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semi-continuous mode for the technical and economic feasibility

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

4

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24 **Abstract**

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

35

36 **Keywords**

37 Mesophilic anaerobic digestion

38 Fed-batch tests

39 Semi-continuous tests

40 Vegetable processing waste

41 Cost-benefit analysis

42 1. INTRODUCTION

43 Italian food industry, with a sales volume of 130 G€, is the second most important industry after the
44 car manufacturing. The Italian food industry plants buy and transform 72% of domestic agricultural
45 raw materials. Inevitably, the processes performed in food industries generate huge amounts of
46 agro-industrial waste. For Italy, Petruccioli et al. (2011) reported productions of 2.4 Mt/y of grape
47 pomace, 0.7 Mt/y of olive pomace and 0.2 Mt/y of both tomato pomace and soybean integuments. It
48 can be estimated that the transformation of 1 t of agricultural raw materials generates from 30 to
49 100 kg of organic waste.

50 Agro-industrial waste, that includes both fruit and vegetable waste, may be stabilized by the
51 anaerobic digestion (AD) process (Khalid et al., 2011). AD is known as a more environmentally
52 friendly and energy saving process for stabilizing high-strength organic waste, than other disposal
53 options like landfilling, incineration, and composting (Hosseini Koupaie et al., 2014; Traversi et al.,
54 2013). In addition to biogas generation, AD benefits include enhancing nutrient characteristics of
55 the dewatered digestate used as fertilizers as well as pathogens reduction. Greenhouse emissions are
56 also decreased because the AD process diverts organic waste from landfills, thus preventing
57 uncontrolled methane and carbon dioxide emissions from decomposition (Xie et al., 2011).

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

64 In order to proceed to the technical and economic assessment of the digester, the potential
65 production of methane from the waste generated in the two plants was determined in both a semi-
66 pilot (6-L, fed-batch mode) and pilot (300-L, semi-continuous mode) scale in mesophilic
67 conditions. Values of the methane yield for waste originated from agro-industrial processes are

68 reported in several studies (Dareioti and Kornaros, 2014; Garcia-Peña et al., 2011; Jiang et al.,
69 2012; Yang et al., 2013). However, only a couple of very recent papers focus on tests carried out on
70 fruit and vegetable waste fed as a single substrate to semi-continuous pilot scale reactors (Fiore et
71 al., 2013; Scano et al., 2014).

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

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

90 The capacity of waste to produce methane obtained in the fed-batch mode was verified using a
91 semi-continuous test carried out in the 300-L digester. The pilot scale test was necessary in order to
92 obtain more reliable results on which to base the design of the full scale digester for the energy
93 valorization of the waste products. The results of tests in the pilot scale are of particular interest

94 especially for highly heterogeneous substrates. The outcomes of the experimentation were
95 employed for a preliminary process and cost-benefit analysis of the full-scale AD digester for
96 treating the waste produced in the two food industry plants.

97

98 **2. MATERIALS AND METHODS**

99 **2.1 Waste origin and characterization**

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

113 Two substrates coming from Plant 1 and Plant 2 were sampled, characterized and employed in the
114 AD tests. The substrate from Plant 1 was a mixture of three waste products. The first waste product
115 (named as “peeling”) originated from the peeling process of vegetables. The second waste product
116 (named as “screen”) was separated from the grid screen positioned at the head of the WWTP
117 contained within the food industry plant. The third waste product (named as “filtrate”) was
118 separated from the cloth filter positioned after the grid screen in the same WWTP. The produced

119 amounts of the three waste products, on dry basis, were the same. For this reason the mix to employ
120 for the tests was obtained by mixing identical amounts of each waste product (on dry basis).

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

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

134 **2.2 Semi-pilot scale test – fed-batch mode**

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

142 The inoculums employed in the tests was prepared from 1,000 mL of digestate collected from the
143 anaerobic digesters of a municipal WWTP located in the same area of the two food industry plants.

144 The inoculums was progressively fed with amounts of primary sludge, coming from the same

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

148 For the test with the VMW, the inoculum had a TS content of $3.26 \pm 0.31\%$, a VS/TS ratio of 64.3
149 $\pm 2.9\%$ and a pH value of 7.23 ± 0.01 (average value on five replicates \pm standard deviation). Each
150 digester was fed with six aliquots of the substrate. Each aliquot contained 15 g of TS and was
151 introduced into the digesters at days 0, 1, 2, 6, 8 and 9 from the beginning of the test. The final TS
152 content was 3% of the working volume (3 L).

153 For the test with the PSW, the inoculum had a TS content of $4.24 \pm 0.21\%$, a VS/TS ratio of $75.1 \pm$
154 0.6% and a pH value of 7.18 ± 0.01 (average value on five replicates \pm standard deviation). Each
155 digester was fed at days 0, 1, 5, 7, 8 and 12 with six aliquots of the substrate, each of them
156 contained 14 g of TS. Differences in fed frequency between test 1 and 2 were due to the limitation
157 in the maximum daily biogas collection capacity (10-12 L) and depended on the biogas production
158 rate. However, also for Test 2, the final TS content in the digester was approximately 3%.

159 For each replicate, the produced biogas was collected in two 5-L Tedlar® bags connected in
160 parallel. The characterization and measure of the biogas volume was carried out daily, throughout
161 the entire duration of the test. The characterization, that is the volumetric composition of the biogas
162 in terms of CH₄, CO₂, O₂ and “balance” (the fraction made up of gases that are different from the
163 first three, that is mainly nitrogen, hydrogen, carbon monoxide and volatile organic compounds),
164 was obtained by flushing 500 mL of biogas through a biogas analyzer (Biogas Check, Geotechnical
165 Instruments Ltd). The volume was measured by replacing volumes of water with biogas and
166 referring the obtained value to standard conditions.

167 **2.3 Pilot scale test – semi-continuous mode**

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

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

171 80-L gasometer and a system for on-line monitoring of the biogas composition. The temperature
172 was automatically monitored and regulated by means of a resistance temperature sensor (Pt 100)
173 positioned along the reactor and connected to the heating system. The biogas collection system
174 included a condensation trap for moisture removal and a flow-meter. The pilot plant was controlled
175 by a PLC system that receives signal from the different sensors and drives the main electrical and
176 pneumatic pieces of equipment. Substrates inside the digester were mixed 15 minutes every hour by
177 a system of re-circulating biogas. The other physical and chemical parameters required for an
178 effective monitoring of the AD process (TS, VS content, pH and FOS-TAC values) were daily
179 determined on the sampled digestate.

180 The inoculums for the test was prepared by mixing 80 L of digestate, collected from the digesters of
181 the WWTP as in Section 2.2, and 160 L of primary sludge from the same plant. The inoculums was
182 considered ready to start the test when its daily biogas production was of less than 1% of the overall
183 volume produced throughout the period of preparation. At the beginning of the semi-continuous test
184 the inoculums had a TS content of $2.22 \pm 0.01\%$, a VS/TS ratio of 62.7 ± 0.2 and a pH value of 7.4
185 ± 0.1 (average value on 3 replicates \pm standard deviation).

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

192 The volume of the fed substrate was of 8 L with a TS content of 240 g and resulting OLR of 1
193 $\text{kgTS/m}^3 \cdot \text{d}$ or $0.87 \text{ kgVS/m}^3 \cdot \text{d}$. Due to the characteristics of the waste product (that is an average
194 TS content of approximately 8% and a VS/TS ratio of 86.8%, see Section 3.1), the daily feedstock
195 was prepared by mixing 2,200 g of the VMW with 5,800 mL of tap water. The AD test had an
196 overall duration of 54 days that were necessary for the loading of 30 volumes of substrate and the

197 subsequent complete depletion of the biogas produced. The feeding of the substrate and the
198 extraction of an equal volume of digestate were carried out in days from Monday to Friday,
199 according to the procedure described as in the follow:

- 200 1. recording of volume and composition (methane, CO₂, oxygen, balance and H₂S) of the biogas
201 produced since the previous recording;
- 202 2. extraction of 8 L of digestate to undergo to pH, TS, VS, FOS-TAC analysis;
- 203 3. loading of 8 L of substrate, prepared as previously described.

204

205 **3. RESULTS AND DISCUSSION**

206 **3.1 Waste characterization**

207 The characteristics of the substrates involved in the digestibility tests are shown in Table 1.

208 The VMW had an average TS content of less than 10% b.w. (by weight), a VS/TS ratio close to
209 90%, an acidic pH value and a elemental composition of C: 45% b.w., H: 6-7% b.w., N: 2-3% b.w.,
210 S: 0.3-0.5% b.w., on dry basis, with a subsequent C/N ratio of approximately 15. A visual test
211 revealed that the sizes of particles ranged from few millimeters (mainly residues from the peeling
212 process) to about 5 centimeters (pieces of vegetables and discarded mushroom caps).

213 The PSW was far more homogeneous than VMW. It had the texture of a sauce with a TS content of
214 approximately 70% b.w. and a VS/TS ratio close to 100%. pH value was determined only with pH
215 strips because of the high oily content of the waste product. For the same reason the determination
216 of the elemental composition was not performed.

217 The molecular formula of the first substrate was obtained from its elemental composition. The
218 molecular formula of the second substrate was obtained from the average composition of a general
219 vegetable (C₁₈H₃₀O_{13.5}N, as for the first substrate), that simulates basil, and sunflower oil
220 (C_{17.9}H_{32.8}O₂). The ratio between basil, as dry matter, and sunflower oil was assumed equal to 1:1
221 b.w.

222

223 3.2 Semi-pilot scale test

224 Both fed-batch tests performed on the two waste products had an overall duration of 22 days.

225 According to 1% criterion of German Guideline VDI 4630 (2006), the tests were stopped when the
226 daily biogas production dropped to values of less than 1% of the total gas production.

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

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

244 With reference to the second substrate (PSW), the daily evolution of the biogas produced is shown
245 in Figure 2. Figure 2a shows two peaks at days 10 and 13 from the beginning of the test. In those
246 days the daily biogas production rose to volumes respectively of 9.5 and 8 NL. Unlike the case
247 described above, the trend of the daily biogas production for the PSW did not show a strong
248 dependence on the feeding rate. The total amount of the fed substrate (84 g of dry substance in 6

249 aliquots) determined an overall biogas production that was between 82 and 92 NL. The five
250 replicates of the semi-pilot scale test returned a biogas specific production of 1.08 ± 0.05
251 Nm^3/kgVS added (average value on five replicates \pm standard deviation), a methane specific
252 production of $0.722 \pm 0.033 \text{ Nm}^3 \text{ CH}_4/\text{kgVS}$ added, with a subsequent average methane content of
253 $66.9 \pm 0.2\%$ b.v. The composition of the biogas produced in the days in which the record of volume
254 and composition was carried out is shown in Figure 2b. The average carbon dioxide content was of
255 $29.8 \pm 2.1\%$. As for the previous substrate, the biogas produced did not show any traces of H_2S and
256 the daily pH check did not highlight critical situations (data not showed).

257 With reference to the second tested substrate, the digestate had a TS content of $3.88 \pm 0.21\%$, a
258 VS/TS ratio of $72.7 \pm 0.9\%$ and a pH value of 7.24 ± 0.02 . A TS and VS depletion close to 100%
259 was calculated from the balance that involved the inoculums, the digestate and the fed substrate
260 (121.1 g wet substrate with a TS content of 69.3%, 84 g TS, 82.3 g VS).

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

267 In order to compare the obtained specific productions of biogas and methane with the maximum
268 production predicted by theoretical calculations, according to Buswell and Neave model
269 (Tchobanoglous et al., 1993), it is necessary to refer to the molecular formula of the two substrates,
270 as reported in Table 1. According to this model, the VMW subjected to AD would give rise to a
271 biogas specific production of $0.848 \text{ Nm}^3/\text{kgVS}$ added and a methane specific production of 0.424
272 Nm^3/kgVS added (as listed in Table 3). The observed values were respectively only 65.3% and
273 69.3% of the values predicted by Buswell and Neave model. On the other hand, the theoretical
274 biogas specific production of the PSW was of $1.14 \text{ Nm}^3/\text{kgVS}$ added, 5% more than the value

275 returned from the fed-batch test.

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

- 278 • in the specific production values of biogas and methane. The biogas specific production of the
279 VMW substrate was approximately 50% of that of the PSW substrate. On the other hand, the
280 methane specific production of the VMW was 40% of that of the PSW;
- 281 • in the response of the AD systems to the feedstock introduction. Reactors fed with the VMW
282 substrate showed a fast production of biogas and methane. Reactors fed with the PSW
283 substrate showed a production of biogas and methane more intense than that from the VMW,
284 but only after a phase of initial delay;
- 285 • in the conversion rate of the fed carbon to methane and carbon dioxide, that was in the order of
286 56% for the VMW substrate and greater than 90% for the PSW substrate;
- 287 • in the ratio between the biogas and methane specific production values obtained from the fed-
288 batch tests and those from Buswell and Neave model.

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

295 On the other hand, the second substrate (PSW) showed a considerable oil content and very small
296 particle sizes. Fatty acids from oil and soluble COD liberated from the vegetable matrix, following
297 the size reduction occurred in pesto sauce preparation, were readily available to AD. However,
298 according to Batsone et al., 2002, the maximum removal rate of fatty acids (6 d^{-1}) is one order of
299 magnitude lower than that of sugars and amino acids (the simplest substances that compose
300 vegetable matrices, respectively equal to 30 and 50 d^{-1}). This may explain the delay observed in the

301 first phase of the test, that was due to the low removal rate of fatty acids. Moreover, the
302 composition of the second substrate may explain the higher values of specific production of biogas
303 and methane, compared to those observed for the VMW, and the high ratio between the
304 experimental value and the theoretical value. These last observations came from the higher presence
305 of completely degradable substances (fatty acids, and available amino acids and sugars from the
306 vegetable matrix) in the PSW substrate than in the VMW.

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

$$315 \ln \frac{B_{\infty} - B}{B_{\infty}} = -kt \quad (1)$$

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

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

326 organic acids in the first days of the test could have made the AD process slower than predicted by
327 the model.

328 The values found for the two substrates are in line with the values of the same parameter found for
329 organic substrates that originates from vegetables, as reported in Galí et al., 2009; Giuliano et al.,
330 2013; Shi et al., 2014. In those works the authors found k values that ranged from 0.2 to 0.4 d⁻¹ and
331 Giuliano et al., 2013 reported values of 0.26 d⁻¹ and 0.34 d⁻¹ respectively for potatoes and onions
332 when BMP tests were carried out in mesophilic conditions.

333 3.3 Pilot scale test

334 Only the VMW was involved in the pilot scale test. The daily production of biogas and the daily
335 specific production of biogas and methane are shown in Figure 4a. Values of production of the days
336 in which the recording of data (volume, biogas composition, see Figure 4b) was not performed
337 (Saturdays, Sundays), were obtained dividing the value recorded the first day after the break by the
338 duration of the break. The diagram of Figure 4a shows that the biogas daily production had a
339 fluctuating trend, that ranged from 50 NL/d to 100 NL/d. The lowest values were registered in the
340 two days that ensued the feeding stops. The highest values were registered when the loading of the
341 substrate was made. The value of the methane specific production, after showing considerable
342 fluctuations until around day 20 from the beginning of the test, stabilized at 0.223 Nm³/kgVS
343 added. The values of the two parameters FOS and TAC showed a trend that decreased from the
344 beginning of the test to the end. However, the ratio between FOS and TAC kept constant in the
345 range 0.2 - 0.3 (see Figure 5a), optimum for an AD process. The daily record of the TS content into
346 the digestate showed a considerable decrease in the first ten days from the beginning of the test,
347 and, then, a subsequent fluctuation around the constant value of 1.50% from day 10 (see Figure 5b).
348 At the end of the test the amount of TS removed was of 55% and the amount of VS removed in the
349 order of 65%. This value was only slightly lower than that found in the fed-batch test (73%). As
350 shown in Table 3 the methane specific production obtained in the semi-continuous test was

351 approximately 76% of that obtained in the fed-batch test and the difference in the average methane
352 content between the two tests was of less than 20%.

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

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

366 Kafle and Kim (2013) tested mixtures of apple waste and swine manure in both batch (1.8 L liquid
367 volume) and continuous modes (4.5 L liquid volume). The outcomes of the tests carried out in the
368 two modes on a mixture that contained 33% apple waste were compared. The methane yield from
369 the continuous test increased, from the first to the second phase of the experimentation, from 182 to
370 241 mL CH₄/gTCOD added. The methane yield registered in the second phase was approximately
371 90% of the value returned from the batch test (267 mL CH₄/gTCOD added).

372 Zupančić and Jemec (2010) evaluated the capacity of several kinds of tannery waste to producing
373 methane, in both mesophilic and thermophilic conditions, using BMP tests carried out in 1,160 mL
374 glass vessels. Tests were repeated in semi-continuous mode in a 30-L digester obtaining specific
375 methane productivity values between 79 and 93% of those obtained in the BMP tests. The ratio
376 between the volume of the digester employed for the semi-continuous tests and that of the reactors

377 for the batch tests was approximately of 30, a value of the same order of magnitude of that of this
378 study (80). For this reason, in spite of the kind of the tested substrates (fleshings, skin trimmings
379 and wastewater sludge), the results of the present study have to be considered consisted with those
380 by Zupančić and Jemec.

381 Wang et al. (2014) investigated the anaerobic digestion performances of kitchen waste and
382 fruit/vegetable waste from lab-scale (working volume of 1.5 L) to pilot-scale (2 and 4 m³),
383 including batch, single-phase and two-phase experiments. The methane yields obtained on a pilot-
384 scale (0.64 LCH₄/gVS) was about 80% of that obtained on a lab-scale (0.725 L CH₄/gVS) under
385 similar VS loading values.

386 Moreover, a study performed in the same apparatus of the present study and in the same operating
387 conditions, on a mix of rice hull and rice bran, returned a ratio of approximately 0.81 between the
388 methane specific production obtained on a semi-continuous mode (0.312 Nm³/kgVS added) and the
389 methane specific production obtained on a fed-batch mode (0.386 Nm³/kgVS added).

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

397 In spite of the results of this last case, usually batch tests returned methane yields higher than those
398 obtained in a semi-continuous mode. This is obviously due to the longer contact time between
399 substrate and microbial population, that allows microorganisms to completely degrade substrates,
400 and to the better mixing conditions achieved in batch reactors (especially if of small size). It can be
401 concluded that tests in a batch (or fed-batch) mode may provide a good estimate of the capacity of a
402 substrate to be degraded in anaerobic conditions and are useful to assess if further tests with higher-

403 scale reactors may be justified. Batch tests performed on a scale higher than usual assure reliable
404 outcomes in the case of high heterogeneity of the substrate, thus guaranteeing the representativeness
405 of the whole sample and avoiding the necessity of reducing sizes. However, the assessment of a full
406 scale reactor for waste energy valorization requires solid methane yield values that can be obtained
407 only from a pilot scale test. In agreement with previous literature studies, the outcomes of this work
408 demonstrated that a successful pilot scale test returned methane yields between 70 and 80% of the
409 values from batch tests.

410 **3.4. Energy valorization**

411 The results obtained in the tests described in Sections 3.2 and 3.3 were employed for a preliminary
412 technical and economic evaluation of a full-scale AD reactor for the energy valorization of the
413 waste products generated in Plant 1 and Plant 2. The methane specific production for the VMW was
414 assumed equal to $0.223 \text{ Nm}^3/\text{kgVS}$ added, the value returned by the semi-continuous test. For the
415 PSW the methane specific production was assumed equal to $0.578 \text{ Nm}^3/\text{kgVS}$ added, that is 80% of
416 the value from the fed-batch test. According to the remarks set out in Section 3.3, this value could
417 be considered acceptable for the design of a full scale plant. The two feeding substrates are
418 generated at a constant rate throughout the year. Their characteristics are also nearly constant.
419 The full scale plant is based on a single-stage reactor that operates in mesophilic condition with an
420 HRT of 30 days. HRT was set equal to the value employed in the semi-continuous test. Because the
421 dry substance fed to the digester is supposed not to exceed 10% (the highest value allowed in an AD
422 wet process), the PSW has to be diluted with water. From a balance that takes into account the
423 amount and the TS content of the PSW and the TS content allowed in the substrate fed to the
424 digester, the yearly amount of water required for dilution was equal to approximately $2,200 \text{ m}^3$, that
425 is 6 L/kg waste. The produced biogas feeds a combined heat and power (CPH) unit based on an
426 internal combustion engine integrated with a heat recovery section. The assumptions concerning the
427 electrical and thermal efficiencies of the CHP unit were based on typical values for commercial
428 units. The main data for the design of the full scale digester are listed in Table 4.

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

434 From the lower heating value (LHV) of the methane generated from the AD process, a first estimate
435 of the daily methane production suggested a CHP unit with a power output of 60 kW. The gross
436 electrical and thermal yields were calculated starting from biogas production and electrical and
437 thermal efficiencies of the CHP unit. The net electrical energy production was obtained by
438 subtracting the internal electrical energy consumption from the gross electrical energy production.
439 The internal electrical energy consumption was assumed to be equal to 10% of the gross production.
440 The available thermal energy was obtained by subtracting the thermal energy requirements of the
441 AD process and the heat losses with the outside from the gross thermal energy production. The
442 thermal energy requirements of the AD process were calculated as the amount of heat necessary to
443 raise the temperature of the fed substrate (yearly average, 15°C) to the temperature of the AD
444 process (35°C). The ratio between the thermal energy requirement of the AD process and the gross
445 thermal energy production resulted to be of about 10%. Taking into account the geometry of the
446 full-scale digester, the materials employed for construction, the temperature of the fed mixture
447 (15°C), soil (15°C) and outside (exterior environment, as monthly averages as reported in the UNI
448 10349 rule), it may be assumed that the amount of thermal energy to balance the thermal losses is of
449 the same order of magnitude of the thermal energy requirement of the AD process (i.e. $7 \cdot 10^4$
450 kWh/y).

451 The annual net electrical energy production of the digester, equal to 470 MWh, was enough to cover
452 from 30% to 50% of the electrical consumption of a food industry plant with a size similar to that of
453 the plants that produced the waste products considered in this study. On the other hand, the

454 available thermal energy (500 MWh/y) may cover from 10% to near 100% of the plant
 455 requirements depending on the energy demand of the processes performed.

456 On the full-scale digester designed as described above, a preliminary economic analysis was carried
 457 out. Two different scenarios that refer to the use of the electrical energy generated only by the AD
 458 process were considered. In the first scenario the electricity was employed for the internal energy
 459 requirements of the food industry plant and no public subsidies were obtained. In the second
 460 scenario the electricity was dispatched to the national grid, in agreement with the current Italian
 461 legislation (DM 6/07/2012) on energy production from renewable sources. In fact, the most recent
 462 Italian policy supports the use of biological by-products deriving from agricultural and food
 463 industries as substrate for AD power plants, especially of small sizes (from 1 to 300 kWe). The fee-
 464 in tariff of 236 €/MWh is the maximum allowed for the electricity generated in AD power plants.
 465 With reference to both scenarios, the total annual costs (C_T) born by the plant for the production of
 466 electricity from the AD process was calculated as in (2)

$$467 \quad C_T = TFC \cdot CCR + C_{O\&M} \quad (2)$$

468 where:

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

$$473 \quad CCR = \frac{i}{1 - (1+i)^{-n}} \quad (3)$$

474 For the economic assessment carried out in this work, the annual interest rate was fixed to 7% and
 475 the operating lifetime to 20 years (as stated by DM 6/07/2012). The TFC was calculated as the sum
 476 of the costs of digester, CPH unit and installation. Plant unit capital cost investment were fixed to
 477 6,000 €/kWe. Since the electrical power of the plant was of 60 kWe and the costs for installation

478 were fixed to 100,000 €, the resulting TFC was approximately of 450,000 € and, consequently, the
 479 annual depreciation 42,500 € (see Table 6).

480 The annual operating and maintenance costs were assumed to be equal to 4% of the TFC, that is
 481 18,000 €. The total labor costs were fixed to 15,000 €, considering 0.5 workers employed on the
 482 plant. Costs for utilities, that is raw materials supply, treating and restoring water and waste
 483 disposal, were assumed to be equal to 4% of the value of the electrical energy (see EER, Table 6), if
 484 dispatched to the national grid as required by Scenario 2. The income from the sale of electrical
 485 energy, as in Scenario 2, was calculated multiplying the annual net electrical energy produced (469
 486 MWh) by the fee-in tariff of 236 €/MWh and resulted of approximately 110,700 €/y. The total
 487 amount of the annual operating costs was then equal to approximately 37,500 €.

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

$$491 \quad CE = \frac{C_T}{E_N} \quad (4)$$

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

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

$$500 \quad CE = \frac{C_T}{E_N} + C_{IC} - T_{FI} \quad (5)$$

501 where the ratio C_T/E_N is the equivalent cost of electrical energy produced in the AD power plant
 502 (see Scenario 1), C_{IC} is the purchase cost of electrical energy for industrial consumers (200 €/MWh)

503 and TFI is the fee-in tariff (236 €/MWh). The equivalent cost of energy for Scenario 2 was then of
504 134 €/MWh.

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

512

513 **5. CONCLUSIONS**

514 The pilot scale test returned a methane yield for the VMW of approximately 80% of that obtained in
515 the smaller scale. This outcome was in line with those of other studies performed in different scales
516 and modes and indicates the success of the pilot scale test. The net electricity produced from the
517 digester accounted for 30% to 50% of the food industry plants' consumption and had a cost of 172
518 €/MWh. The available thermal energy could cover from 10% to nearly 100% of the plant
519 requirements, depending on the heat demand of the processes performed.

520

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629 **Table Legends**

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

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

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

634 Table 4. Main design parameters for the full scale digester

635 Table 5. Main performance outcomes of the full scale digester

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

637

638 **Figure Legends**

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

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

641 Figure 3. Theoretical and experimental trend of the daily biogas production. Theoretical daily
642 biogas production due to each feedstock. (3a) VMW, (3b) PSW643 Figure 4. Semi-continuous test: trend of the daily biogas production and biogas and methane
644 specific production (4a). Biogas composition b.v. for the vegetable mix waste (4b)

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

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658 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

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662 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

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666 Table 3. Comparison of the main results obtained from the two test modes (FB, fed-batch; SC,
 667 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	-	-

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670 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

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673 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 · 10 ⁵
Methane production	Nm ³ /y	1.49 · 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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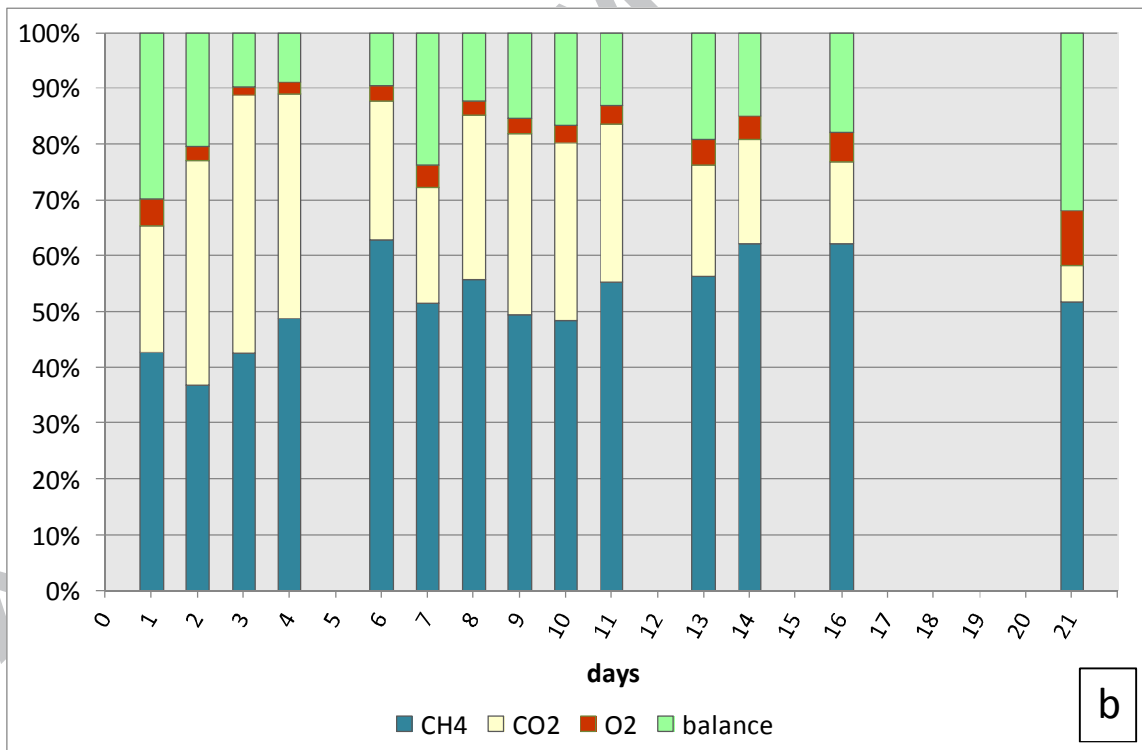
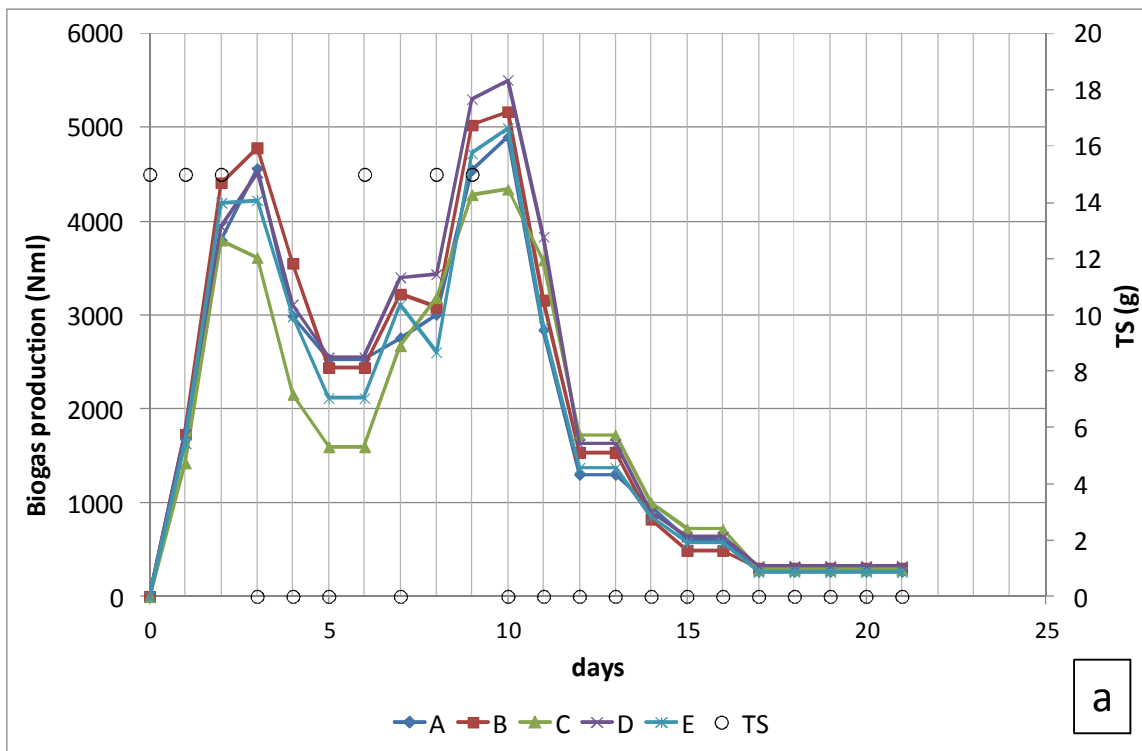
676 Table 6. Outcomes of the economic analysis applied to the two scenarios

Scenario 1		Scenario 2	
Electrical energy employed in the plant		Electrical energy dispatched to the National grid	
Total Fixed Costs, TFC	450,000 €	Total Fixed Costs, TFC	450,000 €
Annual Installment, AI	42,500 €		
<i>Annual Operating and Maintenance costs, C_{O&M}</i>		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 (C _t = 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

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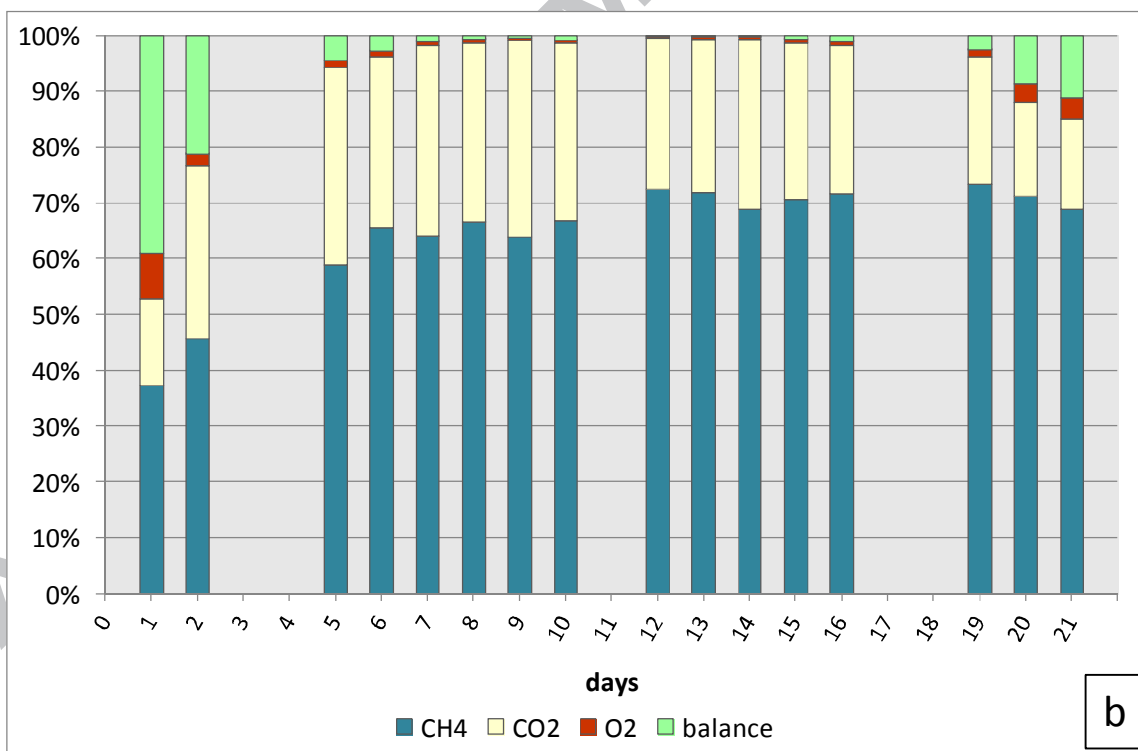
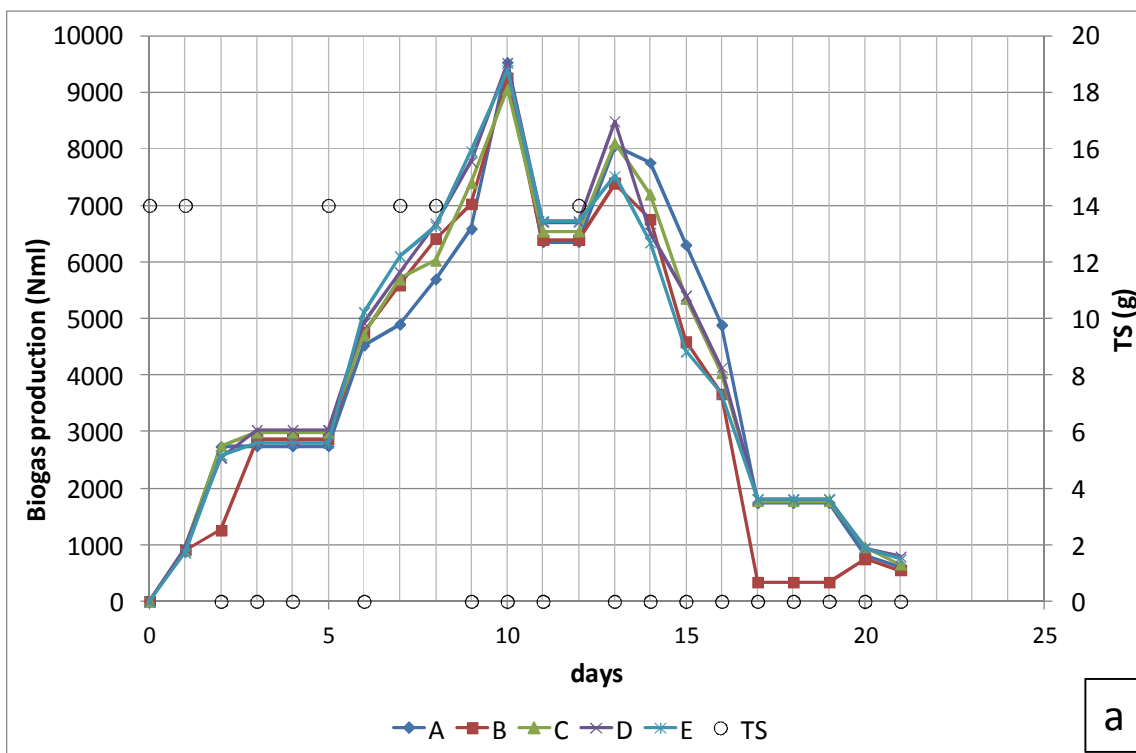
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680 Figure 1. Trend of the daily biogas production (1a) and biogas composition b.v. from VMW (1b)

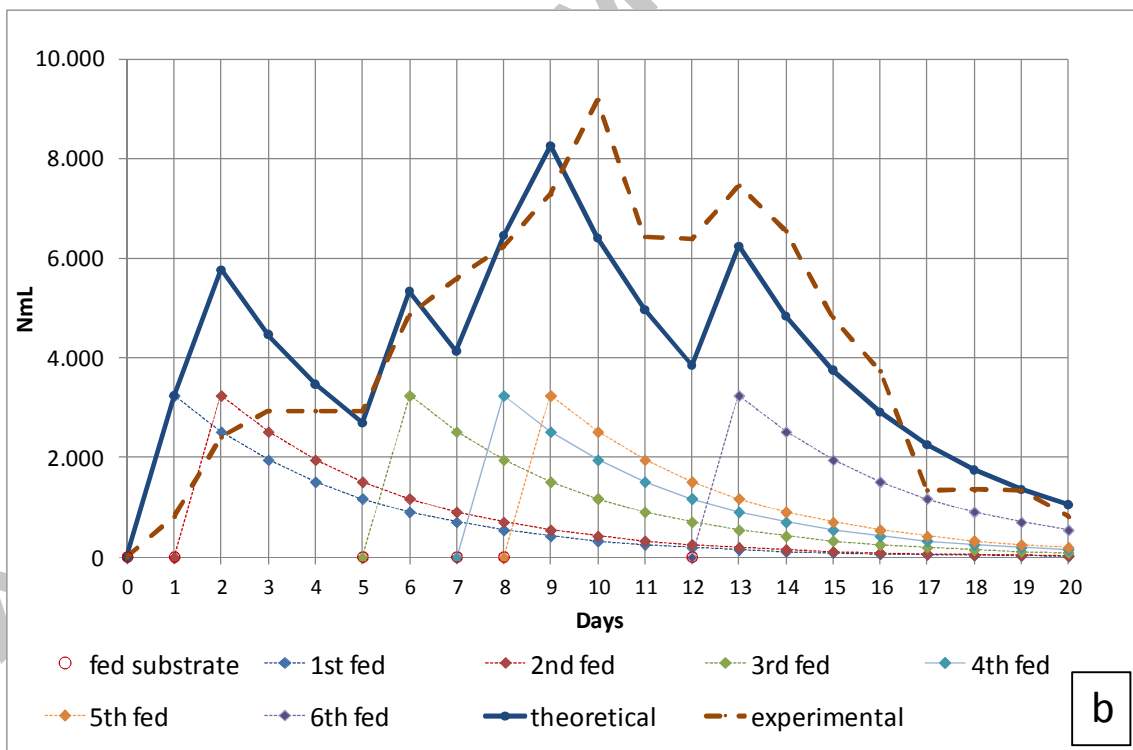
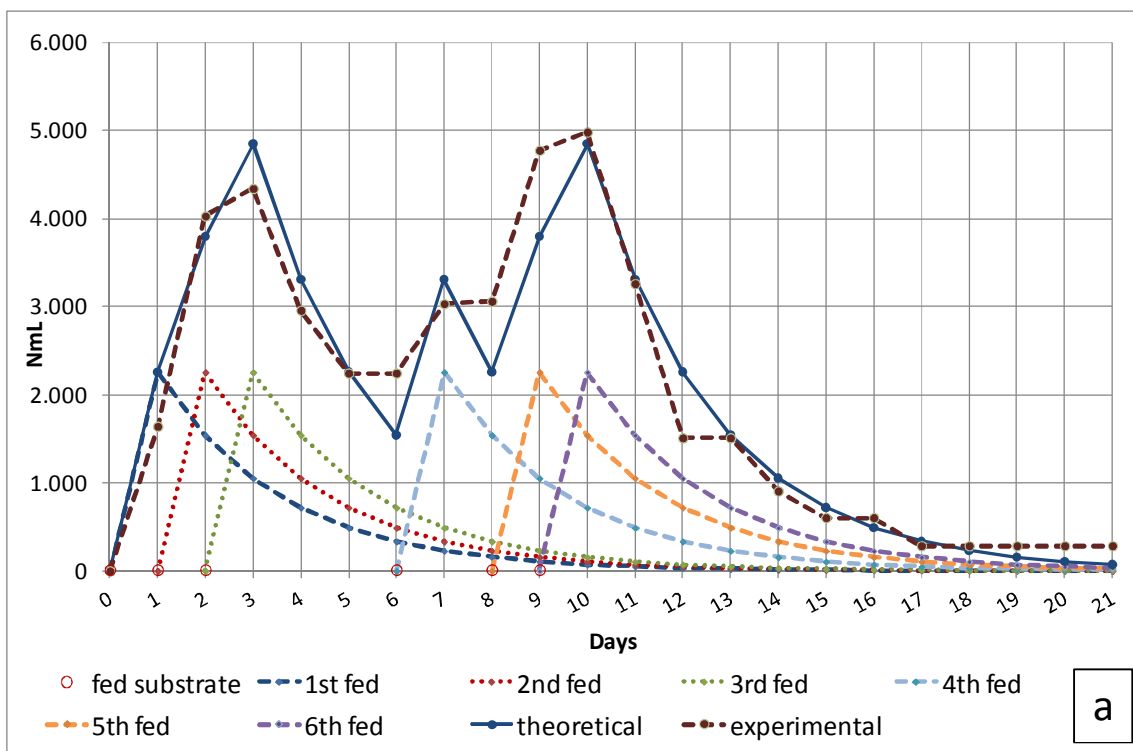
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683 Figure 2. Trend of the daily biogas production (2a) and biogas composition b.v. from PSW (2b)

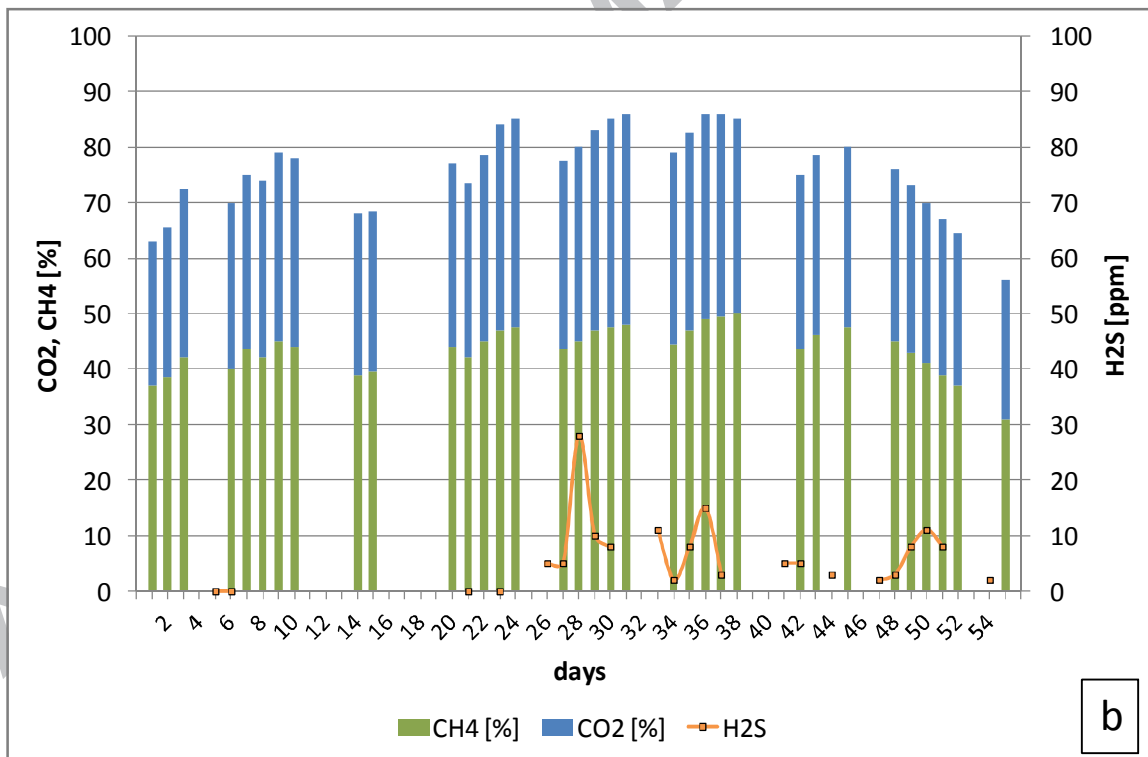
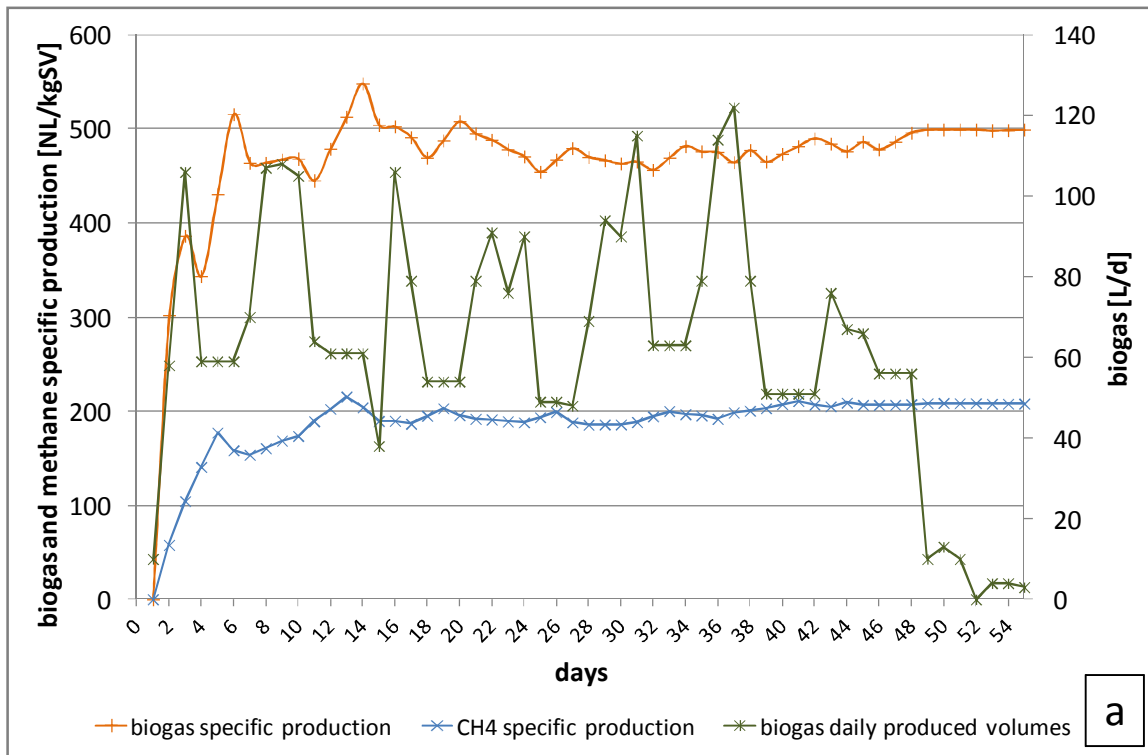
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686 Figure 3. Theoretical and experimental trend of the daily biogas production. Theoretical daily
 687 biogas production due to each feedstock. (3a) VMW, (3b) PSW.

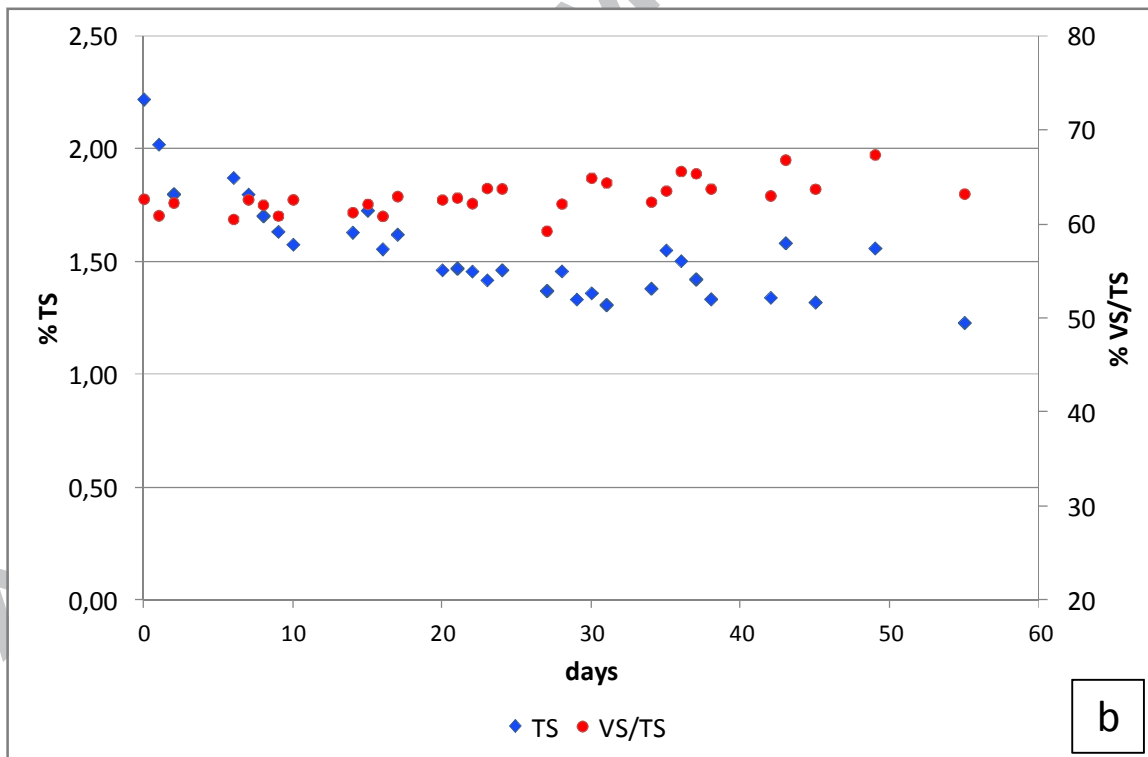
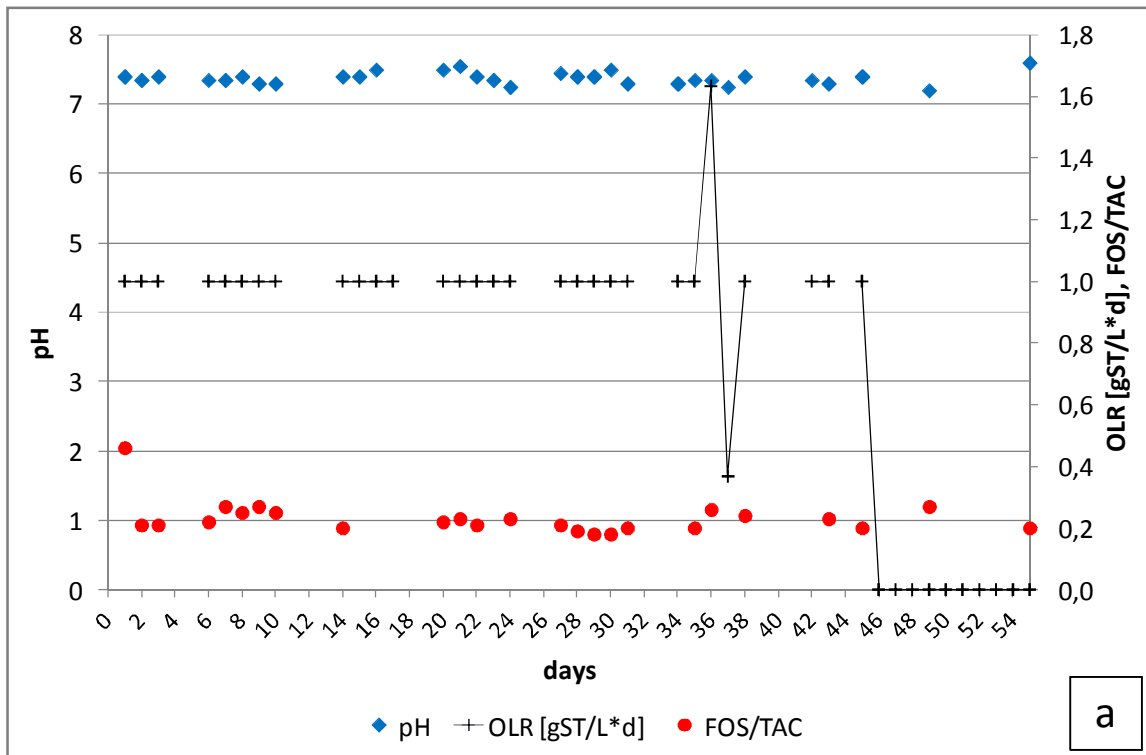
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690 Figure 4. Semi-continuous test: trend of the daily biogas production and biogas and methane
 691 specific production (4a). Biogas composition b.v. for the vegetable mix waste (4b)

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

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697 **Highlights**

698 CH₄ production capacity of vegetable waste was assessed on different scales and modes

699 CH₄ yield from the pilot scale test was about 80% of that from the smaller scale test

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

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

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