more unusual.

The methane production – and the methane yield – can be used to evaluate the anaerobic digestion of OFMWS (de Baere, 2000). In addition, methane yield can be compared with the theoretical methane yield based on the composition of the waste. The methane yield from anaerobic digestion of OFMSW has been studied in recent years (Gunaseelan, 1997; Forster-Carneiro et al., 2004, 2007a, 2007b). Actual researches on anaerobic digestion report that substantial differences are observed in the methane yields and kinetics depending on the food waste or MSW type (Forster-Carneiro et al., 2004; Rao and Singh, 2004). However, few reports can be found on the study of dry anaerobic digestion of food waste, and the explanation of

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solid wastes anaerobic digestion performance in the start-up period from the point of view of the total solids content is not extensively investigated yet.

In this work, the effect of substrate concentration (based on the total solids contents in the reactor) on the mesophilic anaerobic digestion is analyzed in the startup phase. Also, the biogas generation (methane yield) was analyzed.

2. Methods

The experimental assays were performed using separately collected OFMSW from "Las Calandrias" treatment plant located in Jerez de la Frontera (Spain). The mesophilic sludge was selected coming from a waste water treatment plant located in Jerez de la Frontera (Spain). In the previous works, the mesophilic sludge showed to be an appropriate inoculum for anaerobic digestion of food waste (Álvarez Gallego, 2005; Forster-Carneiro et al., 2007a).

2.1. Experimental reactors

The system was conformed by four reactors. The assays were carried out in batch discontinuous reactors with an internal diameter of 12.2 cm and a height of 16.5 cm. The total volume capacity was 1.7 L (laboratory scale). Each reactor had independent agitation system and electric control. The main axis contained 11 horizontal and cylindrical crosses willing to different heights, and it rotated in alternate senses by means of a cylindrical temporize, capable to maintain uniform moisture content and to redistribute soluble substrate and bacteria. The operational temperature was 35 °C, controlled and monitored by means of thermostatic bath model SELECT CORP.

The conditions selected were dry anaerobic digestion (with 20% and 30% TS) at single phase mesophilic (35 °C) condition.

2.2. Characteristics of wastes and inoculums

The physical–chemical characteristics of OFMSW and mesophilic sludge are shown in Table 1.

The solid percentage of OFMSW coming from industrial treatment plant was 57.17% (high humidity degree) and the maximum original size of particles of OFMSW was 30 mm. For lab-scale studies, a previous stage of size reduction was required to increase the specific surface and to obtain more homogeneous samples. Hence, pretreatment of OFMSW, consisting in drying, crushing and shredding until obtaining particle sizes of 10 mm approx., was required to provide a suitable refined digested material, reaching 82.34% initial total solids in the samples.

Later, mesophilic inoculum with 3.64% of TS was added. Finally, water was added to fit the initial total solid contents specified in both digesters: 20% and 30% TS.

Table 1
Main physical–chemical characteristics of substrates and initial mixtures of the reactors

	OFMSW	Sludge	Mixture 20%	Mixture 30%
pH	6.51	7.49	6.68	6.46
Density (g/L)	0.666	0.971	1.035	1.130
Alkalinity (mg$CaCO_3$/L)	14.0	8.2	9.2	12.4
DOC (mg/L)	371.62	56.32	931.1	1423.4
TS (%)	82.34	3.64	16.61	27.87
VS (%)	29.89	1.85	7.03	10.12

2.3. Experimental procedure

The study is programmed to evaluate the mesophilic digestion of OFMSW at two different initial substrate concentrations in the process (coinciding with different total solids concentration): 931.1 mgDOC/L (R20: 20%TS) and 1423.4 mgDOC/L (R30: 30%TS). Four reactors were used (rehearsals for copy) of 2 L total volume and 1.7 L useful volume at discontinuous condition, inoculated with 30% (in volume) of mesophilic digested sludge. The characteristics of the initial mixtures are shown in Table 1.

2.4. Analytical methods

Every 2 days, representative samples of anaerobic digester effluents were taken. The parameters analyzed three times a week were: pH, total solids (TS), volatile solids (VS), alkalinity, volatile fatty acid (VFA) and dissolved organic carbon (DOC). Daily biogas volume and composition (%H_2; %O_2; %N_2; %CO_2 and %CH_4) were analyzed. All analytical determinations were performed according to "Standard Methods" (APHA, 1995), after drying, grinding and dilution of the samples. This procedure is more representative due to the semi-solid characteristic of the substrate (except for TS and VS that is not necessary to dilute the samples) (Guitian Ojea and Carballas, 1968).

The dissolved organic carbon analysis was carried out in a SHIMADZU 5050 DOC Analyzer, by combustion-infrared method (5310B) of "Standard Methods". TS and VS analysis were realized as 2540-B method described in APHA (1995): TS samples were dried in an oven at 105–110 °C, and for VS to the dried ash waste in a furnace at 550 ± 5 °C. The alkalinity of samples was determined in COMPACT TITRATOR S^+ Crison Instruments S.A.

Gaseous analyses were determined by removing a representative sample from the Tedlar bag. The volume of gas produced in the reactor was directly measured using a high precision flow gas meter – WET DRUM TG 0.1 (mbar) – Ritter. – through a bag Tedlar (SKC serie 232). The biogas composition was carried out by gas chromatography separation (SHIMADZU GC-14B) with a stainless steel column packed with Carbosive SII (diameter of 3.2 mm and 3.0 m length) and thermal conductivity detector (TCD). The injected sample volume was 1 cm^3 and the operational conditions were as follows: 7 min at 55 °C; ramped at 40 °C min^{-1} until 150 °C. Constant temperature during

5.5 min. Ramped at 40 °C min^{-1} until 180 °C. Constant temperature during 4.3 min. Detector temperature: 150 °C; injector temperature: 100 °C. The carrier was helium and the flow rate used was 30 mL min^{-1}. A standard gas (by Carburos metálicos S.A.); composition: 4.65% H_2; 5.3% N_2; 69.9% CH_4 and 20.1% CO_2) was used for the calibration of the system.

The fatty acid levels were determined by a gas chromatograph SHIMADZU GC17A equipped with a flame-ionisation detector and capillary column filled with Nukol (polyethylene glycol modified by nitro-terephthalic acid). The temperature of the injection port and detector were 200 °C and 250 °C, respectively. Helium was the carrier gas at 50 mL min^{-1}. In addition, nitrogen gas was used at 30 mL min^{-1} flow rate. Total VFA were calculated as the addition of individual VFA levels. Total acid concentrations were calculated as the addition of individual volatile fatty acid levels.

3. Results and discussion

3.1. Substrate characteristics

Table 1 shows a summary of the physical–chemical characterization of substrate, inoculum and initial mixtures of each reactor. The experiments were carried out in duplicate and the results expressed as mean values. The experiments were concluded when no significant variation of dissolved organic carbon (DOC) and cumulative methane production was observed.

The pH and alkalinity values of the mixtures are in accordance with the composition of OFMSW and the sludge. The density of the mixtures increases in both cases, being higher in the mixture with 30% TS. The initial volatile solids content was 30.53% higher than R20.

3.2. Variation of the operational parameters: DOC and VS

Fig. 1 shows temporal DOC evolution in biomethanization processes of OFMSW reactors with different initial substrate concentrations. Different degradation phases are observed in the process: the left part of the graph corresponds to the hydrolytic and acidogenic stages; the central part corresponds to the development of the methanogenic stage and the right area is related with the final phase of the process, due to the lack or insufficiency in biodegradable organic matter.

During the first days, the external pH control was necessary in both reactors. Four additions of 10 mL NaOH 7 N were realized during these days. This pH control favoured the biodegradation process (Pérez et al., 2005) evaluated as DOC removal. As can be seen in Fig. 1, DOC values decreased as a function of the digestion time. All test showed optimal microbial activity, reaching stable values after 15 or 35 days since inoculation in R20 and R30, respectively. From this moment and until 45 and 80 days operating period, the reactor showed the greatest efficiency of substrate removal with values near to 80.69% and 69.05% DOC removal in R20 and R30, respectively. In relation with VS, between the days 15–45 and the days 35–80, again the best values of VS removal were for R20 with 49.95% VS removal efficiency against the 40.91% for R30.

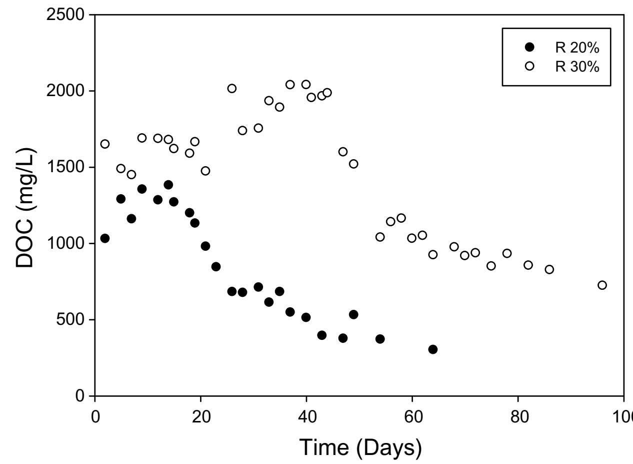

Fig. 1. Changes of DOC (as mgDOC/L) with time (days) in biomethanization processes of OFMSW: ●: R20 ○: R30.

Hence, we conclude that organic matter removal in the reactors took place after 15 and 35 days, respectively for R20 and R30. The DOC removal efficiency values were higher for R20 than that for R30.

3.3. Volatile fatty acids degradation and acidity evolution

The evolution of total acidity and temporal evolutions of individual and total volatile fatty acids (mg/L) in two reactors R20 and R30 are shown in Figs. 2 and 3, respectively. The profile of the temporal evolution of total acidity showed two phases: initially, an increase was observed until the methanogenesis started and later, the acidity decreased until it reached the end of the biodegradation process.

Total fatty acids (as mgAcH/L) showed the same profile evolution. Volatile acids represent a critical control parameter of anaerobic digestion. As can be seen, in this study, the reactors R20 and R30 showed a typical evolution. Initially in the reactors with 20% TS, hydrolytic and acidogenic stages were observed (0–15 days) with a high fatty acid generation between days 6 and 15. After day 15, the hydrolyzed organic matter was transformed to volatile fatty acids, mainly butyric and acetic acids, suggesting the stabilization phase. The concentration of these volatile acids in the digester is determined by their production rate and their removal rate; in this case, the acetic acid removal rate is superior to its production rate. In the reactors with 30% TS, hydrolytic and acidogenic stages were observed (0–35 days) with a high fatty acid generation. Later, the acids begin to degrade.

The higher concentrations of volatile fatty acids generated were with for R30, with values in the range 164.0–1254.0 mg acetic acid/L and 183.0–583.0 mg butyric acid/

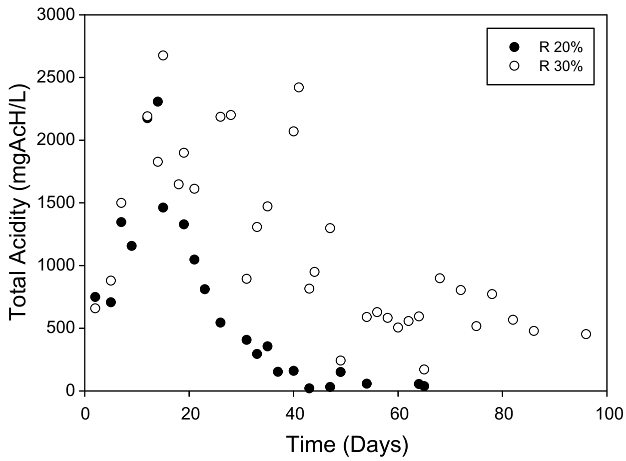

Fig. 2. Changes of total acidity (as mg/L) with time (days) in biomethanization processes of OFMSW: ●: R20 ○: R30.

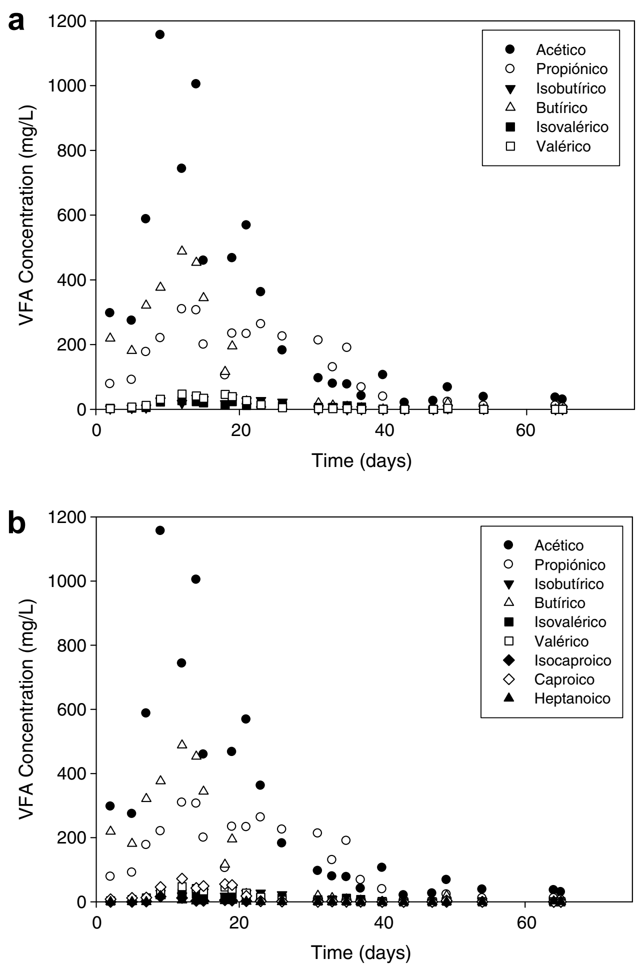

Fig. 3. Changes of individual volatile fatty acids (as mg/L) with the time (hours) in biomethanization processes of OFMSW: (a) R20 (b) R30.

L. For R20, the range was 276.0–1181.0 mg acetic acid/L and 180.0–576.0 mg butyric acid/L. The total VFA value for R20 and R30 reached a maximum value of 2307.59 mg/L and 2743.26 mg/L and no destabilization was observed. These results were compared with other researches and studies, which have been conducted previously (de la Rubia et al., 2005, 2006), where approximately 70% of the digester methane originated from acetate (the most important precursor on which to focus) and the remainder of the digester methane (approximately 30%) originated mainly from the reduction of dioxide of carbon by hydrogen (Chynoweth et al., 2000).

3.4. Acidity/alkalinity evolution

The initial pH of reactors had an average value of approximately 6.7–6.5 for R20 and R30, respectively. In both assays, the daily adjustment of the pH with hydroxide sodium (6N) was necessary until it reached approximately. 8.0 at day 15–20, which is an optimum value in the mesophilic range. In this way, the pH and alkalinity (closed to 0.06 $gCaCO_3$/kg) increased due the NaOH added to the reactor. Both parameters showed suitable values to maintain a stable condition in the digester for optimal biological activity.

Acidity/alkalinity ratio increased until 0.42 for R20 and 0.53 for R30 at day 15 due to the generation of fatty acids in the acidogenic stage. Later, the ratio values decreased until they reached low values for both reactors, more remarkable for R20. Usual values of acidity/alkalinity ratios published by de la Rubia et al. (2005) are near to 0.09 for stable mesophilic processes operating with municipal wastewater sludge at pilot scale conditions.

3.5. Biogas and methane production

Fig. 4 shows the temporal evolution of biogas produced in both reactors. The different solid concentration conditioned the total biogas and the temporal evolution. Thus, for R20 (Fig. 4a), the biogas production was detected in the period of 14–39 days, while for R30 (Fig. 4b) it was observed during the period of 50–76 days, with two different generation trends. This could be caused by the degradation of minority fractions of the substrate.

Fig. 5 shows the evolution of biogas components: H_2, CO_2 and CH_4. The different solid concentration conditioned the hydrogen production during the first days of the study. In reactor R20, the maximum production reached 0.31 L H_2 while in reactor R30 the maximum value was 0.01 L H_2 in the hydrolytic and acidogenic phases.

Methane was present in small percentages, like it corresponds to the hydrolytic stage. All methane detected in this stage corresponds to the activity of the hydrogen utilizing microorganisms, since this component is not detected in the biogas.

Later, hydrogen generation stopped and a progressive increase of methane was observed from day 15 until it reached approximately. 80% at the end of this stage in R20 (Fig. 5a). In reactor R30, acidogenic stage was prolonged until day 35 approximately.

During the later stages, the percentages remained practically constant in 80% methane and 20% carbon dioxide

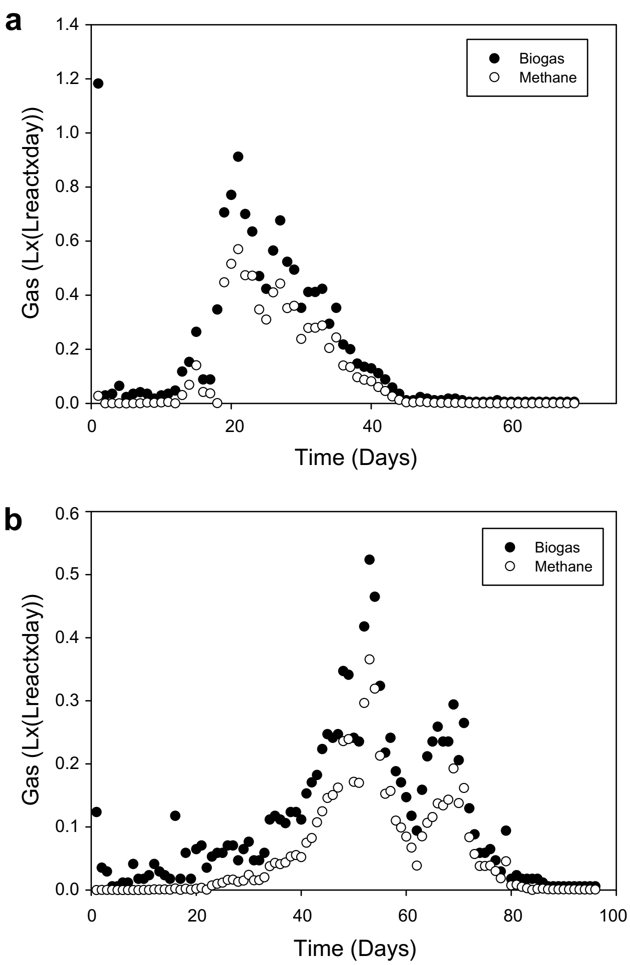

Fig. 4. Evolution of biogas and methane (as L/L d) with time (days) in biomethanization processes of OFMSW: (a) R20 (b) R30.

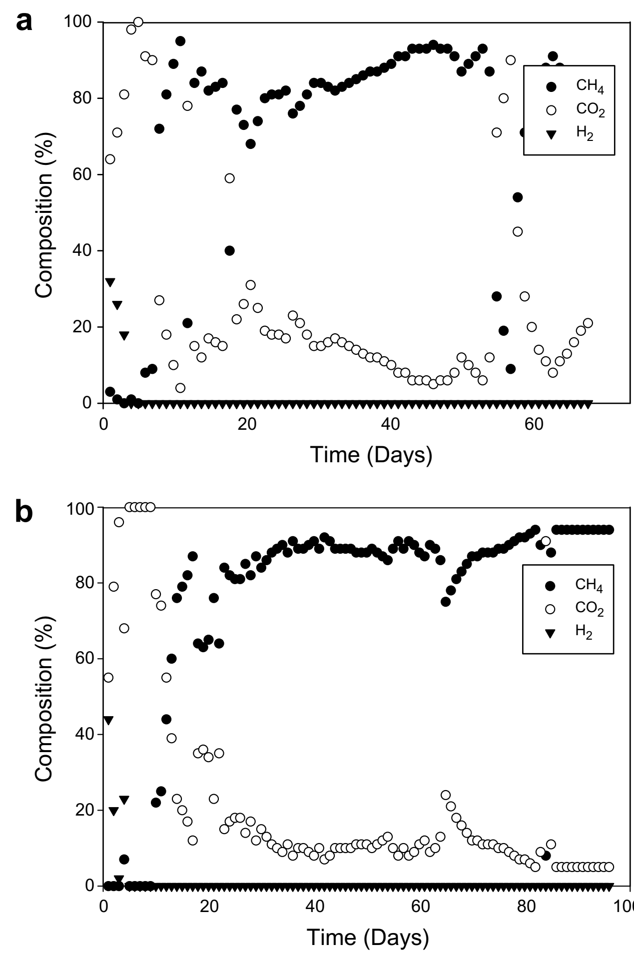

Fig. 5. Changes of biogas components (as%) in biomethanization processes of OFMSW: (a) R20 (b) R30.

for R20. Near to day 60, a distortion was detected that could be caused by the degradation of minority fractions of substrate with less biodegradability that had not still been degraded.

The profile of the temporal accumulation of methane in the produced biogas produced, presented in Fig. 5, shows that the total solids amount contributed substantially in increasing the amount of methane in the biogas. The total accumulated methane and carbon dioxide were 7.01 L and 1.83 L (Fig. 5a).

In R30, methane and carbon dioxide began to accumulate starting from day 35 reaching 90% of methane and 10% of carbon dioxide at the end of the methanogenic stage. The volume of total methane and carbon dioxide generated was 5.53 L and 0.77 L (Fig. 5b).

3.6. Comparative process efficiency

Table 2 shows a summary of performance data at the end of the process for all two reactors studied. The extension of the hydrolytic–acidogenic stage and, therefore, the beginning of the methanogenic phase, is 28 days in reactors with 30% TS, approximately double time that the systems with 20% in solids (15 days). In both cases, DOC removal profiles were similar to VS removal profiles.

The highest methane production was observed in R20 after the second week of inoculation. Over 70 days operation process, the greatest methane yield (as CH_4/gVS_r and $CH_4/gDOC_r$) was obtained for R20 reactor with values of 0.11 LCH_4/gVS_r and 4.11 $LCH_4/gDOC_r$ in methanogenic phase in front of R30 with 0.07 LCH_4/gVS_r and 2.79 $LCH_4/gDOC_r$.

The differences observed in digester with different initial total solids contents are summarized as follows:

(a) The temporal extension of digestion process is a function of the initial percentage of solids in the process: the methanogenic stage begins at day 14 for R20 (20% TS) and at day 28 for R30 (30% TS). Hence, an increment of initial organic matter prolongs the hydrolytic and acidogenic stages.
(b) Biogas and methane production increase from day 14 or 28 in the methanogenic stage, obtaining 7.01 LCH_4 and 5.53 LCH_4 for R20 and R30, respectively.

Table 2
Main results of the mesophilic anaerobic reactors for the degradation of the OFMSW with 20% and 30% TS

	R20	R30
Hydrolytic and acidogenic phases		
Duration of the phase (days)	14	28
Production CH4 (L)	0.14	0.06
Production CO2 (L)	0.65	0.05
Production H2 (L)	0.31	0.01
Methanogenic phase		
Duration of the phase (days)	28	49
Production CH4 (L)	7.01	5.53
Production CO2 (L)	1.83	0.77
Production H2 (L)	0.00	0.00
CH_4/g DOC removal (L)	4.11	2.79
CH_4/g VFA removal (L)	1.93	1.80
CH_4/g VS removal (L)	0.11	0.07

(c) The decrease of biogas and methane generation is detected at day 45 for R20 and at day 80 for R30, due to the consumption of main biodegradable organic matter. Nevertheless, the biogas and methane produced show a later increase toward day 25 (R20) and day 60 (R30). The heterogeneity of the organic waste explains this fact.

4. Conclusions

The results of this research show that the initial substrate concentration influences the mesophilic anaerobic digestion of OFMSW at batch conditions. The methanogenic stage begins at day 14 for R20 (with 931.1 mgDOC/L or 20%TS) and at day 28 for R30 (1423.4 mgDOC/L or 30%TS). Reactor R20 gives maximal total DOC removal of 80.69%. Instead, reactor R30 gives smaller DOC removal, with 69.05% DOC removal. The profile of the temporal evolution of methane in the biogas shows that the initial substrate concentration contributed substantially in increasing the amount of methane in the biogas. Hence, the total methane production in the methanogenic phase was 7.01 L and 5.53 L at the end of the experiments for processes with 20% TS and 30% TS, respectively. The highest methane production was observed in R20 after the second week from inoculation. Over 45 days operation process, the greatest methane yield was obtained for R20 reactor with values of 0.11 LCH_4/gVS_r and 4.11 $LCH_4/gDOC_r$ in methanogenic phase. R30 shows a productivity of methane 17% smaller than the processes with 20% TS.

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