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Energy production from anaerobic co-digestion processing of cow slurry, olive pomace and apple pulp / Riggio, Vincenzo; Comino, Elena; Rosso, Maurizio. - In: RENEWABLE ENERGY. - ISSN 0960-1481. - 83(2015), pp. 1043-1049.

Availability:

This version is available at: 11583/2646111 since: 2016-09-05T09:23:58Z

Publisher:

Elsevier Ltd

Published

DOI:10.1016/j.renene.2015.05.056

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1 ENERGY PRODUCTION FROM ANAEROBIC CO-DIGESTION
2 PROCESSING OF COW SLURRY, OLIVE POMACE AND APPLE
3 PULP.

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7
8 Abstract

9 This paper deals with anaerobic co-digestion of cow slurry, apple pulp and olive
10 pomace mixture and results obtained shown that the production of methane by co-
11 digestion of cow slurry, olive pomace and apple pulp is not only possible but also
12 economically and energetically attractive. Tests were performed with a pilot scale
13 anaerobic digester, 128 l in volume, operating under batch and fed-batch condition. The
14 biogas production, methane yield and quality, plus other operating parameters were
15 evaluated under four feeding regimes, to simulate a real situation. Stable biogas
16 production was obtained of about 400 l/kg Volatile Solids at a Hydraulic Retention Time
17 of 40 days in a mixture containing 85% cow slurry, 10% olive pomace and 5% apple pulp
18 (% by volume). The percentage of methane inside the biogas was around 52% and the
19 maximum COD removal was 63%.

20 Keywords: Anaerobic co-digestion; Methane yield; COD reduction; Digestate yield test;
21 Energy production.

22

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23

24 1. Introduction

25 Many agricultural biogas plants have been, or are going to be, built in Italian territory
26 due to strong public support for renewable energies. These plants are mainly fed with
27 cattle slurry and various type of crops mixture. At the same time, large quantities of agro-
28 industrial by-products have no economic value and are discarded in landfill [1]. In areas
29 and region where agricultural productions are focused on specific cultivation like apples
30 and olives these biomasses could be used in anaerobic digestion plants [2] and could be
31 used to substitute food crops in the anaerobic reactors feeding mixtures. However very
32 few reasearchers has been conducted to investigate the biogas potential of such
33 biomasses, and all the available references are focused on the anaerobic digestion of one
34 biomass type [3], [4], [5] and [6], or the co-digestion of two agro-industrial by-products
35 [7] and [8]. The Autonomous Province of Trento has a surface of approximately 6,200
36 km², equal to slightly more than 2% of the Italian territory; 20% of this surface is below
37 600 meters, about 20% is between 600 and 1,000 meters, while the remaining 60% of the
38 country lies above 1,000 meters. A real flat land does not exist in the territory, although
39 there are flat strips, more or less uncomfortable, which constitute the valley of Adige and
40 of other major streams. Even if the Region has small cultivalbe area it has a 1,633.3 t/yr
41 production of olives [1]. But Trentino Alto Adige Region is also the principal Italian
42 producer of apples [1]. A parallel market exists around olive and apple, and it consist into
43 processing the obtained by-products, such as the olive pomace and the residual material
44 that remains after the crushing of apples for the production of juice. It was demonstrated
45 that in both batch and continuous digesters olive pomace and apple pulp can be codigested
46 with manure and cattle slurry without the need of any chemicals. However, it is still

47 unknown which is the maximum organic loading rate of these two products permitted in
48 continuously operated reactors, and also if a co-digestion of three complementary
49 substrates could bring to a better result in biogas production. Furthermore, optimization
50 of the co-digestion process has not been performed. Finally, a practical aspect that is still
51 under question, is whether or not olive pomace can be quantitatively treated in existing
52 digesters of cattle-raising units and under what conditions. The objective of this study
53 was to evaluate the performance of anaerobic digestion for the treatment and biogas
54 production of different mixtures of cattle slurry, olive pomace and apple pulp. The
55 specific aims were to investigate the efficacy of semi-continuous digester at different and
56 consecutive feeding ratios under mesophilic condition, to determine the methane potential
57 and biogas production quality of different feeding mixtures, and to evaluate the overall
58 performances of the process.

59 2. Methods

60 2.1 Experimental device

61 Trials were carried out using an own designed and constructed experimental pilot
62 digester shown in Fig.1. The reactor had a 316 stainless steel tank realized by a cylinder
63 90 cm high with a diameter of 40.3 cm, closed by two top and bottom caps, for a total
64 volume of 128 l and a reaction volume of about 103 l. It was equipped with a mixing
65 system, blade propeller and a scraper on the bottom; both 316 stainless steel made and
66 activated by a variable speed electric engine. In this reactor the feed system consists in a
67 small hopper equipped with a 2" diameter pipe. This type of feed system was appropriate
68 for fed-batch loads of liquid and semi-liquid biomasses, as clogging was avoided inside
69 the pipe. Two butterfly valves were inserted along the vertical pipe in order to maintain

70 the anaerobic conditions and to stabilize the pressure inside the reactor during the feeding
71 phase. The biomass outlet was allowed through a 2" butterfly valve placed at the bottom
72 of the reactor (as visible in Fig.1). All the biomass feeding and discharging procedures
73 were done manually. The digester and the gasometer were equipped with a complete
74 probe monitoring system: a temperature probe inserted on one side of the reactor; a
75 temperature and a pressure probes placed inside in the gasometer; a pH probe inserted
76 inside the digester. The temperature was automatically controlled to remain inside
77 mesophilic range (about 35°C), the required heat was supplied by an electrical resistance
78 (15 m long). The heating cable was wrapped around the reactor and covered with
79 insulating coat. The system was also equipped with a small tank to collect the condensates,
80 designed to be emptied automatically. The upper part of the gasometer had a
81 counterweight system, realized with two pulleys, linked to a wire potentiometer to
82 measure the tank vertical displacement. The operational relative gauge pressure was about
83 9-10 mbar. The outlet pipe was equipped with a solenoid valve activated by a relay to
84 allow the automatic quick discharge of the produced biogas when the gasometer was
85 completely full. The system was already described in details in previous experiences [9]
86 and [10].

87 2.2 Feed strategy and used material

88 The adopted feed strategy was chosen as a good compromise between laboratory
89 experimentation and real scale. Indeed, inside a full scale reactor the feeding ratio are
90 changed in continuous condition. To simulate this situation was reach a compromise
91 where an initial phase 0 of the experiment was realized under batch condition using only
92 one type of biomass (cattle slurry). Then, the investigation started and a continuous
93 feeding regime was adopted using a fed-batch strategy, using all the three selected

94 biomasses under different feeding ratios. At the end was also decided to evaluate if the
95 final biomass was still active in the production of biogas and in which quantities. For this
96 reasons was realized a digestate methane yield test (performed under batch conditions),
97 that it was used for comparison with the data obtained during the Start-up.

98 The cattle slurry was collected in several sessions directly at the exit of the stable grid
99 from livestock farm, Fontanacervo, located in Villastellone (Piedmont Region, Turin,
100 Italy). Part of this biomass was used to fill the digester, and part was stored at 4°C for
101 feeding the system. The digestate used for the Start-up phase was obtained from a full
102 scale anaerobic bioreactor operating on agro-zootechnical biomasses (Biocanali s.r.l.,
103 Buriasco – TO – Italy). The olive that were harvested at the end of October to the middle
104 of December, were collected from a crusher of “Riva del Gard” (Trentino Region, Italy).
105 The process adopted for the oil extraction was the cold one, executed in batch mode.
106 About 80 kg of olive pomace were collected and stored at 4°C for feeding the system
107 during the co-digestion phases. The apple pulp was collected from a family run farm
108 located in Bleggio (Trentino Region, Italy). This kind of biomass can also be produced in
109 a fixed period of time as apple harvesting time was set between November and February.
110 About 80 kg of the remains of pressed apples coming from the production of apple juice
111 were collected and stored at 4°C. Prior to each feeding procedure the biomasses were
112 warmed to room temperature (about 22-24°C). The inlet biomasses and the outlet
113 digestate details are listed in Tables 1 and 2.

114 2.3 Start-up phase

115 A mixture of slurry and inoculum (coming from a previous digestion test) was used
116 for the beginning and the activation of the experiment, respectively 90% and 10% (w/w

117 – P0). The digester was initially filled with 80 l of mixture and was operated in batch
118 mode. The Start-up phase was conducted until the anaerobic digestion reaction started
119 and the system reached a steady state of biogas production [9]. This initial part lasted 35
120 days, the substrate was stirred every 2 days at 50 Hz (28 rpm) for about 40 min., and the
121 biogas analysis were performed at the same time.

122 2.4 Co-digestion phase

123 Co-digestion of cattle slurry, olive pomace and apple pulp was started to simulate a
124 continuous feeding condition when stable conditions were reached on day 35. This phase
125 was divided into four subsequent parts with different mixture feeding ratios. Each part of
126 the phase lasted about 33 days of fed-batch feeding, and 7 days of anaerobic rest (batch
127 condition with no further feeding). Starting from the situation describe above activation
128 stage (P0) the reactor was fed with a combination of 85% cow slurry, 10 % olive pomace
129 and 5% apple pulp (P1). Feeding was done 3 times a week for a total of 14 times. Also,
130 at the end of P0 phase the biomass volume of the mixture inside the reactor was about 80
131 l. This biomass quantity was gradually reduced to a volume of 70 l during the P1 phase,
132 for easily managed the following fed-batch phases. To decrease the total volume was
133 simply reduced the amount of the organic material introduced inside the reactor during
134 the feeding. The second phase of the co-digestion (P2) started on day 75 when biomass
135 inside the reactor was substituted with an equivalent mass of mixture (75% cattle slurry,
136 15% olive pomace and 10 apple pulp). The feeding operations were the same described
137 for the first part of the co-digestion. The third phase (P3) of the co-digestion phase was
138 performed with a biomass substitution with a combination of 65% cattle slurry, 20% olive
139 pomace and 15% apple pulp. It started on day 115 and the feeding operations were
140 performed similarly to the previous two. The fourth phase of the co-digestion (P4) started

141 on day 153 and aimed to substitute biomass with a combination of 70% cattle slurry, 20%
142 olive pomace and 10% apple pulp. This last mixture was investigated as the Province Law
143 02/05/2012, n. 8 posted on B.U. Autonomus Province of Trento n. 19 of 8/5/2012,
144 introduced a new article, 62-ter, specifically for biogas plants in agricultural areas. In this
145 article was specified that the anaerobic digestion plant must be fed mainly from manure,
146 in an amount equal to at least 70%, which must be produced by the company. The
147 remaining part can be other vegetable biomass resulting from the activities of the same
148 company or produced by farms present in the same territorial context. The feeding
149 procedures were the same of the previous co-digestion trials.

150 Substrate samples were collected at the end of every co-digestion phase for chemical
151 evaluation (Table 2). No immision of nitrogen was done inside the reactor since it was
152 observed that for low percentage (less than 1%) of oxygen in the reactor volume did not
153 adversely affect the anaerobic reaction. The substrate was stirred every time a feeding
154 operation was performed (3 times a week) for 30-45 min at 28 rpm. The pH probe and
155 the gas analyzer were checked, cleaned and calibrated at every starting part. The
156 gasometer was automatically emptied when it reached a pre-established vertical value
157 through the opening of the discharge electro valve.

158 2.5 Digestate methane yield test (DMY)

159 A Digestate Methane Yield test was realized just after the processing of the last
160 mixture (70% cow manure, 20% olive pomace and 10% apple pulp – P4). It was
161 performed after the conclusion pf co-digestion tests, on day 188. The DMY test was
162 conducted in batch condition using the biomass already inside the reactor and the
163 substrate was stirred every two days at 28 rpm for a period of about 45 min, typically

164 when biogas analysis was performed. The main control parameters were constantly
165 checked, as it was the methane concentration inside the biogas. On day 220, after 32 days
166 of detention time, the test was stopped and samples collected for the analysis (Table 2).

167 2.6 Analysis

168 Chemical analyses were performed within 48h by an independent laboratory. The
169 biogas composition and the analysis for the biomass samples for the determination of
170 BOD₅, COD, pH, density, 105°C residual, 550°C residual, volatile solids, ammonia and
171 volatile fatty acids were carried out according to the previous report [9]. The organic
172 loading rate (OLR) and the hydraulic retention time (HRT) were obtained on the basis of
173 the regular substitution of mixture inside the reactor. The C/N ratio was monitored before
174 and after every phases, and it was always inside the range 18-22/1 compatible with good
175 functionality for this type of biomasses. All the experiment was performed in wet
176 condition with a solid fraction inside the mixtures lower than 10%. The aims were to
177 follow with accuracy the different part of the co-digestion test and evaluating the
178 reaction behavior and evolution under different mixture ratios.

179 3. Results and discussion

180 3.1 Start-up phase

181 In the first 35 day period limited biogas production was observed (Fig. 2 – P0). The
182 pH value started from 7.2, reached 8.1 around day 14th and stabilized around 7.8 for the
183 rest of the Start-up phase. The total biogas volume produced was equal to 878 l (Fig. 2 –
184 P0). The CH₄ proportion inside the mixture was 56.59%, for a 497.2 l total volume of
185 methane production. A total of 3.9 kg of VS were processed inside the reactor.
186 Consequently the methane potential of this Start-up phase was equal to 126.9 l-CH₄/kg-

187 VS. The digestion followed the expected steps and the trend of biogas production was
188 similar to trends observed previously in similar studies [10] where the methane potential
189 was 119.17 l-CH₄/kg-VS. Amon et al. [11] found a specific methane yield between 125.5
190 and 166.3 l-CH₄/kg-VS. Braun et al. [12] reported a range between 140 and 266 l-
191 biogas/kg-VS and also Thomè-Kozmiensky [13] and Brachtl [14] found biogas yields
192 between 200 and 300 l-biogas/kg-VS. All these ranges are compatible with the Start-up
193 phase that, gave a value equal to 224.3 l-biogas/kg-VS.

194 3.2 Co-digestion phase

195 The OLR of the different mixtures ranged from 2.75 (P4) to 3.34 (P3) g-VS/l-d (Table
196 3) as a consequence of the increase of olive pomace portion in the feeding. The pH values
197 remained between 7.7 – 8.1, which are fully compatible with the optimal working range
198 after the stabilization obtained in the Start-up phase. The biogas production is presented
199 in Fig. 2 (P1-P2-P3-P4 series). The daily biogas yield shows a very similar trend for P1
200 and P2 mixtures (Fig. 2). P3 also shows a good yield behavior. By contrast, the last part
201 of the co-digestion phase (P4) shows a great difference from the P1 and P2 series, with
202 half the production. All the trends were analyzed, and constant growth rates were
203 observed for almost the entire duration of feeding Subsequently, a progressive and regular
204 biogas yield decrease was recorded, which dropped after about 40 days. In all the stages
205 of the test, the percentage of methane in biogas gradually increased. The highest value
206 was reached typically at the beginning of the second week when the microbiota had
207 adapted to the new mixture. Fig. 2 shows that the CH₄ values were stable between 50-
208 60%. The P1 mixture gave the greatest specific yields -396 l biogas/kg SV and 216 l
209 CH₄/kg SV- but interesting results were also obtained with the P2 mixture that gave a
210 specific yield of 342.5 l biogas/kg SV and 189 l CH₄/kg SV. This was unexpected

211 behavior that can be summarized as very similar to or better than the P1 mixture for the
212 whole feeding period, with minimal decreases only during the feeding rest period (Fig.
213 2). The P3 mixture also gave a specific yield, not so different from that obtained with the
214 previous two combinations, 254 l biogas/kg SV and 141 l CH₄/kg SV. The last mixture
215 (P4) that gave the smallest specific yield of all the whole co-digestion phase started with
216 values of 211 l biogas/kg SV and 116 l CH₄/kg SV.

217 The present investigation shows that anaerobic digestion of cattle slurry, olive pomace
218 and apple pulp can be achieved with good methane yield with a 75:15:10 ratio. Even with
219 an increase of olive pomace and apple pulp to 65:20:15, the level of production of biogas
220 is quite near to the results obtained with the optimum ratio. Slight instability was observed
221 only during the P4 feeding phase. Just after day 4 the P4 mixture became less productive
222 than the P3 mixture, and the total biogas volume produced was 1,655 l (40% less
223 compared to the P3 series). The reasons of this big difference in biogas production could
224 be explained by an accumulation of lipids and polyphenols that were difficult to degrade
225 and may have inhibited certain microbial groups [15].

226 The P4 co-digestion phase started with an inlet mixture of a 70% slurry fraction, of
227 20% olive pomace and a 10% fraction of apple pulp, with a COD value equal to 92.5 g/l,
228 an OLR of 2.56 g-COD/l-d and HRT of 36 days with a COD reduction of 55.5%. All the
229 COD reductions are shown in Table 3. Very few experiments have been conducted on co-
230 digestion of two of the biomasses used (typically slurry and apple pulp, more rarely slurry
231 and olive pomace) and no references have been found to investigate the tested mixture.
232 During trials with several test combinations of apple waste and swine manure co-
233 digestion, Kafle and Kim [8] found a similar methane yield both for batch- and continuous
234 feeding. Llaneza Coalla et al. [7] reported higher methane yield in digestion of different

235 apple pulp tests, but without the use of co-digestion with other biomasses. These authors
236 observed that the $\text{NH}_4^+\text{-N}$ quantity inside the reactor led to a critical accumulation inside
237 the reactor (over 2,500 mg/l). A different situation is reported by Tekin and Dalgıç [6] for
238 the production of methane from olive pomace alone, where high concentrations of fat and
239 the presence of other insoluble compounds led a low yield value. Comparing the methane
240 yield obtained the co-digestion experiments described in this present paper with the data
241 collected by Dinuccio et al. [16] on several agro-industrial single biomasses reveals
242 relevant data. Only whey, 501 l $\text{CH}_4/\text{kg SV}$, that can not be digested without chemical
243 pH correction, and dried maize residues, 317 l $\text{CH}_4/\text{kg SV}$, achieved better values. The
244 substrate that obtained the best production performance was the P1 mixture (85% cattle
245 slurry, 10% olive pomace and 5% apple pulp). Compared with the specific methane yield
246 of the Start-up phase (P0 – only cattle slurry) it rendered an increase in production of
247 about 70%. The P2 combination (75% cattle slurry, 15% olive pomace and 10% apple
248 pulp), that achieved higher OLR and biogas quality then the P1 during the experiment,
249 gave a 48% increase in methane specific yield if compared with the Start-up phase. These
250 results confirm that the co-digestion of these substrates succeed in co-metabolism and
251 strongly contribute to reduce the effect of inhibitory factors. The P1 mixture yield makes
252 it possible to obtain an electricity production of about 2.1 kWhr per t/d (considering a
253 CHP technology with 36% of efficiency).

254 3.3 Digestate Methane Yield Test

255 The digested biomass was used to performed a Digestate Methane Yield test at the
256 end of phase P4, as described in Section 2.5. The OLR was 0.79 g-VS/l-d, with a 455 l of
257 produced total biogas (Fig. 2). Biogas samples collected during the test, led to an average
258 CH_4 proportion of 51.3%, and with this value the amount of methane inside the biogas

259 volume corresponded to 233.4 l. The methane yield was 93.5 l CH₄/kg SV obtained using
260 a quantity of VS (2.5 kg) calculated using the chemical analysis of the initial digestate.
261 The DMY test showed a poor biogas and methane production if compared with similar
262 studies that used different co-digested substrates [17], [18] and [10]. The main process
263 parameters were both very low as visible in Table 3. In experiments conducted in the past
264 it was observed that digestate can still yield an important amount of biogas. In the DMY
265 test describe in this present paper the obtained results were relevant if compared with the
266 Start-up phase. The cumulative curve of both Start-up phase and DMY test can be
267 observed in Fig. 2. The total biogas volume obtained from the DMY test is about the half
268 of what obtained from the digestion of only cattle slurry. The comparison of the methane
269 yield between the two phases showed a decrease of only the 26% between the Start-up
270 and the DMY. The biogas recovered from the digestate could represent a sensible
271 contribution to the global energy balance. Indeed, with the above values was possible to
272 obtain an electricity production of 0.3 kW per t/d (batch digestion and CHP technology
273 with an efficiency of 36%).

274 4. Conclusion

275 The results obtained in this study show that the production of methane by co-digestion
276 of cow slurry, olive pomace and apple pulp is feasible and economically attractive. The
277 P1 and P2 mixtures are very productive and show a very similar biogas production
278 behavior. Infact the methane yields in the experiment performed were equal to 216.3 and
279 189.4 l CH₄/kg SV with an OLR of 2.75 and 3.01 g-VS/l-d respectively. The energy
280 potential of this mixture is reasonable near to energy crop and livestock combinations,
281 and could be used to cost-effectly solve a waste problem in Trentino.

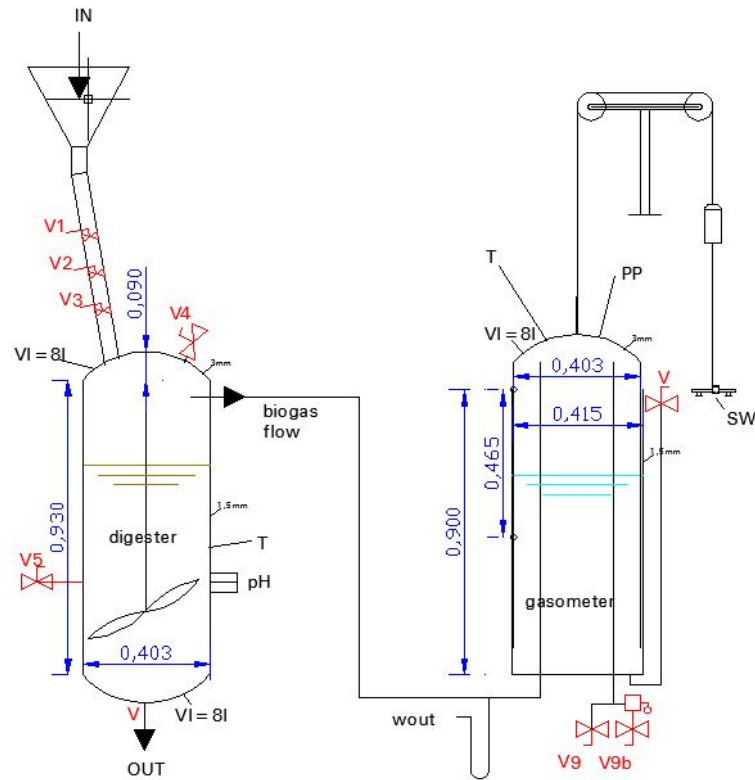
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Figure 1



LEGEND

digester: anaerobic reactor
 V: valve
 T: temperature probe
 pH: pH mobile probe
 IN: mix inlet
 OUT: digestate outlet

SCALE 1:10

wout: condensate drain
 PP: pressure probe
 SW: slide-wire potentiometer
 gasometer: two sections
 biogas tank

Figure 2

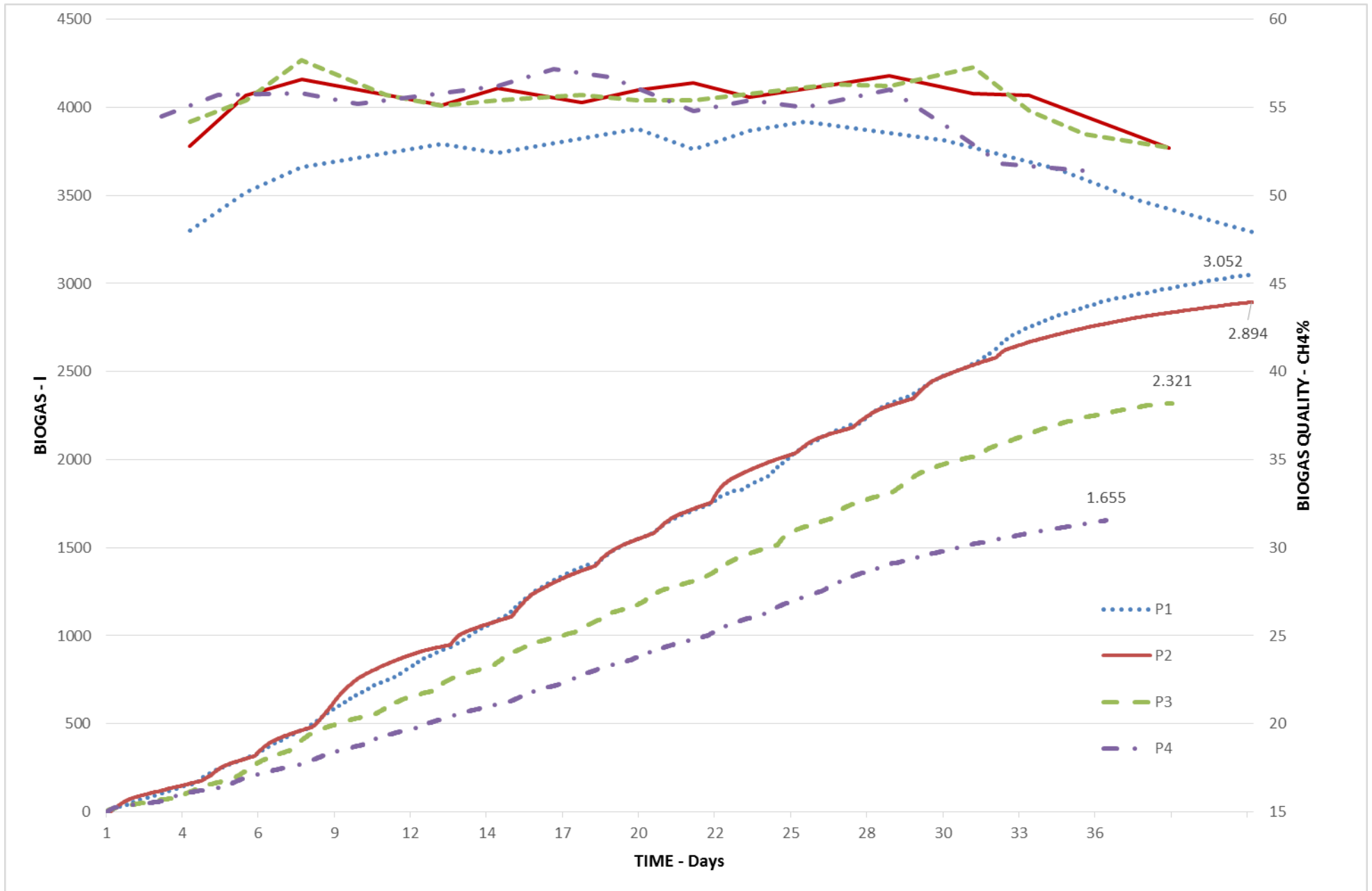


Figure 3

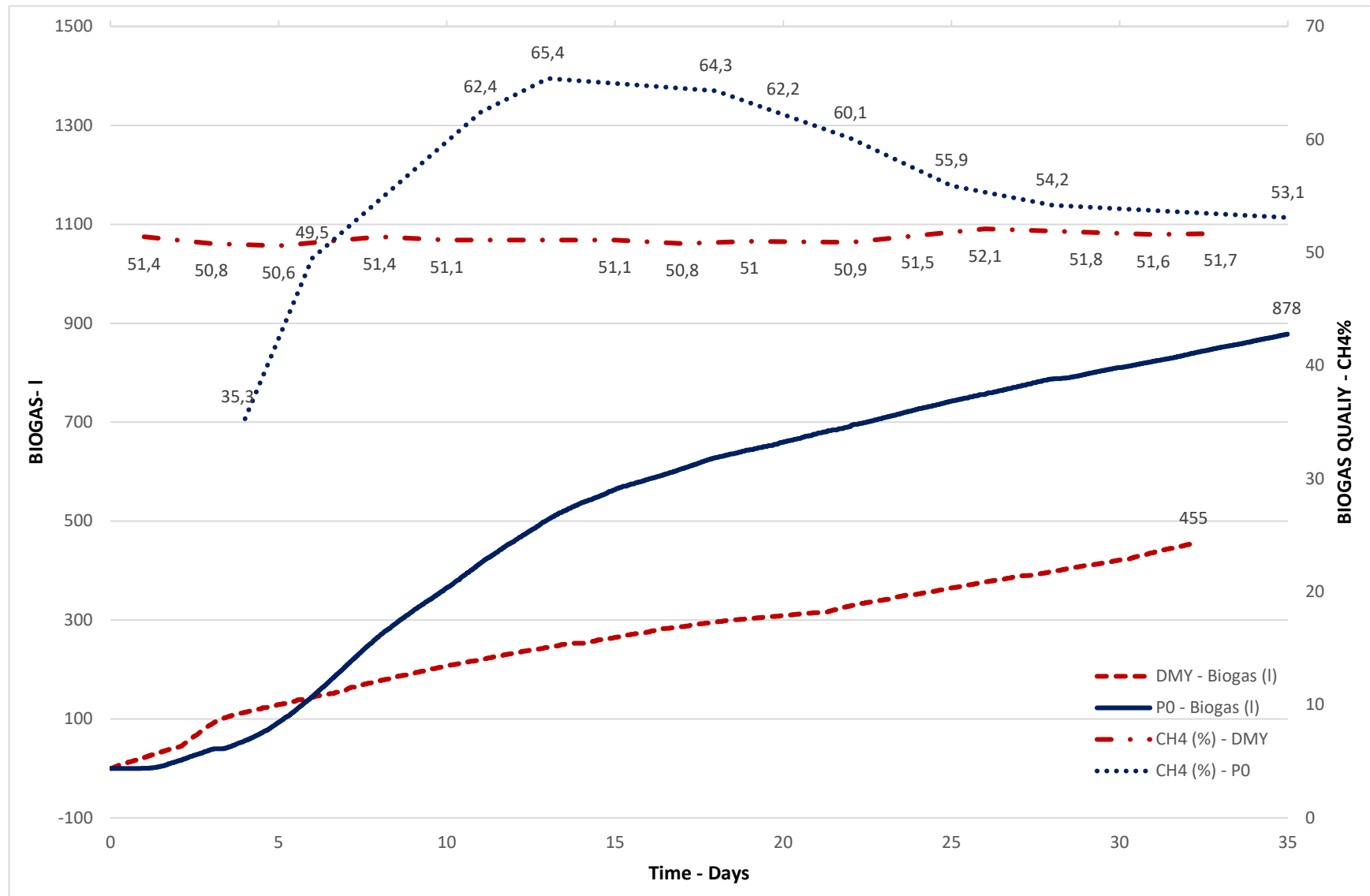


FIGURE CAPTIONS

Figure 1 – Technical scheme of the anaerobic digester reactor used during the experiment with all the main components.

Figure 2 – BIOGAS PARAMETERS OF CO-DIGESTION PHASES - Lower graphs: comparison of biogas production for the four tested phases and the Start-up. First phase P1 with 85% cattle slurry – 10% olive pomace – 5% apple pulp, second phase P2 with 75% cattle slurry – 15% olive pomace – 10% apple pulp, third phase P3 with 65% cattle slurry – 20% olive pomace – 15% apple pulp, fourth phase P4 with 70% cattle slurry – 20% olive pomace – 10% apple pulp and Start-up phase P0 with only cattle slurry. Higher graphs: methane quality inside the biogas mixture for the different feeding phases.

Figure 3 - BIOGAS PARAMETERS OF START-UP AND DMY PHASES - Lower graphs: comparison of biogas production for the Start-up phase and the digestate methane yield test (DMY). Higher graphs: methane quality inside the biogas mixture for the Start-up phase and the digestate methane yield test.