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SCALE EFFECT OF ANAEROBIC DIGESTION TESTS IN FED-BATCH AND SEMI-CONTINUOUS MODE FOR THE TECHNICAL AND ECONOMIC FEASIBILITY OF A FULL SCALE DIGESTER

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Abstract

Methane production capacity in mesophilic conditions of waste from two food industry plants was assessed in a semi-pilot (6L, fed-batch) and pilot (300L, semi-continuous) scale. This was carried out in order to evaluate the convenience of producing heat and electricity in a full scale anaerobic digester. The pilot test was performed in order to obtain more reliable results for the design of the digester. Methane yield, returned from the pilot scale test, was approximately 80% of that from the smaller scale test. This outcome was in line with those from other studies performed in different scales and modes and indicates the success of the pilot scale test. The net electricity produced from the digester accounted for 30% to 50% of the food industry plants' consumption. The available thermal energy could cover from 10% to 100% of the plant requirements, depending on the energy demand of the processes performed.

Keywords

Mesophilic anaerobic digestion

Fed-batch tests

Semi-continuous tests

Vegetable processing waste

Cost-benefit analysis

1. INTRODUCTION

Italian food industry, with a sales volume of 130 G€, is the second most important industry after the car manufacturing. The Italian food industry plants buy and transform 72% of domestic agricultural raw materials. Inevitably, the processes performed in food industries generate huge amounts of agro-industrial waste. For Italy, Petruccioli et al. (2011) reported productions of 2.4 Mt/y of grape pomace, 0.7 Mt/y of olive pomace and 0.2 Mt/y of both tomato pomace and soybean integuments. It can be estimated that the transformation of 1 t of agricultural raw materials generates from 30 to 100 kg of organic waste.

Agro-industrial waste, that includes both fruit and vegetable waste, may be stabilized by the anaerobic digestion (AD) process (Khalid et al., 2011). AD is known as a more environmentally friendly and energy saving process for stabilizing high-strength organic waste, than other disposal options like landfilling, incineration, and composting (Hosseini Koupaie et al., 2014; Traversi et al., 2013). In addition to biogas generation, AD benefits include enhancing nutrient characteristics of the dewatered digestate used as fertilizers as well as pathogens reduction. Greenhouse emissions are also decreased because the AD process diverts organic waste from landfills, thus preventing uncontrolled methane and carbon dioxide emissions from decomposition (Xie et al., 2011).

In spite of the potential advantages of the AD process for the management of agro-industrial waste, at present, in Italy, most of it is sent to composting plants. This work aims to carry out a technical and economic assessment of a more sustainable way of managing waste from food industry processes, by evaluating the convenience of producing heat and electricity in an on-purpose made anaerobic digester. The waste products considered in this work are generated in two food industry plants located in the NW Italy.

In order to proceed to the technical and economic assessment of the digester, the potential production of methane from the waste generated in the two plants was determined in both a semi-pilot (6-L, fed-batch mode) and pilot (300-L, semi-continuous mode) scale in mesophilic conditions. Values of the methane yield for waste originated from agro-industrial processes are reported in several studies (Dareioti and Kornaros, 2014; Garcia-Peña et al., 2011; Jiang et al., 2012; Yang et al., 2013). However, only a couple of very recent papers focus on tests carried out on fruit and vegetable waste fed as a single substrate to semi-continuous pilot scale reactors (Fiore et al., 2013; Scano et al., 2014).

In order to test the capacity of waste to produce methane in anaerobic conditions, different modes (batch, semi-continuous, continuous) and several scales (from lab to full scale) can be used. Traditional biochemical methane potential (BMP) tests have the undeniable advantage of taking up small volumes (usually from 100 mL to 1,000 mL) and requiring very low efforts of personnel during test developments (Angelidaki et al., 2009; Cavaleiro et al., 2013; Lisboa and Lansing, 2013; Raposo et al., 2011; Triolo et al., 2012). However, the procedure does not assure reliable outcomes in the case of highly heterogeneous substrates and it is not suitable to highlight problems such as the irreversible acidification of the reactor (Kolbl et al., 2014).

In this work a first estimate of the methane production capacity of waste was obtained using fed-batch tests in a semi-pilot scale. The authors preferred fed-batch tests to traditional one-fed essays and employed larger vessels than usual (6 L instead of maximum 1 – 1.5 L) in order to test a substrate with high heterogeneity using a solid procedure. The fed-batch mode allowed the authors to feed higher amounts of substrate, guaranteeing the representativeness of the whole sample and avoiding possible inhibition in the methanogenesis phase due to the progressive acidification of the reactor. In addition, because of the effect of particle size and surface area of the substrate on the performance of the AD process (Novarino and Zanetti, 2012; Palmwoski and Muller, 2000; 2003; Zhang and Banks, 2013), the larger volume allowed the authors to test highly heterogeneous substrates with realistic particle sizes.

The capacity of waste to produce methane obtained in the fed-batch mode was verified using a semi-continuous test carried out in the 300-L digester. The pilot scale test was necessary in order to obtain more reliable results on which to base the design of the full scale digester for the energy valorization of the waste products. The results of tests in the pilot scale are of particular interest especially for highly heterogeneous substrates. The outcomes of the experimentation were employed for a preliminary process and cost-benefit analysis of the full-scale AD digester for treating the waste produced in the two food industry plants.

2. MATERIALS AND METHODS

2.1 Waste origin and characterization

Waste products considered in this work are generated in two food industry plants located in the NW Italy. The first plant (Plant 1) transforms and preserves several kinds of vegetables, mainly carrots, potatoes, onions, beetroots and celery with a capacity of 12,000 t/y. Processes carried out in Plant 1 generate around 350-400 t/y of organic waste. The organic waste from Plant 1 has a very high heterogeneity, because it comes from both the peeling process of vegetables and grating and cloth-filtering phases in the wastewater treatment plant (WWTP) contained within the food industry plant. The second food industry plant (Plant 2) specializes in the production of pesto sauce and other kinds of sauces with an end product capacity of 5,000 t/y. Waste is made of residues of production and sub-standard products, the main components of which are basil and sunflower oil. The monthly amount of organic waste is approximately of 25-30 t. Plants with a production capacity similar to that of the plants studied, have electrical consumption in the order of 1,000 - 1,500 MWh/y and thermal consumption that ranges from $5 \cdot 10^2$ to 10^4 MWh/y, depending on the heat demand of the operations performed.

Two substrates coming from Plant 1 and Plant 2 were sampled, characterized and employed in the AD tests. The substrate from Plant 1 was a mixture of three waste products. The first waste product (named as “peeling”) originated from the peeling process of vegetables. The second waste product (named as “screen”) was separated from the grid screen positioned at the head of the WWTP contained within the food industry plant. The third waste product (named as “filtrate”) was separated from the cloth filter positioned after the grid screen in the same WWTP. The produced amounts of the three waste products, on dry basis, were the same. For this reason the mix to employ for the tests was obtained by mixing identical amounts of each waste product (on dry basis).

The substrate from Plant 2 came from the production process of pesto sauce. According to the data supplied from the Plant, the principal components of this waste were basil and sunflower oil, in approximately the same amount. Minor components were cashew nuts, pine nuts and Parmesan cheese.

The physical and chemical characterization of substrates and the digestate, that resulted from the digestibility tests, included pH, total solid (TS) and volatile solid (VS) content, and elemental composition (C, H, N, S – this last analysis only for substrates). All parameters were determined according to standard methods (APHA, AWWA, WEF, 2005). The elemental analysis was performed by means of a CHNS-O Thermo Fisher Flash 2000 Analyzer EA 1112, assuming the oxygen content as the complementary fraction towards C, H, N, S contents. All the analyses were carried out in three replicates, on representative and significant amounts of the samples as in Roati et al. (2012). Determination of VS content and elemental composition were carried out on samples dried at 105°C.

2.2 Semi-pilot scale test – fed-batch mode

Both substrates, that is the vegetable mix waste (VMW) and the waste from pesto sauce production (PSW, pesto sauce waste), were tested in a fed-batch mode. Tests were performed in mesophilic conditions (35°C) in five replicates, using 6-L poly methyl methacrylate (PMMA) digesters placed in a thermostatic bath. The anaerobic environment was prepared by filling digesters with water, then replacing it with nitrogen. This procedure also ensures that the reactors were leak free. In order to simulate real digestion conditions, pH value was not adjusted and no nutrients were added. Each digester was manually mixed for 20–30 s once a day as in Ruffino et al. (2015).

The inoculums employed in the tests was prepared from 1,000 mL of digestate collected from the anaerobic digesters of a municipal WWTP located in the same area of the two food industry plants. The inoculums was progressively fed with amounts of primary sludge, coming from the same

WWTP, to reach a final volume of 3,000 mL. The inoculums was considered ready when its daily biogas production was of less than 1% of the overall production recorded throughout the period of preparation.

For the test with the VMW, the inoculums had a TS content of 3.26 ± 0.31%, a VS/TS ratio of 64.3 ± 2.9% and a pH value of 7.23 ± 0.01 (average value on five replicates ± standard deviation). Each digester was fed with six aliquots of the substrate. Each aliquot contained 15 g of TS and was introduced into the digesters at days 0, 1, 2, 6, 8 and 9 from the beginning of the test. The final TS content was 3% of the working volume (3 L).

For the test with the PSW, the inoculums had a TS content of 4.24 ± 0.21%, a VS/TS ratio of 75.1 ± 0.6% and a pH value of 7.18 ± 0.01 (average value on five replicates ± standard deviation). Each digester was fed at days 0, 1, 5, 7, 8 and 12 with six aliquots of the substrate, each of them contained 14 g of TS. Differences in fed frequency between test 1 and 2 were due to the limitation in the maximum daily biogas collection capacity (10-12 L) and depended on the biogas production rate. However, also for Test 2, the final TS content in the digester was approximately 3%.

For each replicate, the produced biogas was collected in two 5-L Tedlar® bags connected in parallel. The characterization and measure of the biogas volume was carried out daily, throughout the entire duration of the test. The characterization, that is the volumetric composition of the biogas in terms of CH_4, CO_2, O_2 and “balance” (the fraction made up of gases that are different from the first three, that is mainly nitrogen, hydrogen, carbon monoxide and volatile organic compounds), was obtained by flushing 500 mL of biogas through a biogas analyzer (Biogas Check, Geotechnical Instruments Ltd). The volume was measured by replacing volumes of water with biogas and referring the obtained value to standard conditions.

2.3 Pilot scale test – semi-continuous mode

The pilot scale test involved only the VMW from Plant 1. This substrate was chosen because of its higher heterogeneity compared to the other waste.

The AD plant employed for the test was equipped with a 300-L reactor (240 L working volume), an

80-L gasometer and a system for on-line monitoring of the biogas composition. The temperature was automatically monitored and regulated by means of a resistance temperature sensor (Pt 100) positioned along the reactor and connected to the heating system. The biogas collection system included a condensation trap for moisture removal and a flow-meter. The pilot plant was controlled by a PLC system that receives signal from the different sensors and drives the main electrical and pneumatic pieces of equipment. Substrates inside the digester were mixed 15 minutes every hour by a system of re-circulating biogas. The other physical and chemical parameters required for an effective monitoring of the AD process (TS, VS content, pH and FOS-TAC values) were daily determined on the sampled digestate.

The inoculums for the test was prepared by mixing 80 L of digestate, collected from the digesters of the WWTP as in Section 2.2, and 160 L of primary sludge from the same plant. The inoculums was considered ready to start the test when its daily biogas production was of less than 1% of the overall volume produced throughout the period of preparation. At the beginning of the semi-continuous test the inoculums had a TS content of 2.22 ± 0.01%, a VS/TS ratio of 62.7 ± 0.2 and a pH value of 7.4 ± 0.1 (average value on 3 replicates ± standard deviation).

The TS content of the substrate fed to the reactor was fixed to 3%, in order to make possible a comparison between the performances of the two digestion systems (semi-pilot and pilot scale). For the same reason the test was carried out in mesophilic conditions (35°C). The hydraulic retention time (HRT) was fixed to 30 days. This value came from the results (trend of biogas and methane production and hydrolysis constant) obtained for the VMW tested in a fed-batch modality, see Section 3.2.

The volume of the fed substrate was of 8 L with a TS content of 240 g and resulting OLR of 1 kgTS/m^3·d or 0.87 kgVS/m^3·d. Due to the characteristics of the waste product (that is an average TS content of approximately 8% and a VS/TS ratio of 86.8%, see Section 3.1), the daily feedstock was prepared by mixing 2,200 g of the VMW with 5,800 mL of tap water. The AD test had an overall duration of 54 days that were necessary for the loading of 30 volumes of substrate and the subsequent complete depletion of the biogas produced. The feeding of the substrate and the extraction of an equal volume of digestate were carried out in days from Monday to Friday, according to the procedure described as in the follow:

1. recording of volume and composition (methane, CO_2, oxygen, balance and H_2S) of the biogas produced since the previous recording;
2. extraction of 8 L of digestate to undergo to pH, TS, VS, FOS-TAC analysis;
3. loading of 8 L of substrate, prepared as previously described.

3. RESULTS AND DISCUSSION

3.1 Waste characterization

The characteristics of the substrates involved in the digestibility tests are shown in Table 1. The VMW had an average TS content of less than 10% b.w. (by weight), a VS/TS ratio close to 90%, an acidic pH value and a elemental composition of C: 45% b.w., H: 6-7% b.w., N: 2-3% b.w., S: 0.3-0.5% b.w., on dry basis, with a subsequent C/N ratio of approximately 15. A visual test revealed that the sizes of particles ranged from few millimeters (mainly residues from the peeling process) to about 5 centimeters (pieces of vegetables and discarded mushroom caps).
The PSW was far more homogeneous than VMW. It had the texture of a sauce with a TS content of approximately 70% b.w. and a VS/TS ratio close to 100%. pH value was determined only with pH strips because of the high oily content of the waste product. For the same reason the determination of the elemental composition was not performed.
The molecular formula of the first substrate was obtained from its elemental composition. The molecular formula of the second substrate was obtained from the average composition of a general vegetable ($C_{18}H_{30}O_{13.5}N$, as for the first substrate), that simulates basil, and sunflower oil ($C_{17.9}H_{32.8}O_2$). The ratio between basil, as dry matter, and sunflower oil was assumed equal to 1:1 b.w.

3.2 Semi-pilot scale test

Both fed-batch tests performed on the two waste products had an overall duration of 22 days. According to 1% criterion of German Guideline VDI 4630 (2006), the tests were stopped when the daily biogas production dropped to values of less than 1% of the total gas production.

The daily evolution of the biogas produced by the VMW is shown in Figure 1. Figure 1a shows two peaks at days 2-3 and 9-10 from the beginning of the test. In those days the daily biogas production rose to volumes respectively of 4.5 and 5.5 NL. The trend of the daily biogas production showed a strong dependence on the feeding rate. The total amount of the fed waste (90 g of dry substance in 6 aliquots) determined an overall biogas production that was between 40 and 47 NL. The five replicates returned a biogas specific production of 0.554 ± 0.038 Nm^3/kgVS added (average value on five replicates ± standard deviation), a methane specific production of 0.294 ± 0.029 Nm^3 CH_4/kgVS added, with a subsequent average methane content of 53.0 ± 1.8% b.v. (by volume). The composition of the biogas produced in the days in which the record of volume and composition was carried out, is shown in Figure 1b. The average carbon dioxide content was of 32.6 ± 1.5%. Biogas did not show any traces of H_2S. The daily pH check did not highlight critical situations such as the irreversible deviation of pH value toward acidic conditions (data not showed).

The digestate that resulted from the test carried out on the VMW had a TS content of 3.21 ± 0.14%, a VS/TS ratio of 66.2 ± 1.6% and a pH value of 7.10 ± 0.02. A TS depletion of 67.5% and a VS depletion of approximately 73% were calculated from the balance that involved the inoculums, the digestate and the fed substrate (1046.5 g wet substrate with a TS content of 8.60%, 90 g TS, 78.1 g VS).

With reference to the second substrate (PSW), the daily evolution of the biogas produced is shown in Figure 2. Figure 2a shows two peaks at days 10 and 13 from the beginning of the test. In those days the daily biogas production rose to volumes respectively of 9.5 and 8 NL. Unlike the case described above, the trend of the daily biogas production for the PSW did not show a strong dependence on the feeding rate. The total amount of the fed substrate (84 g of dry substance in 6 aliquots) determined an overall biogas production that was between 82 and 92 NL. The five replicates of the semi-pilot scale test returned a biogas specific production of 1.08 ± 0.05 Nm^3/kgVS added (average value on five replicates ± standard deviation), a methane specific production of 0.722 ± 0.033 Nm^3 CH_4/kgVS added, with a subsequent average methane content of 66.9 ± 0.2% b.v. The composition of the biogas produced in the days in which the record of volume and composition was carried out is shown in Figure 2b. The average carbon dioxide content was of 29.8 ± 2.1%. As for the previous substrate, the biogas produced did not show any traces of H_2S and the daily pH check did not highlight critical situations (data not showed).

With reference to the second tested substrate, the digestate had a TS content of 3.88 ± 0.21%, a VS/TS ratio of 72.7 ± 0.9% and a pH value of 7.24 ± 0.02. A TS and VS depletion close to 100% was calculated from the balance that involved the inoculums, the digestate and the fed substrate (121.1 g wet substrate with a TS content of 69.3%, 84 g TS, 82.3 g VS).

A balance that involves carbon was carried out for the two substrates in order to compare the amount of carbon in the feedstock with the amount of carbon that was converted to methane or carbon dioxide (average amount on the five replicates). The results are detailed in Table 2. It can be seen that the AD process performed in a fed-batch mode converted 56% of the carbon from the VMW and 92% of the carbon from the PSW to methane and carbon dioxide. These results are in line with the results that concern TS and VS depletion.

In order to compare the obtained specific productions of biogas and methane with the maximum production predicted by theoretical calculations, according to Buswell and Neave model (Tchobanoglous et al., 1993), it is necessary to refer to the molecular formula of the two substrates, as reported in Table 1. According to this model, the VMW subjected to AD would give rise to a biogas specific production of 0.848 Nm^3/kgVS added and a methane specific production of 0.424 Nm^3/kgVS added (as listed in Table 3). The observed values were respectively only 65.3% and 69.3% of the values predicted by Buswell and Neave model. On the other hand, the theoretical biogas specific production of the PSW was of 1.14 Nm^3/kgVS added, 5% more than the value returned from the fed-batch test.

With reference to the afore-described results, the most important differences in the behavior of the two substrates (VMW and PSW) were observed:

- in the specific production values of biogas and methane. The biogas specific production of the VMW substrate was approximately 50% of that of the PSW substrate. On the other hand, the methane specific production of the VMW was 40% of that of the PSW;
- in the response of the AD systems to the feedstock introduction. Reactors fed with the VMW substrate showed a fast production of biogas and methane. Reactors fed with the PSW substrate showed a production of biogas and methane more intense than that from the VMW, but only after a phase of initial delay;
- in the conversion rate of the fed carbon to methane and carbon dioxide, that was in the order of 56% for the VMW substrate and greater than 90% for the PSW substrate;
- in the ratio between the biogas and methane specific production values obtained from the fed-batch tests and those from Buswell and Neave model.

The afore-mentioned differences could be attributed to the composition and particle size distribution of the two substrates. The VMW substrate was composed solely of vegetable matrix, rich in fiber and lignin, therefore refractory to biological degradation. Due to the structure and particle sizes of the VMW substrate, the fraction available for biological degradation must first be subjected to the processes of disintegration and hydrolysis. It is therefore unlikely that an accumulation of reaction intermediates occurs and slows the course of the AD process.

On the other hand, the second substrate (PSW) showed a considerable oil content and very small particle sizes. Fatty acids from oil and soluble COD liberated from the vegetable matrix, following the size reduction occurred in pesto sauce preparation, were readily available to AD. However, according to Batsone et al., 2002, the maximum removal rate of fatty acids (6 d^{-1}) is one order of magnitude lower than that of sugars and amino acids (the simplest substances that compose vegetable matrices, respectively equal to 30 and 50 d^{-1}). This may explain the delay observed in the first phase of the test, that was due to the low removal rate of fatty acids. Moreover, the composition of the second substrate may explain the higher values of specific production of biogas and methane, compared to those observed for the VMW, and the high ratio between the experimental value and the theoretical value. These last observations came from the higher presence of completely degradable substances (fatty acids, and available amino acids and sugars from the vegetable matrix) in the PSW substrate than in the VMW.

The characterization of waste products from Plant 1 and Plant 2 was completed through the evaluation of the disintegration rate constant (Batstone et al., 2002). The disintegration rate constant describes the kinetics of the first of the two processes (i.e. disintegration and hydrolysis) that make up the solubilization phase. In fact, according to Batstone et al., 2002, the disintegration process is commonly the limiting phase of the whole AD process when there is no accumulation of intermediary products. According to Angelidaki et al., 2009, using the first part of the experimental curve build for the determination of the ultimate methane production of a given substrate, it is possible to define the constant for a first order model as in Equation 1:

$$ln\frac{B_\infty - B}{B_\infty} = -kt \qquad (1)$$

where B_∞ is the value of the ultimate biogas production and B is the biogas production at a given time, t. The method described as in Angelidaki et al., 2009 for the interpretation of results from BMP tests in a batch mode, was here adapted for the results obtained from tests carried out in a fed-batch mode. The parameter value for k was estimated using non linear least squares curve fitting of the net cumulative methane production (see Figure 3).

Curve fitting returned values of 0.382 d^{-1} and 0.254 d^{-1} respectively for the VMW and the PSW. However, as it can be seen in Figure 3, the model employed is suitable for describing the trend of the biogas production from the VMW, but it demonstrated not to be adequate for the PSW. In this second case, the disintegration process cannot be considered the limiting phase of the overall AD process because of the high amount of organic acids from sunflower oil. A possible accumulation of organic acids in the first days of the test could have made the AD process slower than predicted by the model.

The values found for the two substrates are in line with the values of the same parameter found for organic substrates that originates from vegetables, as reported in Galí et al., 2009; Giuliano et al., 2013; Shi et al., 2014. In those works the authors found k values that ranged from 0.2 to 0.4 d^{-1} and Giuliano et al., 2013 reported values of 0.26 d^{-1} and 0.34 d^{-1} respectively for potatoes and onions when BMP tests were carried out in mesophilic conditions.

3.3 Pilot scale test

Only the VMW was involved in the pilot scale test. The daily production of biogas and the daily specific production of biogas and methane are shown in Figure 4a. Values of production of the days in which the recording of data (volume, biogas composition, see Figure 4b) was not performed (Saturdays, Sundays), were obtained dividing the value recorded the first day after the break by the duration of the break. The diagram of Figure 4a shows that the biogas daily production had a fluctuating trend, that ranged from 50 NL/d to 100 NL/d. The lowest values were registered in the two days that ensued the feeding stops. The highest values were registered when the loading of the substrate was made. The value of the methane specific production, after showing considerable fluctuations until around day 20 from the beginning of the test, stabilized at 0.223 Nm^3/kgVS added. The values of the two parameters FOS and TAC showed a trend that decreased from the beginning of the test to the end. However, the ratio between FOS and TAC kept constant in the range 0.2 - 0.3 (see Figure 5a), optimum for an AD process. The daily record of the TS content into the digestate showed a considerable decrease in the first ten days from the beginning of the test, and, then, a subsequent fluctuation around the constant value of 1.50% from day 10 (see Figure 5b). At the end of the test the amount of TS removed was of 55% and the amount of VS removed in the order of 65%. This value was only slightly lower than that found in the fed-batch test (73%). As shown in Table 3 the methane specific production obtained in the semi-continuous test was approximately 76% of that obtained in the fed-batch test and the difference in the average methane content between the two tests was of less than 20%.

The ratio between the methane specific production returned from the two tests (batch vs. semi-continuous) performed in this study was compared with that from other studies in order to verify its consistency. Unfortunately, to the knowledge of the Authors, very few studies report the results of tests carried out on different scales and modes on the same substrate. Moreover, some of them were carried out on very small scale (Kafle and Kim, 2013; Zhang et al., 2013). In the cases reported and discussed as in the follow, the amount of dry substrate fed to both batch and semi-continuous (or continuous) reactors was similar, so as to make possible a comparison on the digestion performances between the different modes and scales.

Zhang et al. (2013) performed tests in both batch and semi-continuous modes employing reactors with the identical active digestion volume of 1 L. The aim of their study was to test a dual solid-liquid system for the digestion in mesophilic conditions of food waste that contained considerable amounts of organic substance in the liquid phase. The methane yields obtained in semi-continuous mode was between 60% and 80% of the values obtained in the batch mode.

Kafle and Kim (2013) tested mixtures of apple waste and swine manure in both batch (1.8 L liquid volume) and continuous modes (4.5 L liquid volume). The outcomes of the tests carried out in the two modes on a mixture that contained 33% apple waste were compared. The methane yield from the continuous test increased, from the first to the second phase of the experimentation, from 182 to 241 mL CH_4/gTCOD added. The methane yield registered in the second phase was approximately 90% of the value returned from the batch test (267 mL CH_4/gTCOD added).

Zupanĉiĉ and Jemec (2010) evaluated the capacity of several kinds of tannery waste to producing methane, in both mesophilic and thermophilic conditions, using BMP tests carried out in 1,160 mL glass vessels. Tests were repeated in semi-continuous mode in a 30-L digester obtaining specific methane productivity values between 79 and 93% of those obtained in the BMP tests. The ratio between the volume of the digester employed for the semi-continuous tests and that of the reactors for the batch tests was approximately of 30, a value of the same order of magnitude of that of this study (80). For this reason, in spite of the kind of the tested substrates (fleshings, skin trimmings and wastewater sludge), the results of the present study have to be considered consisted with those by Zupanĉiĉ and Jemec.

Wang et al. (2014) investigated the anaerobic digestion performances of kitchen waste and fruit/vegetable waste from lab-scale (working volume of 1.5 L) to pilot-scale (2 and 4 m^3), including batch, single-phase and two-phase experiments. The methane yields obtained on a pilot-scale (0.64 LCH_4/gVS) was about 80% of that obtained on a lab-scale (0.725 L CH_4/gVS) under similar VS loading values.

Moreover, a study performed in the same apparatus of the present study and in the same operating conditions, on a mix of rice hull and rice bran, returned a ratio of approximately 0.81 between the methane specific production obtained on a semi-continuous mode (0.312 Nm^3/kgVS added) and the methane specific production obtained on a fed-batch mode (0.386 Nm^3/kgVS added).

On the contrary, Jard et al. (2012) obtained results which are in contrast with those of the above-discussed studies. They tested the biogas production of two kinds of microalgae (*Palmaria Palmata* and *Saccharina Latissima*) in both batch (500 mL) and semi-continuous reactors (3 L). Methane yields obtained in the semi-continuous reactor were around 25% higher than those from the BMP essays. According to the authors, AD typically produces higher yields of methane when run as a continuous rather than a batch process due to the progressive adaptation of the microbial population.

In spite of the results of this last case, usually batch tests returned methane yields higher than those obtained in a semi-continuous mode. This is obviously due to the longer contact time between substrate and microbial population, that allows microorganisms to completely degrade substrates, and to the better mixing conditions achieved in batch reactors (especially if of small size). It can be concluded that tests in a batch (or fed-batch) mode may provide a good estimate of the capacity of a substrate to be degraded in anaerobic conditions and are useful to assess if further tests with higher-scale reactors may be justified. Batch tests performed on a scale higher than usual assure reliable outcomes in the case of high heterogeneity of the substrate, thus guaranteeing the representativeness of the whole sample and avoiding the necessity of reducing sizes. However, the assessment of a full scale reactor for waste energy valorization requires solid methane yield values that can be obtained only from a pilot scale test. In agreement with previous literature studies, the outcomes of this work demonstrated that a successful pilot scale test returned methane yields between 70 and 80% of the values from batch tests.

3.4. Energy valorization

The results obtained in the tests described in Sections 3.2 and 3.3 were employed for a preliminary technical and economic evaluation of a full-scale AD reactor for the energy valorization of the waste products generated in Plant 1 and Plant 2. The methane specific production for the VMW was assumed equal to 0.223 Nm^3/kgVS added, the value returned by the semi-continuous test. For the PSW the methane specific production was assumed equal to 0.578 Nm^3/kgVS added, that is 80% of the value from the fed-batch test. According to the remarks set out in Section 3.3, this value could be considered acceptable for the design of a full scale plant. The two feeding substrates are generated at a constant rate throughout the year. Their characteristics are also nearly constant.

The full scale plant is based on a single-stage reactor that operates in mesophilic condition with an HRT of 30 days. HRT was set equal to the value employed in the semi-continuous test. Because the dry substance fed to the digester is supposed not to exceed 10% (the highest value allowed in an AD wet process), the PSW has to be diluted with water. From a balance that takes into account the amount and the TS content of the PSW and the TS content allowed in the substrate fed to the digester, the yearly amount of water required for dilution was equal to approxiametey 2,200 m^3, that is 6 L/kg waste. The produced biogas feeds a combined heat and power (CPH) unit based on an internal combustion engine integrated with a heat recovery section. The assumptions concerning the electrical and thermal efficiencies of the CHP unit were based on typical values for commercial units. The main data for the design of the full scale digester are listed in Table 4.

The main outcomes that result from the energy balance applied to the digester are listed in Table 5. The overall volume of the digester was calculated taking into account the daily amount of fed substrate, the HRT and the digester filling coefficient. The density of the fed substrate (10% TS) was fixed equal to 1,000 kg/m^3, then the daily amount of the fed substrate resulted to be equal to approximately 8 m^3 and the overall volume of the digester of 300 m^3.

From the lower heating value (LHV) of the methane generated from the AD process, a first estimate of the daily methane production suggested a CHP unit with a power output of 60 kW. The gross electrical and thermal yields were calculated starting from biogas production and electrical and thermal efficiencies of the CHP unit. The net electrical energy production was obtained by subtracting the internal electrical energy consumption from the gross electrical energy production. The internal electrical energy consumption was assumed to be equal to 10% of the gross production. The available thermal energy was obtained by subtracting the thermal energy requirements of the AD process and the heat losses with the outside from the gross thermal energy production. The thermal energy requirements of the AD process were calculated as the amount of heat necessary to raise the temperature of the fed substrate (yearly average, 15°C) to the temperature of the AD process (35°C). The ratio between the thermal energy requirement of the AD process and the gross thermal energy production resulted to be of about 10%. Taking into account the geometry of the full-scale digester, the materials employed for construction, the temperature of the fed mixture (15°C), soil (15°C) and outside (exterior environment, as monthly averages as reported in the UNI 10349 rule), it may be assumed that the amount of thermal energy to balance the thermal losses is of the same order of magnitude of the thermal energy requirement of the AD process (i.e. $7 \cdot 10^4$ kWh/y).

The annual net electrical energy production of the digester, equal to 470 MWh, was enough to cover from 30% to 50% of the electrical consumption of a food industry plant with a size similar to that of the plants that produced the waste products considered in this study. On the other hand, the available thermal energy (500 MWh/y) may cover from 10% to near 100% of the plant requirements depending on the energy demand of the processes performed.

On the full-scale digester designed as described above, a preliminary economic analysis was carried out. Two different scenarios that refer to the use of the electrical energy generated only by the AD process were considered. In the first scenario the electricity was employed for the internal energy requirements of the food industry plant and no public subsidies were obtained. In the second scenario the electricity was dispatched to the national grid, in agreement with the current Italian legislation (DM 6/07/2012) on energy production from renewable sources. In fact, the most recent Italian policy supports the use of biological by-products deriving from agricultural and food industries as substrate for AD power plants, especially of small sizes (from 1 to 300 kWe). The fee-in tariff of 236 €/MWh is the maximum allowed for the electricity generated in AD power plants.

With reference to both scenarios, the total annual costs (C_T) born by the plant for the production of electricity from the AD process was calculated as in (2)

$$C_T = TFC \cdot CCR + C_{O\&M} \quad (2)$$

where:

- TFC, total fixed costs, are the costs for the implementation of the digester power plant;
- CCR, capital charge rate, calculated as in (3), where (i) is the annual interest rate and (n) the operating lifetime;
- $C_{O\&M}$, annual operating and maintenance costs.

$$CCR = \frac{i}{1-(1+i)^{-n}} \quad (3)$$

For the economic assessment carried out in this work, the annual interest rate was fixed to 7% and the operating lifetime to 20 years (as stated by DM 6/07/2012). The TFC was calculated as the sum of the costs of digester, CPH unit and installation. Plant unit capital cost investment were fixed to 6,000 €/kWe. Since the electrical power of the plant was of 60 kWe and the costs for installation were fixed to 100,000 €, the resulting TFC was approximately of 450,000 € and, consequently, the annual depreciation 42,500 € (see Table 6).

The annual operating and maintenance costs were assumed to be equal to 4% of the TFC, that is 18,000 €. The total labor costs were fixed to 15,000 €, considering 0.5 workers employed on the plant. Costs for utilities, that is raw materials supply, treating and restoring water and waste disposal, were assumed to be equal to 4% of the value of the electrical energy (see EER, Table 6), if dispatched to the national grid as required by Scenario 2. The income from the sale of electrical energy, as in Scenario 2, was calculated multiplying the annual net electrical energy produced (469 MWh) by the fee-in tariff of 236 €/MWh and resulted of approximately 110,700 €/y. The total amount of the annual operating costs was then equal to approximately 37,500 €.

With reference to the first scenario, the equivalent Cost of Energy (CE), as in (4), was calculated through the ratio between the total annual costs (C_T) and the annual net electrical energy production:

$$CE = \frac{C_T}{E_N} \quad (4)$$

The economic analysis applied to the first scenario returned an equivalent electrical energy production cost for the full-scale AD reactor of 172 €/MWh, which is lower than the purchase cost of electrical energy for industrial consumers (200 €/MWh, Scano et al., 2014).

The second scenario takes into account the option of delivering the electrical energy produced by the AD reactor to the national grid, according to the current Italian legislation (DM 6/07/2012). In this case, the economic analysis was carried out with the same assumptions (interest rate, operating lifetime, TFC, operating and maintenance costs) employed for the assessment of Scenario 1. The equivalent cost of energy was calculated as in (5),

$$CE = \frac{C_T}{E_N} + C_{IC} - T_{FI} \quad (5)$$

where the ratio C_T/E_N is the equivalent cost of electrical energy produced in the AD power plant (see Scenario 1), C_{IC} is the purchase cost of electrical energy for industrial consumers (200 €/MWh) and TFI is the fee-in tariff (236 €/MWh). The equivalent cost of energy for Scenario 2 was then of 134 €/MWh.

The economic analysis for Scenario 2 was concluded with the evaluation of the Net Present Value (NPV, after 20 years) that resulted of 302,000 €, the Pay-Back Time of 8.5 years and the ratio between the present TFC and the NPV of 0.67. This means that the expenses born for the construction and installation of the AD power plant were fully recovered after 8.5 years. Moreover, the revenues from the dispatch of electricity to the national grid made possible to reconstitute approximately 70% of the sum of money at the end of the useful lifetime for the implementation of a new plant (digester, CHP unit and installation).

5. CONCLUSIONS

The pilot scale test returned a methane yield for the VMW of approximately 80% of that obtained in the smaller scale. This outcome was in line with those of other studies performed in different scales and modes and indicates the success of the pilot scale test. The net electricity produced from the digester accounted for 30% to 50% of the food industry plants' consumption and had a cost of 172 €/MWh. The available thermal energy could cover from 10% to nearly 100% of the plant requirements, depending on the heat demand of the processes performed.

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Table Legends

Table 1. Physical and chemical characterization of the substrates employed in the tests

Table 2. Results of the carbon balance (substrate – biogas)

Table 3. Comparison of the main results obtained from the two test modes (FB, fed-batch; SC, semi-continuous)

Table 4. Main design parameters for the full scale digester

Table 5. Main performance outcomes of the full scale digester

Table 6. Outcomes of the economic analysis applied to the two scenarios

Figure Legends

Figure 1. Trend of the daily biogas production (1a) and biogas composition b.v. for the VMW (1b)

Figure 2. Trend of the daily biogas production (2a) and biogas composition b.v. for the PSW (2b)

Figure 3. Theoretical and experimental trend of the daily biogas production. Theoretical daily biogas production due to each feedstock. (3a) VMW, (3b) PSW

Figure 4. Semi-continuous test: trend of the daily biogas production and biogas and methane specific production (4a). Biogas composition b.v. for the vegetable mix waste (4b)

Figure 5. Semi continuous test: trend of pH, FOS-TAC, OLR (5a). Trend of TS and VS/TS (5b)

Table 1. Physical and chemical characterization of the waste employed in the tests

Sample	TS (%)$_{wet}$	VS/TS ratio (%)$_{dry}$	pH	C (%)$_{dry}$	H (%)$_{dry}$	N (%)$_{dry}$	S (%)$_{dry}$	Molecular formula $_{(dry)}$
Peeling	6.29	85.8	4.5					
Screen	10.4	85.0	5.0	45.2	6.32	2.93	0.316	$C_{18}H_{30}O_{13.5}N$
Filtrate	9.08	89.6	6.0					
Pesto	69.3	98.0	acidic		N.A.			$C_{48.5}H_{86}O_{17}N$

Table 2. Results of the carbon balance (substrate – biogas)

Substrate	C amount (g) feedstock	C (moles) feedstock	C (moles) CH_4 biogas	C (moles) CO_2 biogas	C conversion (%)
Vegetable mix waste	35.4	2.95	1.02	0.63	56
Waste from pesto sauce production	50.0	4.16	2.64	1.18	92

Table 3. Comparison of the main results obtained from the two test modes (FB, fed-batch; SC, semi-continuous)

	Buswell and Neave model	Fed-batch	Semi-continuous	SC vs. FB ratio
Vegetable mix waste				
Biogas specific production (Nm^3/kg VS)	0.848	0.554 ± 0.038	0.503	0.91
Methane specific production (Nm^3/kg VS)	0.424	0.294 ± 0.029	0.223	0.76
Average methane content (%)	50	53.0 ± 1.8	44.3	0.84
Waste from pesto sauce production				
Biogas specific production (Nm^3/kg VS)	1.14	1.08 ± 0.05	-	-
Methane specific production (Nm^3/kg VS)	0.713	0.722 ± 0.033	-	-
Average methane content (%)	63	66.9 ± 0.2	-	-

Table 4. Main design parameters for the full scale digester

	u.m.	Waste from Plant 1 (VMW)	Waste from Plant 2 (PSW)
Waste generation	t/y	400	360
Waste TS content	% b.w.	10.0	69.3
Waste VS/TS ratio	-	0.850	0.980
Methane specific production	Nm^3/kg VS	0.223	0.578
Fed substrate TS content	% b.w.	10.0	10.0
HRT	days	30	30
Digester filling coefficient	-	0.8	0.8
CHP electrical efficiency	-	0.35	0.35
CHP thermal efficiency	-	0.42	0.42
Process temperature	°C	35	35
Temperature of the fed substrate	°C	15	15

Table 5. Main performance outcomes of the full scale digester

	u.m.	
Working digester volume	m^3	238
Overall digester volume	m^3	297.5
Average biogas production	Nm^3/y	$2.28 \cdot 10^5$
Methane production	Nm^3/y	$1.49 \cdot 10^5$
Biogas primary energy production	MWh/y	1490
Gross electrical energy production	MWh/y	521
Gross thermal energy production	MWh/y	625
Electrical energy consumption	MWh/y	52.1
Thermal energy consumption	MWh/y	135
Net electrical energy production	MWh/y	469
Available thermal energy	MWh/y	490
CHP power output	kW	60

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Table 6. Outcomes of the economic analysis applied to the two scenarios

Scenario 1 **Electrical energy employed in the plant**		**Scenario 2** **Electrical energy dispatched to the National grid**	
Total Fixed Costs, TFC	450,000 €	Total Fixed Costs, TFC	450,000 €
Annual Installment, AI	42,500 €		
Annual Operating and Maintenance costs, $C_{O\&M}$		Electrical energy fee-in tariff	236 €/MWh
• Labor	15,000 €	Net electrical energy production	469 MWh/y
• Maintenance (4% TFC)	18,000 €	Electrical Energy Revenues, EER	110,700 €/y
• Utilities (4% EER, scenario 2)	5,000 €		
Total annual $C_{O\&M}$	37,500 €	Net cash flow (EER – $C_{O\&M}$)	73,200 €/y
Total annual costs (Ct = AI + $C_{O\&M}$)	80,500 €	**Net Present Value (after 20 years), NPV**	**302,000 €**
Net electrical energy production	469 MWh/y	**Pay-back time**	**8.5 y**
Cost of Energy, CE	**172 €/MWh**	**NPV/TFC**	**0.67**

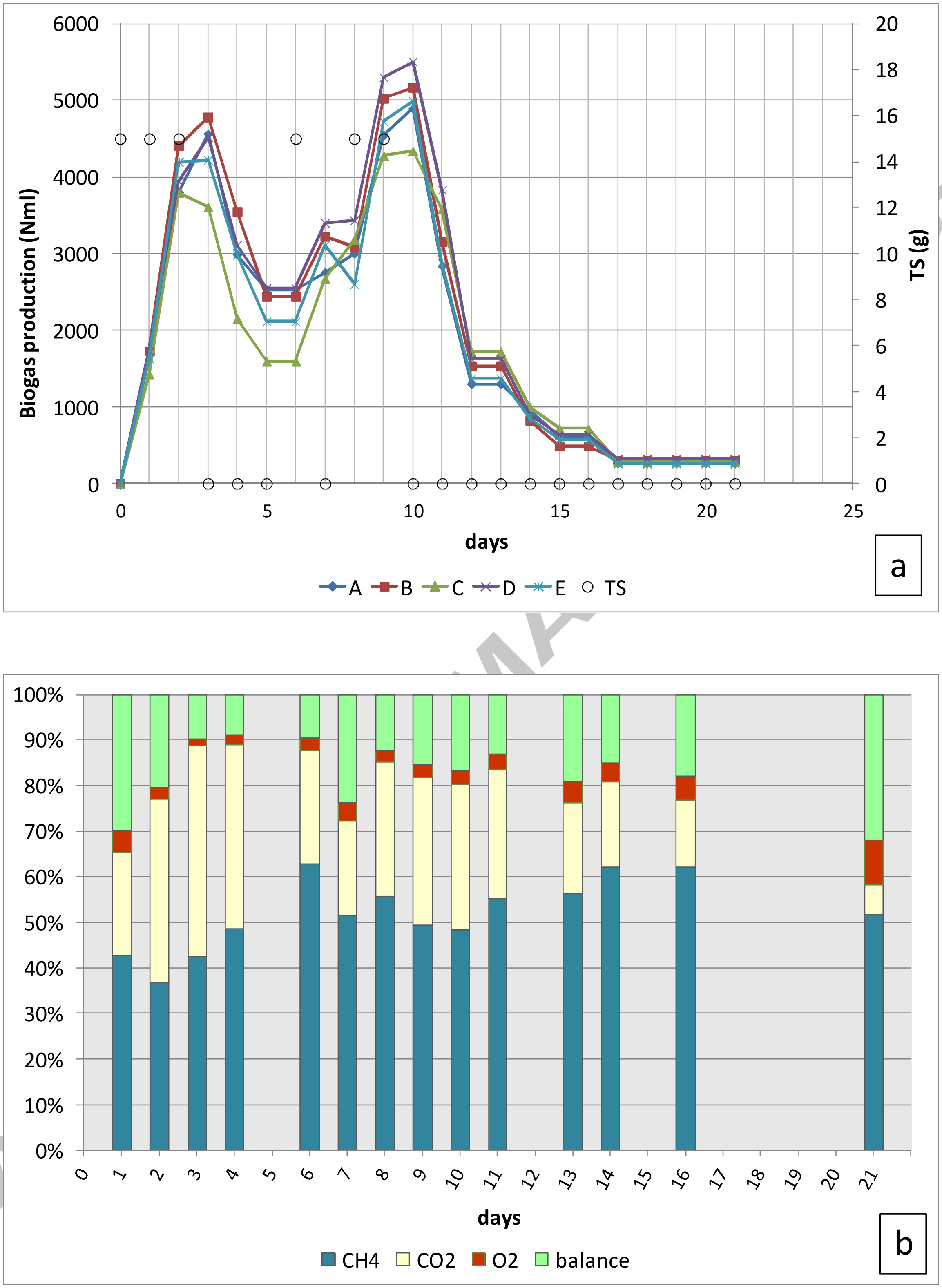


Figure 1. Trend of the daily biogas production (1a) and biogas composition b.v. from VMW (1b)

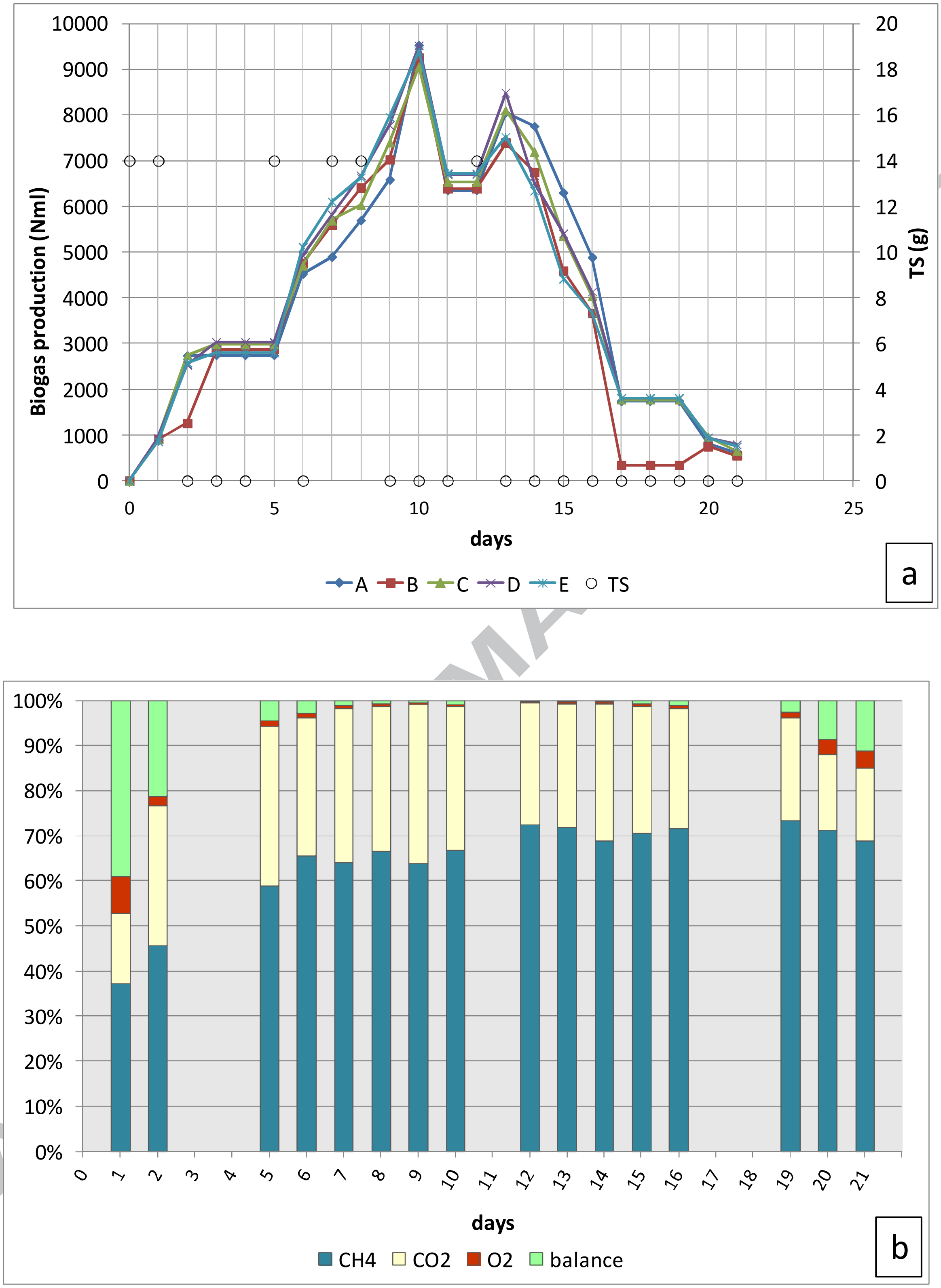
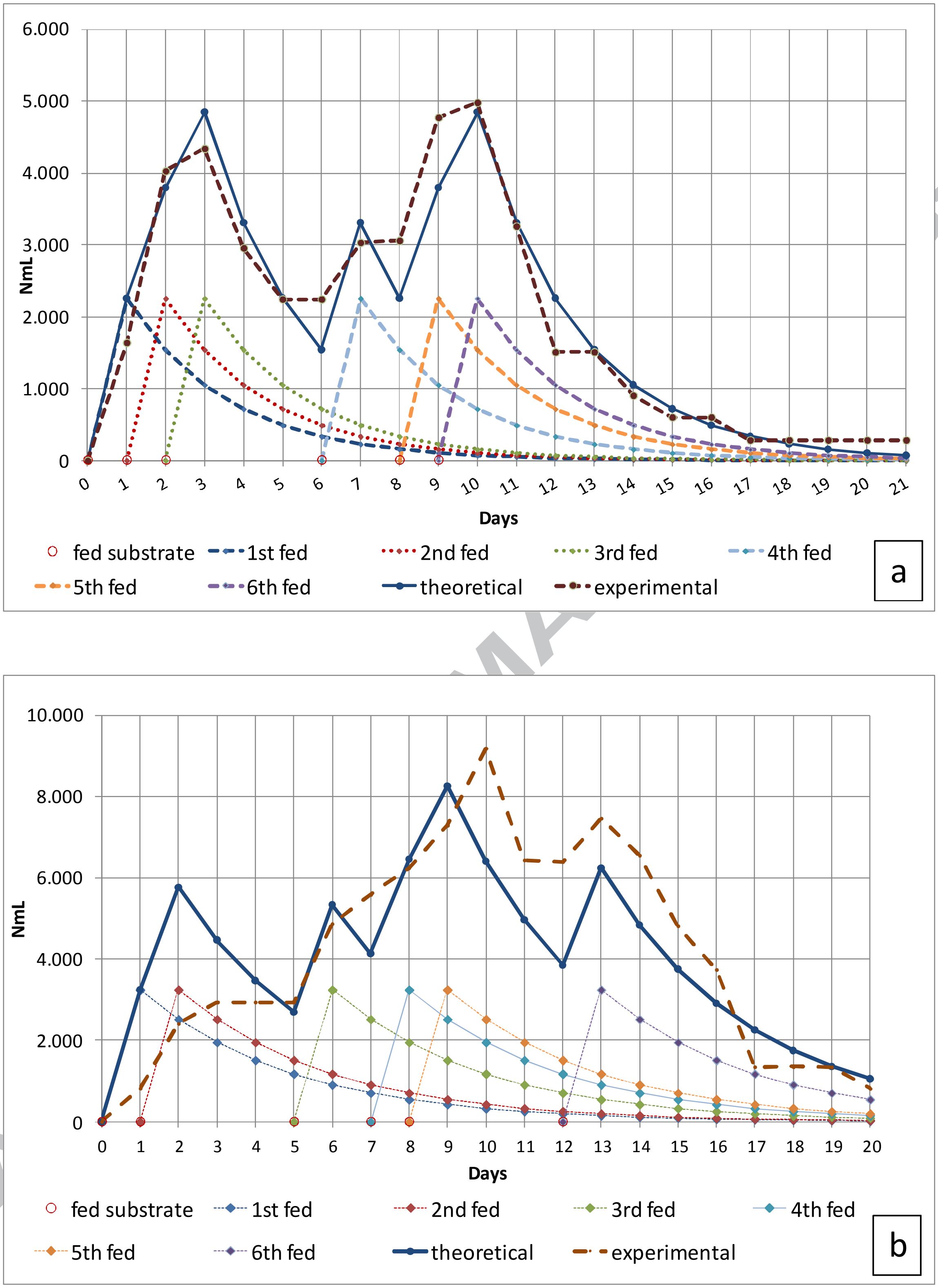


Figure 2. Trend of the daily biogas production (2a) and biogas composition b.v. from PSW (2b)



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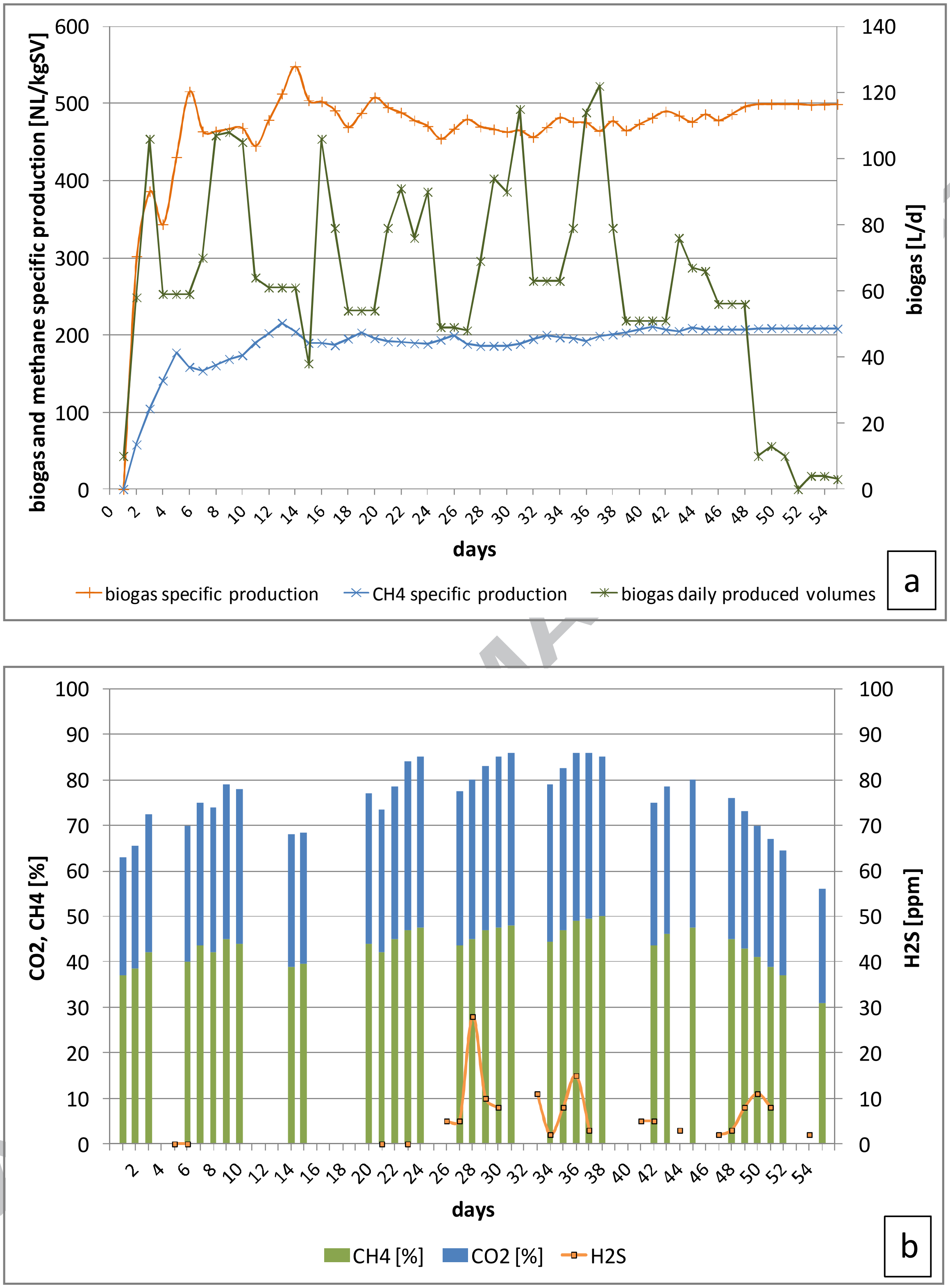

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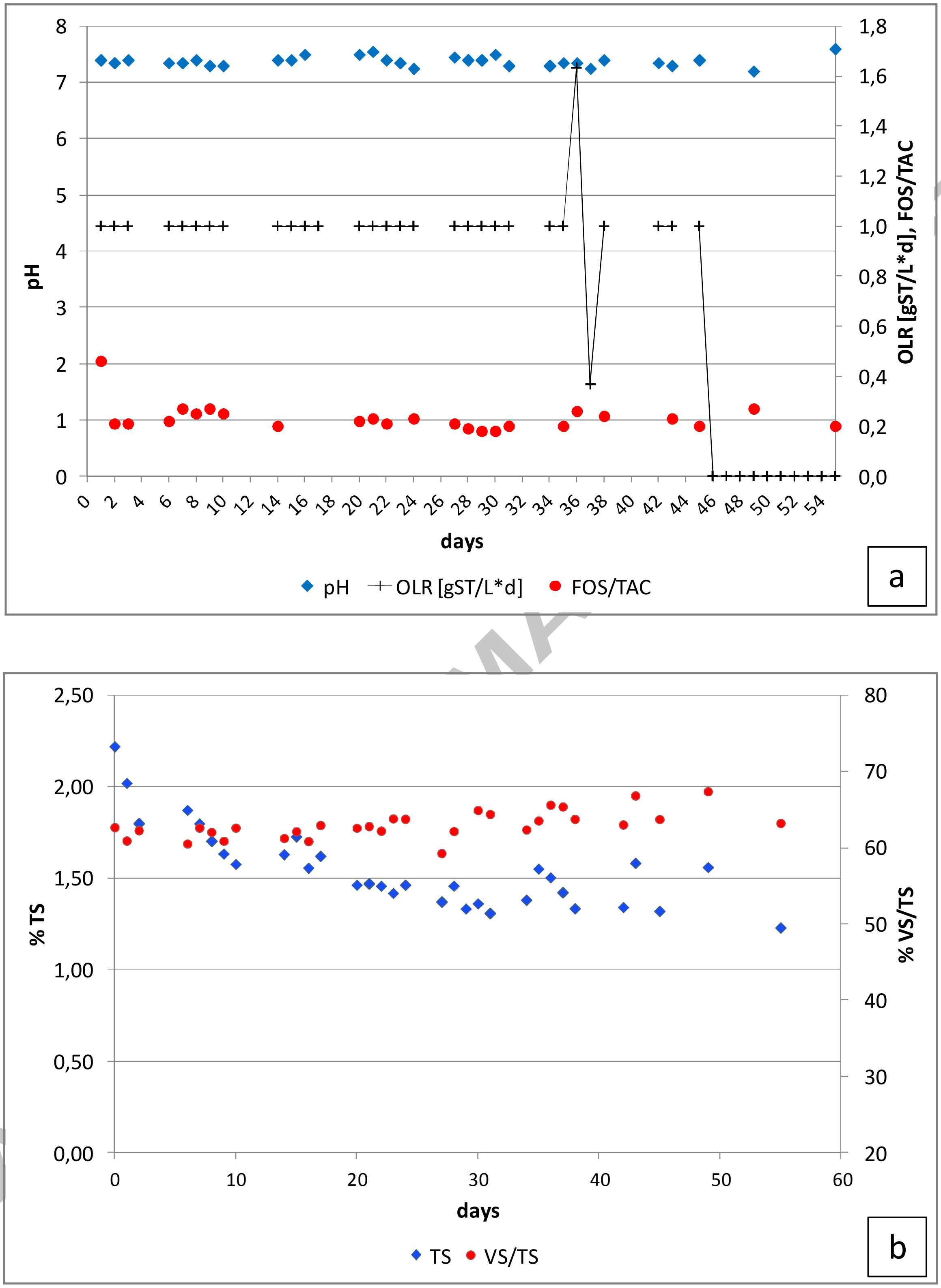

Figure 5. Semi continuous test: trend of pH, FOS-TAC, OLR (5a). Trend of TS and VS/TS (5b)

Highlights

CH_4 production capacity of vegetable waste was assessed on different scales and modes

CH_4 yield from the pilot scale test was about 80% of that from the smaller scale test

Produced net electricity was from 30% to 50% of the food industry plant consumption

Available thermal energy can cover from 10% to near 100% of the plant requirement