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# Composting potential of different inoculum sources in the modified SEBAC system treatment of municipal solid wastes

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#### Abstract

The aim of the work described here was to analyse the biomethanization process for three types of organic fraction of municipal solid waste (OFMSW) using two different inoculum sources in a sequential leach-bed anaerobic reactor under thermophilic (55 °C) and dry conditions (20%TS). The OFMSWs studied were: separately collected food waste (SC\_OFMSW) from restaurants, synthetic waste (ST\_OFMSW) and mechanically selected municipal waste (MS\_OFMSW). The inoculum studied were digested mesophilic sludge (SLUDGE) and digested SC\_OFMSW. The results show that SLUDGE gave the best performance and this was therefore selected for the rest of the experiments. Three assays were performed to analyse the biomethanization processes of three types of organic waste. The results suggest that all three wastes give rise to an acclimation stage with acidogenic/acetogenic activity between days 5 and 20–30 and a stabilization phase associated with methanogenic activity. In conclusion, a mixture of OFMSW (regardless of its origin) and SWINE arranged in layers in the reactor, as well as SLUDGE used with an inoculum source, enhanced the fast start up phase of a modified sequential leach-bed system under dry thermophilic conditions.

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### 1. Introduction

The generation of municipal solid waste (MSW) is a serious problem for urban communities. Organic solids are present in very large quantities as products or waste from agriculture, the food industry and market waste. Spain generates approximately 24 million tonnes of MSW annually (Macé et al., 2005). According to data published by the National Plan of Urban Residuals (2000–2006), 40–45% of all MSW is the organic fraction of municipal solid waste (OFMSW).

The composition of the OFMSW is influenced by various factors, including regional differences, climate, collection frequency, season, cultural practices and changes in composition (Tchobanoglous et al., 1997). In this respect, numerous papers have focussed on aspects related to the anaerobic digestion biodegradation of the OFMSW according to its origin: e.g., food waste, fruit and vegetables, kitchen waste, household waste and municipal waste.

The removal and alternative treatment of the organic fraction from landfill sites is likely to have an impact by increasing the methane yields as the concentration of food waste in municipal refuse increases. Food waste is a biodegradable component of refuse and a range of management alternatives exist, including anaerobic digestion and aerobic composting (Cecchi et al., 1992).

Anaerobic digestion has several advantages over traditional solutions (landfill, incineration and aerobic composting) and these include better handling of wet wastes and the production of useful digester gas (Pavan et al., 1998; Chynoweth et al., 2002).

Anaerobic digestion, also called fermentation or biomethanization, uses closed reactors to control the

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#### Nomenclature

MSW municipal solid waste	TS	total solids (g/kg)
OFMSW organic fraction of municipal solid waste	VS	volatile solids (g/kg)
SC_OFMSW separately collected organic fraction of	TSS	total suspended solids (g/L)
municipal solid waste	VSS	volatile suspended solids (g/L)
ST_OFMSW synthetic waste organic fraction of muni-	TOC	total organic carbon (g/L)
cipal solid waste	DOC	dissolved organic carbon (g/L)
MS_OFMSW mechanically selected organic fraction of	COD	chemical oxygen demand (g/L)
municipal solid waste	TNK	total nitrogen Kjeldahl (mg/L)
LEACH modified sequential leach-bed anaerobic pro-	N–NH	4 ammonia Nitrogen
cess		

anaerobic process and limit the uncontrolled production of greenhouse gases that pose a threat to the environment. In this process anaerobic microorganisms digest the organic material to produce carbon dioxide and methane, which can be collected and used as a fuel for heating and/or to produce electricity (biogas). The methane produced in this way is competitive in terms of efficiency and cost with other biomass energy forms (Chynoweth et al., 2001). The stabilized solid residue, which averages 40–60% by weight of the feedstock (Kulik, 1997), is an excellent soil conditioner that has a high nutrient content after approximately 30 days (Angenent et al., 2002).

Anaerobic digestion has proven to be a viable option for the management and stabilization of the organic fraction of municipal solid waste (OFMSW). Conventional anaerobic digesters require feed material with total solids content below 10%. However, modern systems can deal with feeds that have total solids contents of over 20% (Bolzonella et al., 2003a). Anaerobic digestion processes in semi-dry (Pavan et al., 1994) and dry conditions (total solids content of 20–35%) are considered capable of producing an inert biosolids product with higher methane productivity (Mata-Alvarez et al., 2000; De Baere, 2000). In this approach sludge (De la Rubia et al., 2001; Paredes et al., 2005) can be added to the municipal organic waste. In addition, there is considerable interest in applying dry anaerobic digestion under thermophilic conditions (55 °C) (Ahring, 1992) to treat the organic fraction of municipal solid waste (OFMSW) (Kim et al., 2002).

Two main technologies have been used for the rapid treatment of OFMSW: a sequential leach-bed anaerobic process (O'Keefe et al., 1993) and CSTR reactors (Pavan et al., 2000) or Batch systems (Lissens et al., 2001). Both technologies have very simple designs and there are numerous reports on their use. However, the CSTR systems are the least expensive high solid digesters. The sequential leach-bed anaerobic process was developed to overcome common problems associated with anaerobic reactor designs: i.e., the high-solids content (20–80%), inoculation, mixing and instability (Nopharatana et al., 1998; Pullamm-anappallil et al., 2001). In conclusion, this system is superior and more economical than other competing tech-

nologies and is also a source of employment and improves the hygiene and aesthetics of the community. A similar process is sequential batch anaerobic composting (SEBAC), which was employed by Chynoweth et al. (1992) (SEBAC homepage, 2005).

The sequential leach-bed technology requires two reactors: one containing unsorted fresh waste (hereafter called reactor A) and another with anaerobically stabilized waste (hereafter called reactor B). The process involves wetting fresh waste with stabilized waste until a leachate (moisture free) trickles out of the bed (O'Keefe and Chynoweth, 2000). The stabilized waste contains a balanced active and anaerobic population of acid forms and methanogens. The procedure is repeated until a balanced active bacterial population is stabilized in the bed of fresh waste. This bed can subsequently be used to start the inoculation of a new bed (Chugh et al., 1999). The process is simple in design, easy to operate and guarantees stability with a built-in mechanism for the prevention of imbalance. The process does not require solid handling during the digestion process.

The start up is generally considered the most critical step in the operation of anaerobic digesters. The source of microorganisms, the size of the inoculum, and the initial mode of operation are all important factors during start up (Hobson and Wheatley, 1993). The mesophilic anaerobic sludge digester has proven to provide an excellent inoculum source in previous studies (Forster-Carneiro et al., 2004) and this inoculum should be readily available (Ahring, 1994) because it is grown in a similar anaerobic environment (Kim et al., 2002).

A new configuration for the leach-bed process was proposed in previous studies by our research group with the aim of facilitating the percolation of the leachate and, consequently, enhancing the biodegradation of restaurant wastes (Forster-Carneiro et al., 2004). The protocol involves the use of pre-treated waste that mixes rice hulls or garden waste with the OFMSW and this mixture is then arranged in layers with animal excrement (the modified sequential leach-bed anaerobic process will hereafter be called LEACH). Studies aimed at comparing the start up and stabilization phases in sequential leach-bed anaerobic processes under dry and thermophilic conditions have not been published to date for the treatment of the two most important types of municipal solid waste: food waste (from a university restaurant) and municipal solid waste (from a treatment plant).

The aim of the work described here was to study the anaerobic digestion process for three types of organic fraction of municipal solid waste (OFMSW) using two different inoculum sources (sludge and SC\_OFMSW digest) in a modified sequential leach-bed anaerobic digestion (LEACH) process under dry ( $\geq$  30%) and thermophilic (55 °C) conditions. The emphasis was placed on the fast conversion of the OFMSW to biogas in order to achieve the rapid onset of a balanced microbial population in the LEACH process in a particular digestion stage: the start up phase. In addition, the effects of several operational parameters on the start up strategy for each type of OFMSW were explored.

# 2. Materials

# 2.1. Anaerobic reactors

The experiments were carried out in leach-bed discontinuous reactors made from PVC with an internal diameter of 0.30 m and a total height of 0.50 m. The capacity of each reactor was 25 L (laboratory scale) for a single-phase anaerobic process and discontinuous digester. The cover of each reactor incorporated three separate ports for three different functions: (1) the addition of sludge feed; (2) retrieval of the leachate and (3) measurement of the biogas composition and production. The reactor did not have any mechanical parts inside. This configuration allowed the systems to operate under high-solids conditions without any adverse effects on leachate circulation and without the need for maintenance of mechanical devices.

A schematic representation of these reactors is shown in Fig. 1. The leachate from reactor B was recycled to reactor A with a peristaltic pump on a daily basis. Reactors A and B were both independently connected to a 40 L Tedlar bag in order to collect any evolved gas.

Anaerobic digestion was performed under thermophilic conditions (55 °C). The reactors were kept inside a special room constructed with galvanized steel foil (40 kg/m<sup>3</sup>) (FAYMO-M, Spain). The temperature was controlled by three electric heaters (model PC-1000W, S&P, Spain) and monitored by digital sensors (Thermo digital-TFFI, Spain) installed within the room. An electric fan circulated air inside the room.

#### 2.2. Substrate selection and characterization

Five different sequential leach-bed anaerobic processes were evaluated in this study and these are denoted as LEACH 1, 2, 3, 4 and 5.

The unsorted and fresh organic fractions selected for use in reactor A for each process were as follows:

- (1) Separately collected biowaste fraction (SC\_OFMSW) obtained from a university campus restaurant (Cádiz-Spain).
- (2) Synthetic food waste (ST\_OFMSW) organic fraction (highly biodegradable and reproducible feedstock) was selected and mixed. The original mixture was produced by Martin et al. (1999), but in this study



Fig. 1. Schematic diagram of the experimental LEACH reactors.

Table 1

Composition	of	synthetic	organic	fraction	of	municipal	solid	waste
(ST_OFMSW	') fc	r reactor A	A of LEA	ACH 4				

Composition		Weight (kg)
Vegetables	Lettuce	13.0
	Cauliflower	3.0
	Cabbage	1.0
Vitamins and minerals (fruit)	Pear	2.5
	Banana	5.0
	Golden apple	2.5
	Fuji apple	2.5
	Orange	7.0
Proteins (meat)	Meat	3.5
Vitamins and minerals	Onion	4.0
	Carrot	1.0
	Potatoes	9.0
	Tomatoes	2.0
Legume	Rice	3.5
	Bean	2.0
	Chickpea	2.0
Carbohydrate (Pasta)	Macaroni	4.0
	Shark pasta	1.0
	Chickpea	4.0
Glucide	Bread	4.0

some foods were changed on the basis of regional differences (Spain). The final synthetic waste composition is shown in Table 1.

(3) Mechanically selected municipal fraction (MS\_OFMSW) obtained from the *Municipal Treatment Plant "Calandrias"*, which is located in Jerez de la Frontera (Spain). The inoculum sources selected for use in reactor B for each process were as follows:

- (1) Mesophilic digested sludge (SLUDGE) obtained from the "Guadalete" Wastewater Treatment Plant, which is located in Jerez de la Frontera (Spain).
- (2) A mixture of digested SC\_OFMSW and swine excrement from a previous experiment under dry thermophilic conditions.

The initial physical and chemical characteristics of the three kinds of OFMSW and inoculum used are given in Table 2. The SC\_OFMSW and ST\_OFMSW are both high-solids substrates, with an average organic content of 71.4% and 73.5% (measured by VS), respectively – values that are significantly higher than that for MS\_OFMSW (44.1%). The majority of the total solids present in the SC\_OFMSW and ST\_OFMSW were volatile organic solids. The readily biodegradable organic matter present in food waste (75%) with a high moisture content enhanced the biological activity of these samples and demonstrated the viability of anaerobic digestion. The MS\_OFMSW contained a large amount of inorganic material, mainly from soil/sand and small inorganic particles.

The initial TOC values of SC, ST and SS\_OFMSW were 36.7, 70.8 and 14.8 g/L, respectively, and the initial TNK values were 18.0, 27.0 and 17.0 g/kg, respectively (Table 2). The COD:N ratios of the balanced leachates were 20.4, 28.4 and 9.5, respectively. The C:N ratio of the food waste was consistent with the range required for biological transformations and, in accordance with a study by Bouallagui et al. (2005), the COD:N ratio was around 100:4 for

#### Table 2

Initial mean characteristic of the organic wastes (residue) and initial leachate of the reactors

Analysis	SWINE	RESIDUE OFMSW			LEACHATE				
					Reactor_A			Reactor_B	
		Restaurant waste (SC)	Synthetic waste (ST)	Municipal waste (MS)	SC_OFMSW	ST_OFMSW	MS_OFMSW	SLUDGE	SC_OFMSW digest
Density (kg/m <sup>3</sup> )	1200	507	750	395	1015	1015	1010	1100	1050
Total solids (%)	57.0	83.6	90.8	82.8	3.4	3.2	4.9	4.3	12.0
Volatile solids (%)	53.0	71.4	73.5	44.1	1.4	1.9	2.4	2.1	9.0
pH	7.4	7.6	7.3	7.9	5.9	4.5	6.3	8.0	
Alkalinity (g/L)	0.5	0.5	0.7	0.5	0.3	0.2	0.9	0.6	0.9
N–ammonia (g/L)	0.2	0.8	0.4	0.2	2.4	1.6	3.7	1.6	3.0
TNK (g/kg)	14.0	18.0	27.0	17.0	28.0	33.0	47.0	61.0	29.0
Total acid (mg/L)	554.3	1920.0	1441.0	1974.7	1322.0	833.0	983.0	1902.0	1743
Phosphorus (g/kg TS)	0.4	1.9	2.3	4.2	1.2	0.8	0.9	2.0	1.3
TOC (g/L)	41.28	36.7	70.8	14.8	_	_	_	_	_
DOC(g/L)	_	_	_	_	88.7	129.4	54.3	34.4	42.4
COD(g/L)	49.3	34.0	76.7	16.3	83.3	116.5	74.2	47.8	49.0
C:N	35.2	20.4	28.4	9.5	29.7	35.3	15.7	7.8	17.0

fruit and vegetable waste. Indeed, the optimum C:N ratio for microbial activity in the bioconversion of vegetable biomasses to methane is 100–128:4 (Kivaisi and Mtila, 1998).

The digested SC\_OFMSW was a high-solids substrate with an average organic content of 9.0% (measured by VS) – a value that is significantly higher than that in SLUDGE (2.1%).

#### 2.3. Sample pre-treatment and reactor preparation

The samples were pre-treated using the protocol optimized in previous studies (Forster-Carneiro et al., 2004). The pre-treatment was carried out in order to improve the consistency of the SC\_OFMSW and SS\_OFMSW and to enhance the potential leachate. Small amounts of rice hulls or garden residues were mixed with SC\_OFMSW and SS\_OFMSW to enhance the leachate recycling process. Swine excrement (SWINE) was also mixed in to improve nutrient and microbe levels. Pre-treatment was not applied to the municipal solid waste from the treatment plant (MS\_OFMSW) because this material already had a good consistency.

A total of five LEACH systems were built for this study. The compositions of the LEACH systems are given in Table 3. LEACH 1 consisted of SC\_OFMSW (reactor A) and SC\_OFMSW and SWINE (reactor B). LEACH 2 consisted of SC\_OFMSW (reactor A) and SLUDGE (reactor B). LEACHs 3, 4 and 5 each consisted of OFMSW (reactor A) and SLUDGE (reactor B) to give a total of six reactors. Reactor A contained two waste materials in layers: OFMSW (SC\_OFMSW, ST\_OFMSW or MS\_OFMSW, respectively) and SWINE. Each LEACH system had two layers of OFMSW (SC\_OFMSW, ST\_OFMSW or MS\_OFMSW) (1.0 kg per layer) and two layers of swine (1.5 kg per layer). The layers were separated by a mesh (2 mm) with another mesh (15 mm) and a layer of glass balls located at the bottom of the reactor.

#### 2.4. Analytical methods

The parameters analysed for substrate characterization were as follows: Density, total solids (TS), volatile solids

(VS), fixed solids (FS), total suspended solids (TSS), volatile suspended solids (VSS), fixed suspended solids (FSS), pH, alkalinity, total nitrogen kjeldahl (TNK), total acid, ammonia nitrogen (N–NH<sub>4</sub>), total organic carbon (TOC) and chemical oxygen demand (COD). Daily analyses were performed on the leachates from reactors A and B: TS, VS, FS, COD, DOC, pH, alkalinity, N–NH<sub>4</sub>, and the biogas production and composition.

All analytical determinations were performed according to "Standard Methods" (APHA, 1989).

The alkalinity of each sample was determined using a COMPACT TITRATOR S+(Crison Instruments S.A.). The TOC and DOC analyses were carried out using a SHI-MADZU 5050TOC Analyser for combustion-infrared (5310B), again using "Standard Methods".

Gas produced in the reactor was collected in a 40 L Tedlar Bag, with biogas samples obtained on a daily basis and then analysed. The volume of biogas was measured directly using a WET DRUM TG 01 (mbar) high precision gas flow meter (Trallero and Schlee S.A.) through a CALI 5 BOND<sup>TM</sup> meter displacement bag (Trallero and Schlee S.A.). Gas composition analyses were carried out using a Tedlar bag. The biogas composition was analysed by gas chromatography (SHIMADZU GC-14B) using a stainless steel column packed with Carbosieve SII (3.2 mm diameter and 2.0 m length). A thermal conductivity detector (TCD) was attached to the system. The injected sample volume was 1 cm<sup>3</sup> and the operating conditions were as follows: 7 min at 55 °C; ramped at 27 °C min<sup>-1</sup> to 150 °C; detector temperature: 255 °C; injector temperature: 100 °C. The carrier gas was helium and the flow rate was  $30 \text{ mL min}^{-1}$ . A standard gas (from Carburos Metálicos S.A.) was used to calibrate the system and this had the following composition: 4.65% H<sub>2</sub>; 5.3% N<sub>2</sub>; 69.9% CH<sub>4</sub> and 20.1% CO<sub>2</sub>.

Total acid concentration was calculated by the addition of individual volatile fatty acid levels (VFA). The fatty acid levels were determined by gas chromatography – SHIMA-DZU GC-17 A equipped with a flame-ionization detector and capillary column filled with Nukol (polyethylene glycol modified by nitroterephthalic acid). The injection port and detector temperatures were 200 °C and 250 °C, respectively. Helium was the carrier gas with a flow rate of

Table 3 Composition of reactors A and B in the LEACH systems

LEACH systems	Composition					
	Reactor A	Reactor B				
LEACH 1	Two layer (SC_OFMSW + 15% RH) and two layer (SWINE + 15% RH)	SC_OFMSW digest				
LEACH 2	Two layer (SC_OFMSW + 15% RH) and two layer (SWINE + 15% RH)	SLUDGE				
LEACH 3	Two layer (SC_OFMSW + 15% RH) and two layer (SWINE + 15% RH)	SLUDGE				
LEACH 4	Two layer (ST_OFMSW + 15% RH) and two layer (SWINE + 15% RH)	SLUDGE				
LEACH 5	Two layer (MS_OFMSW + 15% RH) and two layer (SWINE + 15% RH)	SLUDGE				

 $50 \text{ mL min}^{-1}$ . The nitrogen flow rate was  $30 \text{ mL min}^{-1}$ . Total VFA was calculated by the addition of individual VFA levels.

#### 3. Results and discussion

# 3.1. Start up strategy and physical and chemical characteristics

A typical operational cycle for this technology is described in Fig. 1. The strategy is as follows: reactor B contains stabilized waste (inoculum sources) and is used to start the degradation of a fresh bed of waste in reactor A.

The layered mixture of OFMSW and SWINE in reactor A could enhance the fast start up phase of the dry anaerobic digestion process under thermophilic conditions in comparison to the reactor with OFMSW only. This configuration represents a significant modification of a conventional sequential leach-bed system in that the studies described in the literature to date did not involve the use of layers.

Five LEACH systems were investigated using this approach.

# 3.2. Performance of start up strategy in LEACHs 1 and 2: effect of the inoculum

The temporal evolution of the DOC concentrations of the leachate and the DOC removal percentages in LEACHs

1 and 2 are shown in Fig. 2a. The initial DOC concentrations were 49.9 and 16.6 g/L for LEACHs 1 and 2, respectively. The DOC values were found to decrease steadily after the first week. DOC removal values were similar in LEACHs 1 and 2 (approximately 35%) after 40 days.

The temporal evolution of pH and N–NH<sub>4</sub> during the start up phase is presented in Fig. 2b. The pH values of the leachates from LEACHs 1 and 2 were adjusted to about 7.5–8 in the first week with sodium hydroxide (6 N). After the first week, pH control was not necessary as the values were similar and remained constant until day 40 in LEACHs 1 and 2 (7.5 and 7.9, respectively). In contrast, the initial N–NH<sub>4</sub> concentrations in LEACHs 1 and 2 were 3.1 and 0.9 g/L, respectively, and these only became similar from day 15 onwards.

LEACH 1 showed a steady increase in accumulative biogas and methane production starting in the second week of the experiment (see Fig. 2c). The cumulative methane and mean biogas production levels after 30 days were 100.3 L and 8.7 L/day, respectively. In contrast, the cumulative methane and mean biogas production values in LEACH 2 were 255.4 L and 13.9 L, respectively, and these are the highest values of all the LEACH systems in the first 40 days of experimentation.

In this study LEACH 2 [with SC\_OFMSW (reactor A) and SLUDGE (reactor B)] reached a higher efficiency in the dry thermophilic digestion than LEACH 1 [with SC\_OFMSW (reactor A) and SC\_OFMSW digested with



Fig. 2. Reactor performance data: (a) DOC evolution and removal levels of the LEACHs 1 and 2; (b) pH and N–NH<sub>4</sub> levels of the LEACHs 1 and 2; (c) biogas and methane production and cumulative methane of the LEACH 1; and (d) biogas and methane production and cumulative methane of the LEACH 2.

SWINE (reactor B)]. The most notable results for LEACH 2 are the fast start up in the second day and the achievement of an initial stable phase after only 15 days. Under these conditions the performance of this system gave higher methane production (10.3 L/day) and higher VS removal (62.7%). On the basis of these results, digested mesophilic sludge was selected as the inoculum source for the subsequent assays.

# 3.3. Performance of the start up strategy in LEACHs 3, 4 and 5: effect of the nature of the MSW

The bioprocess conversion efficiency profiles with time for total and volatile solid concentrations are shown in Fig. 3. As can be seen, the initial solid concentrations in the leachate samples were 33.8, 31.6 and 49.0 gTS/kg and 14.4, 18.7 and 24.4 gVS/kg for LEACHs 3, 4 and 5, respectively. Both TS and VS values increased up to days 6, 10 and 14 for LEACHs 4, 5 and 6, respectively. This period corresponds to an acclimation stage in the leachate management strategy. In contrast, reactor B (SLUDGE) showed a stable performance from the first day, providing the microorganisms, moisture and nutrients required for the conversion of OFMSW to methane.

After the first week the total and volatile solid levels began to decrease. The VS removal values for LEACHs 3, 4 and 5 were 23.6%, 25.1% and 14.3%, respectively. The highest values reached for the total and volatile solids show that the solid removal percentages increased in all systems: LEACHs 3 and 4 increased by 50% and 55%, respectively, and LEACH 5 increased by 33.1%TS and 17.2%VDS removal.

These results show that between days 6 and 20 an acclimation stage (*acidogenic/acetogenic*) occurred and this is associated with the exponential phase of microorganism growth. The speed of growth of the bacteria corresponds to the reproduction time and their capacity to assimilate substrate. All systems showed this acclimation period



Fig. 3. Temporal evolution of total (TS) and volatile solids (VS) solids and removal percentage of total and volatile solids, in LEACHs 3, 4 and 5.

between the start up and stabilization (*methanogenic*) phases.

The temporal evolution of the DOC leachate concentrations and the DOC and COD removal percentages in LEACHs 3, 4 and 5 are shown in Fig. 4. The initial DOC concentrations were 82.5, 115.4 and 54.3 gDOC/L for LEACHs 3, 4 and 5, respectively. The DOC and COD concentrations in the reactors decreased steadily with time, apart from LEACH 5.

After 30 days the final DOC and COD concentrations in LEACHs 3 and 4 were half of the initial concentrations. The food wastes studied showed similar waste decomposition patterns and gave similar values of organic matter removal. In the case of LEACH 5, the DOC and COD removal values were similar (19.2% and 15.0%, respectively) but lower than those obtained for LEACHs 1 and 2 after 30 days of experimentation.

3.3.1. pH, alkalinity and ammonia variations in the leachate

The temporal evolution of pH, alkalinity and N–NH<sub>4</sub> during the start up phase is presented in Fig. 5. Initially, the pH of the leachates from LEACHs 1 and 2 were low (5.9 and 4.5, respectively) compared to that of LEACH 3 (pH 6.3). The pH values decreased to 5.0 and 3.8, respectively, in the first week for LEACHs 3 and 4. It is well known that the progress of rapid decomposition can be slowed by changes in the pH – this phenomenon is due to the sensitivity of methane bacteria to low pH values (Bolzonella et al., 2003b). The control of pH with sodium hydroxide (6 N) was necessary in the first week (except for LEACH 5).

In general, all LEACH systems showed appropriate alkalinity and ammonia levels to maintain a stable pH in the digester for optimal biological activity. The pH and alkalinity levels suggest that LEACH 5 had a higher



Fig. 4. Temporal evolution of dissolved organic carbon (DOC) and removal percentage of dissolved organic carbon (DOC) and chemical oxygen demand (COD) in LEACHs 3, 4 and 5.



Fig. 5. Variation of pH, alkalinity and ammonia nitrogen levels in the leachate samples of LEACHs 3, 4 and 5 for 30 days in each experiment.

buffering capability. The ammonia concentration varied only during the first stage, coinciding with the hydrolytic phase that fundamentally involves the hydrolysis of proteins. Different types of behaviour were observed for food waste and municipal waste in terms of the evolution of N–NH<sub>4</sub>. In the case of the food waste N–NH<sub>4</sub> had a small influence on the methanogenic activity but for municipal waste this factor had a much more marked influence. LEACH 5 could be inhibited by N–NH<sub>4</sub> concentrations of around 3500 mg/L.

The LEACH process allows the undigested soluble organic matter in the reactor to be transferred little by little to the digested reactor, and this leachate strategy contributes to methanogenic bacteria. In this study the leachate strategy allowed the stabilization of the LEACH 3 and 4 systems in less than 30 days. However, in the case of LEACH 5 this situation was not achieved.

According to Ahring et al. (1995), butyrate and isobutyrate concentrations increased significantly 1 or 2 days after the imposed perturbation, which makes these acids suitable for process monitoring and important for process control of the anaerobic biological system. In this work, all parameters (pH, N–NH<sub>4</sub>, total VFA/alkalinity ratio and total VFA) proved adequate to maintain a stable process. In addition, the total VFA results indicate strong microbiological activity that is translated into a sharp increase in the acidity of the means favoured by the hydrolysis of the organic compounds. Significant increases in the concentrations of butyrate and isobutyrate were not detected in this work.

# 3.3.2. Comparative anaerobic performance by gas composition and production

A comparison of the anaerobic performance for LEACHs 3, 4 and 5 is presented in Fig. 6 for the start up anaerobic process. In all of the LEACHs investigated here, deoxygenation occurred within about three days after start up. The daily generation of biogas in LEACHs 3 and 4 increased slowly in the first 20 days but LEACH 5 showed different behaviour. LEACH provided a good level of biogas production during all of the experiments.

After 30 days the mean biogas production levels obtained in LEACHs 3, 4 and 5 were 3.2, 2.2 and 2.2 L/ day, respectively (Fig. 6a). The highest mean biogas production between days 20 and 30 was obtained in LEACHs

1 and 2 (6.3 and 4.3 L/day) and the lowest in LEACH 5 (1.7 L/day). Similar results were obtained for the percentage of methane in the total biogas produced. After 30 days the methane percentages obtained in LEACHs 3, 4 and 5 were 36.0%, 32.0% and 23.4%, respectively (Fig. 6b).

The evolution of cumulative methane production in LEACHs 3, 4 and 5 can be seen in Fig. 6c. The cumulative methane production from LEACHs 3 and 4 followed a similar trend – in both reactors the production increased quickly after day 20, which is the end of the acclimation period and initial methanogenic phase.

VS reduction, total methane production and methane yield can be used as criteria to judge the success of an anaerobic digestion process. LEACHs 3 and 4 showed higher VS reduction and biogas production and, furthermore, LEACH 3 showed higher methane production in the biogas. In terms of the cumulative methane production,



Fig. 6. Comparative anaerobic performance in gas characteristics in the LEACHs 3, 4 and 5, after 30 days of experiment: biogas production (a); cumulative methane (b); and methane percentages (c).

LEACH 5 showed the highest values after 30 days. In terms of the specific methane yield, LEACH 5 showed a lower global effectiveness (0.14 L CH<sub>4</sub>/gCOD) in comparison to LEACHs 1 and 2 (0.23 and 0.24 L CH<sub>4</sub>/gCOD, respectively) (Table 4).

According to Fernandez et al. (2001), during the start up for solid waste treatment the two-phase anaerobic system is much better to optimize methane production. Our studies suggest that the use of the double phase process for the source-sorted organic fraction of municipal solid waste alone could give more stable conditions (Pavan et al., 2000). However, SEBAC processes guarantee system stability with a built-in mechanism to prevent imbalance and there is also a leachate management strategy (or leachate recirculation) to enhance the degradation of solid waste. Recent studies show that the performance of SEBAC on OFMSW is 0.30 L CH<sub>4</sub>/gVS and the biochemical methane potential data for different feedstocks is between 0.05 and 0.15 L CH<sub>4</sub>/gVS and 0.15 and 0.25 L CH<sub>4</sub>/gVS during the stabilization phase. These results are consistent with those reported in Table 4.

The final results suggest different behaviour patterns for these two wastes: (1) the MS\_OFMSW showed a methanogenic pattern throughout the whole experiment (6–30 days) (with higher methane production) and (2) the SC and ST\_OFMSW showed two stages: an acidogenic/acetogenic

Table 4 Methane yield of LEACHs 3, 4 and 5 after 30 days and 90 days of each experiment

	Methane yield			Organic matter removal (%)			
	CH <sub>4</sub> /VS (L/g)	CH <sub>4</sub> /COD (L/g)	CH <sub>4</sub> /DOC (L/g)	TS (g/L)	VS (g/L)	COD (g/L)	DOC (g/L)
30 days							
LEACH 3	0.17	0.23	0.21	21.6	23.6	49.3	51.0
LEACH 4	0.15	0.24	0.20	24.1	25.1	58.0	47.6
LEACH 5	0.14	0.10	0.15	20.8	14.3	15.0	19.2
90 days							
LEACH 5							
Reactor A (MS OFMSW)	0.22	0.21	0.24	51.0	67.2	79.8	62.4
Reactor B (SLUDGE)	0.30	0.34	0.21	46.9	61.9	54.0	33.6



Fig. 7. Reactor performance data of the LEACH 5: (a) total solid (TS) and volatile (VS) solid evolution and removal levels; (b) DOC evolution and DOC and COD removal; (c) pH, alkalinity and  $N-NH_4$  levels; and (d) biogas and methane characteristics.

phase in the range 5–20 days and a subsequent methanogenic phase. The different patterns seen in the two processes could be due to the inoculum percentage used, since the biogas production rate increased at the same rate as the inoculum percentage increased.

#### 4. MS\_OFMSW system performance

All three LEACH systems were designed for the comparative study of the start up and stabilization phases. The leachate recycle configuration employed by Chynoweth et al. (1992) enabled the bioconversion of biodegradable organics in less than 30 days and, for this reason, each experiment was carried out for 30 days. The LEACH 5 experiments lasted 90 days in order to understand the biodegradation and performance of the reactor.

The control parameters of the LEACH 5 leachate are shown in Fig. 7. As can be seen in Fig. 6a, both TS and VS values (g/L) showed a steady decrease from day 15. The highest VS removal efficiency was 67.2% at the end of the experiment. LEACH 5 showed similar organic matter efficiency expressed as VS or COD removal (Fig. 7b). The evolution of DOC only starts from day 28 and this decreased to 20.4 g/L. At the end of the process, DOC and COD removals were 62.4% and 79.8%, respectively. It can be concluded that the MS\_OFMSW biodegradation was highly effective; nevertheless, this biodegradation occurred later than in LEACHs 3 and 4. LEACHs 3 and 4 presented TOC and COD removal values of around 50% after 30 days but for LEACH 5 this level was only achieved after 84 days.

A suitable alkalinity or buffer capacity is necessary to maintain a stable pH in the digester for optimal biological activity. The pH, alkalinity and ammonia evolution levels in the leachate for LEACH 5 are shown in Fig. 7c. In general, LEACH 5 did not show suitable alkalinity (1.3 g/L) or ammonia levels (3.4-2.0 g/L), but it was possible to maintain a stable pH in the range 6.3-8.3 without pH control.

LEACH 5 showed a steady increase in cumulative biogas and methane production starting from the second day of the experiment (Fig. 7d). The cumulative biogas and methane production levels at the end of 90 days were 79.9 L and 22.7 L, respectively, and these are the highest production rates of all the LEACHs in the first 30 days of experimentation. Finally, the mean methane yield of LEACH 5 was 0.21 L CH<sub>4</sub>/gCOD (Table 4).

The results of this study show that the dry thermophilic system LEACH 5 could be inhibited by ammonia in the first 30 days. In the stabilization phase the methane production was higher than the organic biodegradation (after day 40). These results suggest a methanogenic pattern during the whole experimental period.

### 5. Conclusions

Digested mesophilic SLUDGE was selected as an inoculum source because it exhibited the best performance (methane production of 10.3 L/day and VS removal of 62.7%).

The biomethanization processes for three organic wastes [separately collected food waste from a restaurant (SC\_OFMSW), synthetic waste (ST\_OFMSW) and mechanically selected municipal waste (MS\_OFMSW)] were studied in LEACH systems under thermophilic and dry conditions. The results obtained show that all three organic wastes studied exhibit the classical waste decomposition pattern with a fast start up phase beginning within 0–5 days, an acclimation stage (acidogenic/acetogenic phases) between days 5 and 20–30 and a subsequent stabilization phase.

However, different decomposition patterns were observed between two types of waste (food and municipal waste): (1) the MS\_OFMSW showed a methanogenic pattern throughout the whole experimental period (the methane production was superior to the organic biodegradation) and (2) the SC and ST\_OFMSW showed a methanogenic pattern only in the stable phase (after 20 days) and this gave higher levels of organic biodegradation and methane production. Under these conditions LEACHs 3 and 4 gave a VS removal of around 50% (or approximately 50% DOC removal) and methane yields of 0.23 and 0.24 L CH<sub>4</sub>/gCOD, respectively. In contrast, LEACH 5 gave only low organic carbon and solid removal values.

After 90 days, the main results were approximately 80% COD removal and a methane yield of 0.21 L CH<sub>4</sub>/gCOD.

The data obtained confirm that the modified sequential leach-bed system used, under dry thermophilic conditions and with the organic waste mixed with swine digest waste and rice hulls arranged in layers, improved the performance of the anaerobic process and enabled the treatment of municipal solid urban waste of different origins. The process was complete and a high level of methane production was achieved in less than 30 days. Furthermore, the mesophilic sludge provided a suitable inoculum source for the three classes of OFMSW studied.

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#### References

- Ahring, B.K., 1992. Turn-over of acetate in hot springs at 70 °C. In: Proceedings of Thermophilies: Science and Technology, Reykjavik-Iceland. 130.
- Ahring, B.K., 1994. Status on science and application of thermophilic anaerobic digestion. In: Seventh International Symposium on Anaerobic Digestion, Cape Town-South Africa. 328–337.

- Ahring, B.K., Sandberg, M., Angelidaki, I., 1995. Volatile fatty acids as indicators of process imbalance in anaerobic digestors. Appl. Microbiol. Biotechnol. 43, 559–565.
- APHA-American Public Health, Association. American Water Works Association, Water Environmental Federation, 1989. Standard Methods for the examination of Water and Wastewater. 17th ed. Washington, DC.
- Angenent, L.T., Sung, S., Raskin, L., 2002. Methanogenic population dynamic during starup of a full scale anaerobic sequencing batch reactor treating swine waste. Water Res. 36, 4648–4654.
- Bolzonella, D., Innocenti, L., Pavan, P., Traverso, P., Cecchi, F., 2003a. Semi-dry thermophilic anaerobic digestion of the organic fraction of municipal solids waste: focusing on the start up phase. Bioresource Technol. 86, 123–129.
- Bolzonella, D., Battistoni, P., Mata-Alvarez, J., Cecchi, F., 2003b. Anaerobic digestion of organic solid wastes: process behaviour in transient conditions. Water Sci. Technol. 48 (4), 1–8.
- Bouallagui, H., Touhami, Y., BenCheikh, R., Hamdi, M., 2005. Bioreactor performance in anaerobic digestion of fruit and vegetable wastes. Proc. Biochem. 40, 989–995.
- Cecchi, F., Pavan, P., Mata-Alvarez, J., 1992. Fast digester start up under mesophilic conditions using thermophilic inoculum. Water Sci. Technol. 25, 391–398.
- Chugh, S., Chynoweth, D.P., Clarke, W., Pullammanappallil, P., Rudolph, V., 1999. Degradation of unsorted municipal solids waste by a leach-bed process. Bioresource Technol. 69, 103–115.
- Chynoweth, D.P., Owens, J., O'Keefe, D., Earle, J.F.K., Bosch, G., Legrand, R., 1992. Sequential batch anaerobic composting of the organic fraction of municipal solid waste. Water Sci. Technol. 25, 327– 339.
- Chynoweth, D.P., Owens, J.M., Legrand, R., 2001. Renewable methane from anaerobic digestion of biomass. Renew. Energ. 22, 1–8.
- Chynoweth, D., Haley, P., Owens, J., Rich, E., Townsend, T., and Choi, H., 2002. Anaerobic composting for recovery of nutrients, compost, and energy from solid wastes during space missions, Int. Conf. Environ. 01, 2351.
- De Baere, L., 2000. Anaerobic digestion of solids waste: state of the art. Water Sci. Technol. 41, 283–290.
- De la Rubia, M.A., Pérez, M., Martínez, A., Romero, L.I., Sales, D., 2001. Comparative study of mesophilic and thermophilic anaerobic digestion of sludge. In: The 9th International Conference on Anaerobic Digestion. Amberes-Netherlands. 103–106.
- Fernandez, B., Porrier, P., Chamy, R., 2001. Effect of inoculum-substrate ratio on the start up of solid waste anaerobic digesters. Water Sci. Technol. 44 (4), 103–108.
- Forster-Carneiro, T., Fernandez Guelfo, L.A., Pérez García, M., Romero García, L.I., Álvarez Gallego, C.A., 2004. Optimization of start up phase from municipal solids waste in SEBAC process. Chem. Biochem. Eng. Q. 18, 429–439.
- Hobson, P.N., Wheatley, A.D., 1993. Anaerobic Digestion Modern Theory and Practice. Elsevier Science Publishers, Ltd., Essex, U.K.

- Kim, M., Ahn, Y., Speece, R.E., 2002. Comparative process stability and efficiency of anaerobic digestion: mesophilic vs. thermophilic. Water Res. 36, 4369–4385.
- Kivaisi, A.K., Mtila, M., 1998. Production of biogas from waster hyacinth in a two stage bioreactor. World J. Microbiol. Biotechnol. 14, 125–131.
- Kulik, A., 1997. Anaerobic digestion gains ground in Europe. World Wastes J. 9–15.
- Lissens, G., Vandevivere, P., De Baere, L., Biey, E.M., Verstraete, W., 2001. Solid waste digestors: process performance and practice for municipal solid waste digestion. Water Sci. Technol. 44 (8), 91–102.
- Macé, S., Bolzonella, D., Mata-Álvarez, J., 2005. Full scale implementation of AD Technology to treat the organic fraction of municipal solid waste in Spain. In: 4th International Symposium Anaerobic Digestion of Solid Waste. Copenhagen (Denmark), 1, 409–416.
- Martin, D.J., Potts, L.G.A., Reeves, A., 1999. Small-scale simulation of waste degradation in landfills. Biotechonol. Lett. 19, 683–686.
- Mata-Álvarez, J., Macé, S., Llabrés, P., 2000. Anaerobic of organic solids wastes. an overview of research achievements and perspectives. Bioresource Technol. 74, 3–16.
- Nopharatana, A., Clarke, W.P., Pullammanappallil, P.C., Silvey, P., Chynoweth, D.P., 1998. Evaluation of methanogenic activities during anaerobic digestion of municipal solids waste. Bioresource Technol. 64, 169–174.
- O'Keefe, D., Chynoweth, D.P., Barkdoll, A.W., Nordstedt, R.A., Owens, J.M., Sinfontes, J.R., 1993. Sequential batch anaerobic composting of municipal solids waste (MSW) and yard waste. Water Sci. Technol. 27, 77–86.
- O'Keefe, D., Chynoweth, D.P., 2000. Influence of phase separation, leachate recycle and aeration on treatment of municipal solid waste in simulated landfill cells. Bioresource Technol. 72, 55–66.
- Paredes, C., Cegarra, J., Bernal, M.P., Roig, A., 2005. Influence of olive mill wastewater in composting and impact of the compost on a Swiss chard crop and soil properties. Environ. Int. 31, 305–312.
- Pavan, P., Musacco, A., Cecchi, F., Bassetti, A., Mata-Alvarez, J., 1994. Thermophilic semi-dry anaerobic digestion process of the organic fraction of municipal solids waste during transient conditions. Environ. Technol. 15, 1173–1182.
- Pavan, P., Battistoni, P., Traverso, P., Musacco, A., Cecchi, F., 1998. Effect of addition of anaerobic fermented OFMSW (organic fraction of municipal solids waste) on biological nutrient removal (BNR) process: preliminary results. Water Sci. Technol. 38, 327–334.
- Pavan, P., Battistoni, P., Mata-Alvarez, J., Cecchi, F., 2000. Performance of thermophilic semi-dry anaerobic digestion process changing the feed biodegradability. Water Sci. Technol. 41 (3), 75–82.
- Pullammanappallil, P.C., Chynoweth, D.P., Lyberatos, G., Svoronos, S.A., 2001. Stable operation of anaerobic digestion under high concentrations of propionic acid. Bioresource Technol. 78, 165–169.
- SEBAC homepage, 2005. </www.agen.ufl.edu/~sinfontes/sigarca>.
- Tchobanoglous, G., Hilary, T., Vigil, S.A., 1997. Integrated solids waste management: engineering principles and management issues. Published by Mc Graw-Hill, Inc.