

# Energy recovery from biowaste: influence of hydraulic retention time on biogas production in dry-anaerobic digestion

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**Abstract.** The hydraulic retention time (HRT) is a key parameter in dry-anaerobic digestion to set during the reactor configuration in order to achieve the optimal biogas production. For this reason, the study compared the results of two experimental tests operating with an HRT of 23 and 14 days. During the tests, the feedstock was organic fraction of municipal solid waste with a solid content of 33% and the digester was a pilot-scale plug-flow reactor operating in thermophilic condition. The highest specific biogas production of  $311.91 \text{ Nl}_{\text{biogas}} \text{ kg}^{-1} \text{ TVS d}^{-1}$  was achieved when the HRT was set to 23 days. On the contrary, the highest methane production rate of  $1.43 \text{ Nl}_{\text{CH}_4} \text{ l}^{-1} \text{ d}^{-1}$  was achieved for an HRT of 14 days. In addition, the volatile solids removal (49.15% on average) and the energy content ( $4.8 \text{ MJ kg}^{-1} \text{ TVS}$  on average) were higher for HRT 23 days than for HRT 14 days. The results indicated that in dry-anaerobic digestion of organic fraction of municipal solid waste, 23 days is a suitable HRT for energy recovery.

## 1 Introduction

In European Union (EU), anaerobic digestion (AD) is a largely spread process to convert biowaste in bio-energy [1]. Furthermore, AD achieves the goals of EU policies promoting Bioeconomy [2] and Circular Economy [3]. Indeed, biogas can be upgraded to biomethane [4] while the liquid residue can become a more valuable product as compost, biopolymer or bioplastic [5,6].

Dry-AD process refers to biowaste with high solid content ( $>20\%$ ) and high organic loading rate (OLR). Among the biowaste, the Organic Fraction of Municipal Solid Waste (OFMSW) has the above-mentioned characteristics and is easy-available substrate [7]. However, its variability in time (season) and space (different waste collection area and system) is one of the main critical issue to overcome for the success of the process [7,8]. To overcome this problem, OFMSW can be treated in different types of reactors: vertical, horizontal, operating in continuous or in batch mode are used [9]. Usually, dry-AD digesters operate in thermophilic conditions ( $55^\circ\text{C}$ ) to accelerate the kinetic of the biological process, but in case of failure need longer time to recover than wet digesters [10]. The OLR, the sludge retention time (SRT), the hydraulic retention time (HRT) and methane ( $\text{CH}_4$ ) yield are the main process parameters to addressed to achieve the optimal working condition of a digester.

Focusing on the HRT, high values lead to maximum TVS removal. For example, a TVS removal efficiency of 74.8% was reached in a digester operating in mesophilic conditions [11]. On the contrary, in the co-digestion of food-waste and cattle manure, an HRT of 5 days led to the highest  $\text{CH}_4$  production while an HRT of 15 days showed the maximum  $\text{CH}_4$  yield [12].

Many researchers studied the influence of HRT on the biogas production, but in dry-AD this issue need to be deeper investigated [10]. Consequently, this study investigates the influence of the HRT on the biogas production aiming at maximizing its energy content. To achieve this goal, the performances of a pilot-scale plug-flow reactor (PFR) were compared testing two HRT: 23 and 14 days.

## 2 Materials and methods

### 2.1 Reactor design and set-up

The pilot-scale PFR was developed by DESTEC and DIF and was realized by Cavalzani Srl. The digester was made of stainless steel, with a cylindrical form and a total volume of 37 L. A water jacket with a thickness of 54 mm, a height of 163 mm and a total volume of 7 L. A hydraulic pump (PQm 90, Pedrollo SpA) recirculated water from a water bath to the water jacket. A digital immersion heater thermostat (FA 90, Falc

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Instruments Srl) heated the water tank to ensure a temperature of 55°C.

The substrate was fed to the reactor by means of a cylinder and a piston. A lock nut and a sealing ring provided a gas tight system and avoided the entrance of air during the feeding operation. Once the substrate filled the cylinder, the piston was operated manually, and the substrate was pushed inside the PFR. In this area, a set of blades, with a hollow extreme and jointed on a horizontal shaft, continuously mixed the feedstock and the digestate. The mixing system homogenised along the cross-section of the PFR; while the digestate was transported towards the outlet only by the entrance of the daily feedstock. The shaft was jointed to a gearmotor (3GAA092540-CSJ, ABB Spa and W75 U D30 40 P90 B14 B3, Bonfiglioli Spa) whose speed was regulated to 8 rpm through an inverter (FVR1.5AS1S-7E, Fuji Electric CO., Ltd). In the outlet zone, the digestate was discharged from the main body opening a ball valve (2"), while the biogas left the PFR from a gas fitting located in the upper part. A volumetric counter measured the daily biogas production. The PFR and the volumetric counter were linked by a 3-2 ways direct-acting solenoid valve (type 6014, Burkert Spa). After passing the solenoid valve an infrared sensor (Gascard NG, Edinburgh Sensors, UK) monitored continuously the concentration of CH<sub>4</sub> in the biogas.

In the central area, two sensors (InPro 4281i, Mettler-Toledo S.p.A.) of pH and temperature were installed to control the process stability.

Hardware and software platforms (National Instruments, Italy Srl) were developed to automatically control the process.

## 2.2 Inoculum and feedstock

The inoculum was obtained from a full-scale anaerobic digester operating in thermophilic conditions and treating OFMSW (Asja Impianti, Foligno PG, Italy). Before fill-in the PFR, the inoculum was screened with a sieve opening size of 10 mm to remove plastics, small twigs, or inert materials and to avoid damages to the mixing system.

The foodwaste (FW) was a mix of OFMSW and Green Waste (GW). OFMSW was collected from a district in which a door-to-door (DDR) collection system was applied. To separate the non-biodegradable fractions (i.e. plastics, bones, inert materials, textile, metal, glass, cardboard) from the kitchen and yard waste on a sample of around 260 kg a picking analysis was performed. The impurities were removed, and a sample of 150 kg was shredded and was characterized as described in section 2.4. GW was obtained from the same collection district, was shredded, was sieved at 1 mm and was characterized as described in section 2.4. OFMSW and GW were stored at -19°C to prevent further biodegradation before usage. Table 1 illustrates the initial characteristics of the substrates. The FW was the same throughout the experimental period.

**Table 1.** Initial characteristics of the feedstocks.

	IN	OF-MSW	GW	FW

TS [%]	18.9±0.3	28.8±0.5	45.1±0.1	31.9±0.1
TVS/TS [%]	51.7±0.1	87.4±1.7	88.5±1.1	68.1±0.1
pH [-]	8.4±0.1	5.12	5.74	5.1±0.3
Bulk Density [g/ml]	1.01±0.1	1.05±0.04	0.31±0.02	0.92±0.05
TOC [%] *	5.5±0.83	9.9±1.5	20.9±3.1	14.85±2.22
N [%] *	0.76±0.09	0.52±0.06	0.48±0.06	0.51±0.06
C/N [-]	7±0.7	19±1.9	43±4.3	29.8±5.5

\*dry basis

## 2.3 Experimental runs

Table 2 reports the design of experiment. In literature, many studies report dry-AD digesters operating at 60 to 13 days of HRT [13,14]. From one side, high HRT correspond to high specific gas production (SGP) and process stability [13,15]. From the other side, a low HRT leads to low SGP and process stability. In the light of these evidences and considering a potential full-scale applicability the PFR operated at 23 days (Run1) and 14 days (Run2) of HRT. As consequence, the inlet feeding flow (Q<sub>in</sub>) was obtained dividing the digester working volume by the HRT and the OLR was calculated dividing the volatile solid content of the substrate by the digester working volume.

**Table 2** Experimental runs: operational parameters.

	Run1	Run2
HRT [d]	23	14
Q <sub>in</sub> [l/d]	1.22	2.00
OLR [kg <sub>grvs</sub> m <sup>-3</sup> d <sup>-1</sup> ]	7.24	12.18

Before the tests began, 28 L of inoculum was degassed to remove biodegradable matter [16]. After 7 days a stable biogas production was reached and consequently the bacterial consortia achieved the endogenous metabolism. At that time, the continuous feeding started. Firstly, Run1 was studied and then Run2 was applied.

In both runs, the PFR was fed every working day and the same quantity of digestate was removed to maintain a steady state. Daily, OFMSW and GW were defrosted and mixed in a ratio of 0.34 kg<sub>GW</sub>/kg<sub>OFMSW</sub> to achieve a final total solid (TS) content of 33%. Finally, the substrate was manually homogenized. To inoculate the FW a recirculation ratio of 0.3 was defined.

For each run, was considered to achieve a steady state after a transient period corresponding to one HRT. Each HRT condition was kept for a period corresponding to two times the HRT.

Daily, TS, total volatile solid on dry basis (TVS/TS), and pH were determined on FW and withdrawn digestate.

## 2.4 Analytical procedures

Moisture content and TS were determined gravimetrically after drying the samples for 24 h at 105°C. TVS/TS was used as index of the organic matter in the feedstock and was determined gravimetrically after burning the dried samples for 3 h at 550°C. pH was evaluated using a pH meter (pH 7 +DH2, XS Instruments) applying a ratio of 10 g of sample to 100 ml of deionized water [17]. Total organic carbon (TOC), Nitrogen (N) and C/N ratio were evaluated in an external laboratory in accordance to EN 15407:2011 and UNI EN 13137:2002 + UNI EN 15407:2011 respectively.

Bulk density of the feedstocks and the digestate was determined modifying a methodology proposed by [18]. A 3 L carafe was fill-in with the FW until a capacity of 2L was reached. At this time, the carafe was left to fall for 20 times from a height of approximately 10 cm. Then, the weight and the volume of the sample within the carafe was measured and the bulk density was calculated dividing the weight by the volume.

The volumetric biogas production was measured using a stainless-steel volumetric counter developed at DIF-UNIFI. Accordingly to [19] the variation of the liquid height inside the volumetric counter was directly related to the volume of biogas. The liquid height was set to 6.77 cm and was monitored using a set of three electrodes (072.500, Finder SpA). Once a week, a biogas sample was collected to be analysed by gas-chromatography (Micro GC Gas 180 Analyzer, INFICON, Basel, Switzerland) to determine the concentration of CH<sub>4</sub>, carbon dioxide (CO<sub>2</sub>), nitrogen (N<sub>2</sub>), oxygen (O<sub>2</sub>) and hydrogen sulphide (H<sub>2</sub>S).

## 2.5 Performance Evaluation

The performances were evaluated considering the stability of the process: pH and CH<sub>4</sub> concentration in the biogas (% v/v); the biogas productivity as SGP (NL<sub>biogas</sub> kg<sup>-1</sup><sub>TVS</sub> d<sup>-1</sup>), the specific methane production (SMP, in NL<sub>CH<sub>4</sub></sub> kg<sup>-1</sup><sub>TVS</sub> d<sup>-1</sup>), and the reduction efficiency of the total volatile solids (RE<sub>TVS</sub>, in %). More in detail RE<sub>TVS</sub> was calculated using the following equation:

$$RE_{TVS} = (TVS_{in} - TVS_{out}) / TVS_{in} \quad (1)$$

Where TVS<sub>in</sub> was the total volatile solid content of the FW and TVS<sub>out</sub> was the total volatile solid content of the outlet digestate.

In addition, the energy content of the biogas was calculated by multiplying the SMP and the lower heating value (LHV) of CH<sub>4</sub> that was assumed to be 34 MJ m<sup>-3</sup>.

## 3 Results and Discussion

### 3.1 Inoculum and feedstock characterization

Table 1 reports the most important characteristics of the inoculum and the feedstocks used in the experimental runs.

In AD, the optimal value for C/N ratio ranges from 20 to 30 [20]. However, a value of 32 resulted the best to reduce ammonia toxicity during the anaerobic process [15]. Concerning the inoculum, the C/N ratio was slightly higher than those reported in previous study [11]; while the other feedstocks showed higher C/N ratio than the inoculum. The OFMSW showed a C/N ratio slightly lower than those typical of a DDR collection system [8]. However, considering the mixing ratio of 0.34 kg<sub>GW</sub>/kg<sub>OFMSW</sub> the final FW achieved a good C/N ratio.

To the best of our knowledge, studies on methodologies to determine the bulk density of waste and specifically of OFMSW are lacking. Values ranging from 0.9 to 0.6 g cm<sup>-3</sup> were found by [18]. In this study, the average bulk density of the FW was 0.92 ± 0.05 g ml<sup>-1</sup> (n=67).

### 3.2 Process performance evaluation

#### 3.2.1 Biogas production

Table 3 resumes the results of the experimental tests at different HRT. Data were reported as averages over the stationary period. The results of the degassing phase are not shown, but before starting the feeding operation, the digestate in the intermediate sections had an average pH of 7.70 ± 0.02, a stable biogas production of 4.66 NL d<sup>-1</sup> with a CH<sub>4</sub> content of 56.38%.

Focusing on Run1, the average pH of the steady phase was lower than those of the transient period (results not shown), but the values were always in the optimal range for dry-AD [9] and near the neutrality as also shown in an analogous studies [15,21]. This result showed an intrinsic buffer capacity of the system. On the contrary, CH<sub>4</sub> content and biogas production were lower than those expected considering analogous C/N ratio and OLR [15]. Focusing on CH<sub>4</sub> concentration, a study introducing an OLR of two times lower than those applied in Run1 found similar CH<sub>4</sub> concentration and the author detected the acidification of the reactor [22]. For this reason, the low CH<sub>4</sub> concentration in biogas was not only caused by the high OLR. Consequently, among the other factors affecting the process the high GW and TS content of the feedstock could have been the main contributors to this low yield. Firstly, GW introduced lignin that is a slowly biodegradable matter and can accumulate in the reactor especially at high loading rate [11]. In fact, the lignin content in the digestate increased from 25.41% in dry basis to 38.07 % on dry basis after 26 days of continuous feeding. In addition, the increment of the TS content from 20% to 30% can reduce the methane yield of 17% [23]. With regard to the other parameters, the highest SGP was 311.91 NL<sub>biogas</sub> kg<sup>-1</sup><sub>TVS</sub> d<sup>-1</sup>; while the highest SMP was 175.58 NL<sub>CH<sub>4</sub></sub> kg<sup>-1</sup><sub>TVS</sub> d<sup>-1</sup>.

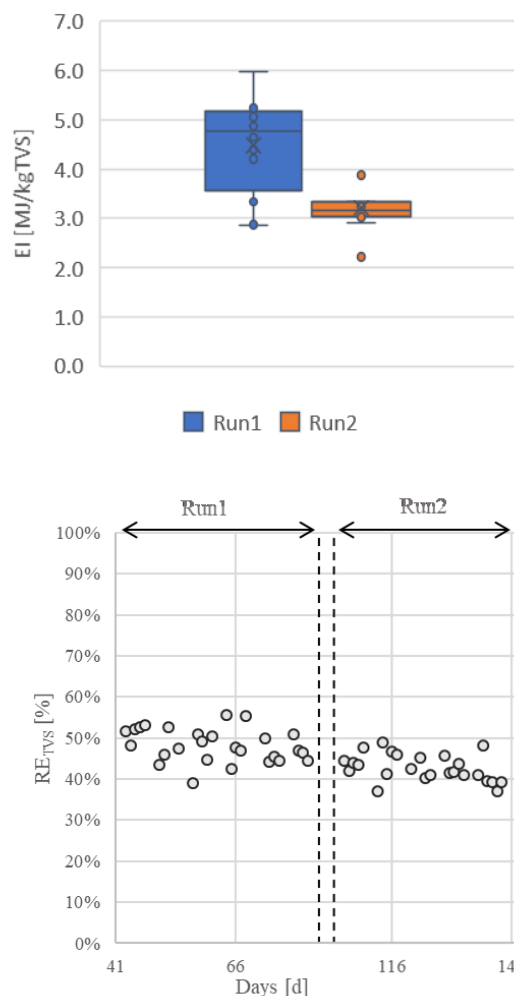
**Table 3.** Results of the experimental tests: Run1 and Run2

	Run1	Run2
pH [-]	7.47 ± 0.08	7.73 ± 0.06
Biogas Production [NI d <sup>-1</sup> ]	51.65 ± 9.26	67.29 ± 6.10
SGP [NIbiogas kg <sup>-1</sup> TVS d <sup>-1</sup> ]	240.57 ± 66.71	190.57 ± 14.4
CH <sub>4</sub> [%]	57.71 ± 3.58	55.75 ± 2.49
SMP [NICH <sub>4</sub> kg <sup>-1</sup> TVS d <sup>-1</sup> ]	104.98 ± 44.22	94.23 ± 13.90
MPR [NICH <sub>4</sub> l <sup>-1</sup> d <sup>-1</sup> ]	0.85 ± 0.26	1.11 ± 0.2
OLR [kgTVS d <sup>-1</sup> ]	7.49 ± 1.92	12.60 ± 0.45
RE <sub>TVS</sub> [%]	48.81 ± 4.81%	42.51 ± 3.09%
EI [MJ kg <sup>-1</sup> TVS]	4.48 ± 0.93	3.20 ± 0.47

\*n=3 for SGP, CH<sub>4</sub>, EI and OLR

Moving to Run2, Table 3 shows the results of the first month of stationary state. The intermediate pH resulted higher than those observed in Run1 because was added new inoculum to maintain a constant working volume. By comparison, the biogas production increased by 27.31% when HRT decreased from 23 to 14 days; while CH<sub>4</sub> concentration was comparable. On the other hand, SGP and SMP slightly decreased indicating a too short retention time for the complete degradation of the organic matter within the FW. Indeed, the RE<sub>TVS</sub> decreased by 13.63%. On the contrary, the methane production rate (MPR, NI<sub>CH<sub>4</sub></sub> L<sup>-1</sup> d<sup>-1</sup>) increased by 30.54% showing a CH<sub>4</sub> yield higher than those achieved shorting the HRT from 25 days to 5 days and using cattle manure and food waste as substrate [12].

Fig. 1 shows the trend of the volatile solids removal efficiency of the systems during the steady state. The dashed lines separate the two stages and the results of the transient state is not showed. More in detail, Run1 showed the highest RE<sub>TVS</sub>, but a high standard deviation was highlighted. However, the RE<sub>TVS</sub> seems to have a negative trend in the last part of the steady period especially when HRT was 14 days. Thus, could indicate an accumulation of lignin or non-biodegradable matter inside the reactor because of the short retention time. However, the biological process was stable overall the trials.



**Fig. 1.** Trend of RE<sub>TVS</sub> during the experimental tests.

Despite of the low values of RE<sub>TVS</sub>, the results achieved in the trials was higher than those reported both in mesophilic [11] and thermophilic conditions [22] for previous study on dyr-AD using a PFR as reactor for the biological process.

### 3.2.2 Energy content

Table 3 reports the average energy content of the biogas applying different HRT to the reactor; while Fig.2 depicts the EI data using a box plot graph.

**Fig. 2.** Energy content of the biogas with different HRT.

Although the average EI of the biogas in Run1 resulted 28% higher than those of Run2. However, the

standard deviation revealed a high degree of dispersion of the data in Run1 that is also indicated by distance between the different parts of the box in Fig.3. This result was less evident in Run2. The data showed a negative correlation between the HRT and the EI of the biogas. This results confirms the evidences already highlighted in previous research [15,22] indicating that the optimization of biogas production and energy content is achieved operating at high retention time.

### 3.3 Conclusion

The study investigated the influence of HRT on biogas production in dry-AD of OFMSW for energy recovery. During the experimental tests, the biogas production and the methane production rate increased by 27.3% and 30.54% respectively when the HRT decreased from 23 to 14 days. On the other hand, the specific biogas production decreased by 10.7% indicating an uncomplete degradation of the volatile solids within the substrate when the HRT became shorter. In addition, this result was reflected by a low value of volatile solids efficiency (42.51%). Finally, the energy content is maximum when HRT was 23 days and increased by 31.1% in respect to HRT 14 days

Summarising, Run1 showed the highest specific biogas production, methane yield and energy content for dry-AD of OFMSW. Further investigations are ongoing to deeper investigate the process: volatile fatty acids, biochemical methane potential tests and respirometric tests will be done on the digestate.

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