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

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## Abstract

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

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

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## 1. Introduction

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

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

## 2. Methods

### 2.1 Experimental device

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

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

## 2.2 Feed strategy and used material

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

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

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

### 2.3 Start-up phase

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

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

## 2.4 Co-digestion phase

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

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

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

## 2.5 Digestate methane yield test (DMY)

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



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

## 2.6 Analysis

Chemical analyses were performed within 48h by an independent laboratory. The biogas composition and the analysis for the biomass samples for the determination of BOD<sub>5</sub>, COD, pH, density, 105°C residual, 550°C residual, volatile solids, ammonia and volatile fatty acids were carried out according to the previous report [9]. The organic loading rate (OLR) and the hydraulic retention time (HRT) were obtained on the basis of the regular substitution of mixture inside the reactor. The C/N ratio was monitored before and after every phases, and it was always inside the range 18-22/1 compatible with good functionality for this type of biomasses. All the experiment was performed in wet condition with a solid fraction inside the mixtures lower than 10%. The aims were to follow with accuracy the different part of the co-digestion test and evaluating the reaction behavior and evolution under different mixture ratios.

## 3. Results and discussion

### 3.1 Start-up phase

In the first 35 day period limited biogas production was observed (Fig. 2 – P0). The pH value started from 7.2, reached 8.1 around day 14th and stabilized around 7.8 for the rest of the Start-up phase. The total biogas volume produced was equal to 878 l (Fig. 2 – P0). The CH<sub>4</sub> proportion inside the mixture was 56.59%, for a 497.2 l total volume of methane production. A total of 3.9 kg of VS were processed inside the reactor. Consequently the methane potential of this Start-up phase was equal to 126.9 l-CH<sub>4</sub>/kg-

VS. The digestion followed the expected steps and the trend of biogas production was similar to trends observed previously in similar studies [10] where the methane potential was 119.17 l-CH<sub>4</sub>/kg-VS. Amon et al. [11] found a specific methane yield between 125.5 and 166.3 l-CH<sub>4</sub>/kg-VS. Braun et al. [12] reported a range between 140 and 266 l-biogas/kg-VS and also Thomè-Kozmiensky [13] and Brachtl [14] found biogas yields between 200 and 300 l-biogas/kg-VS. All these ranges are compatible with the Start-up phase that, gave a value equal to 224.3 l-biogas/kg-VS.

### 3.2 Co-digestion phase

The OLR of the different mixtures ranged from 2.75 (P4) to 3.34 (P3) g-VS/l-d (Table 3) as a consequence of the increase of olive pomace portion in the feeding. The pH values remained between 7.7 – 8.1, which are fully compatible with the optimal working range after the stabilization obtained in the Start-up phase. The biogas production is presented in Fig. 2 (P1-P2-P3-P4 series). The daily biogas yield shows a very similar trend for P1 and P2 mixtures (Fig. 2). P3 also shows a good yield behavior. By contrast, the last part of the co-digestion phase (P4) shows a great difference from the P1 and P2 series, with half the production. All the trends were analyzed, and constant growth rates were observed for almost the entire duration of feeding. Subsequently, a progressive and regular biogas yield decrease was recorded, which dropped after about 40 days. In all the stages of the test, the percentage of methane in biogas gradually increased. The highest value was reached typically at the beginning of the second week when the microbiota had adapted to the new mixture. Fig. 2 shows that the CH<sub>4</sub> values were stable between 50-60%. The P1 mixture gave the greatest specific yields -396 l biogas/kg SV and 216 l CH<sub>4</sub>/kg SV- but interesting results were also obtained with the P2 mixture that gave a specific yield of 342.5 l biogas/kg SV and 189 l CH<sub>4</sub>/kg SV. This was unexpected

behavior that can be summarized as very similar to or better than the P1 mixture for the whole feeding period, with minimal decreases only during the feeding rest period (Fig. 2). The P3 mixture also gave a specific yield, not so different from that obtained with the previous two combinations, 254 l biogas/kg SV and 141 l CH<sub>4</sub>/kg SV. The last mixture (P4) that gave the smallest specific yield of all the whole co-digestion phase started with values of 211 l biogas/kg SV and 116 l CH<sub>4</sub>/kg SV.

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

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

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

### 3.3 Digestate Methane Yield Test

The digested biomass was used to performed a Digestate Metahen Yield test at the end of phase P4, as described in Section 2.5. The OLR was 0.79 g-VS/l-d, with a 455 l of produced total biogas (Fig. 2). Biogas samples collected during the test, led to an average  $\text{CH}_4$  proportion of 51.3%, and with this value the amount of methane inside the biogas

volume corresponded to 233.4 l. The methane yield was 93.5 l CH<sub>4</sub>/kg SV obtained using a quantity of VS (2.5 kg) calculated using the chemical analysis of the initial digestate. The DMY test showed a poor biogas and methane production if compared with similar studies that used different co-digested substrates [17], [18] and [10]. The main process parameters were both very low as visible in Table 3. In experiments conducted in the past it was observed that digestate can still yield an important amount of biogas. In the DMY test describe in this present paper the obtained results were relevant if compared with the Start-up phase. The cumulative curve of both Start-up phase and DMY test can be observed in Fig. 2. The total biogas volume obtained from the DMY test is about the half of what obtained from the digestion of only cattle slurry. The comparison of the methane yield between the two phases showed a decrease of only the 26% between the Start-up and the DMY. The biogas recovered from the digestate could represent a sensible contribution to the global energy balance. Indeed, with the above values was possible to obtain an electricity production of 0.3 kW per t/d (batch digestion and CHP technology with an efficiency of 36%).

#### 4. Conclusion

The results obtained in this study show that the production of methane by co-digestion of cow slurry, olive pomace and apple pulp is feasible and economically attractive. The P1 and P2 mixtures are very productive and show a very similar biogas production behavior. Infact the methane yields in the experiment performed were equal to 216.3 and 189.4 l CH<sub>4</sub>/kg SV with an OLR of 2.75 and 3.01 g-VS/l-d respectively. The energy potential of this mixture is reasonable near to energy crop and livestock combinations, and could be used to cost-effectly solve a waste problem in Trentino.

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Figure 1

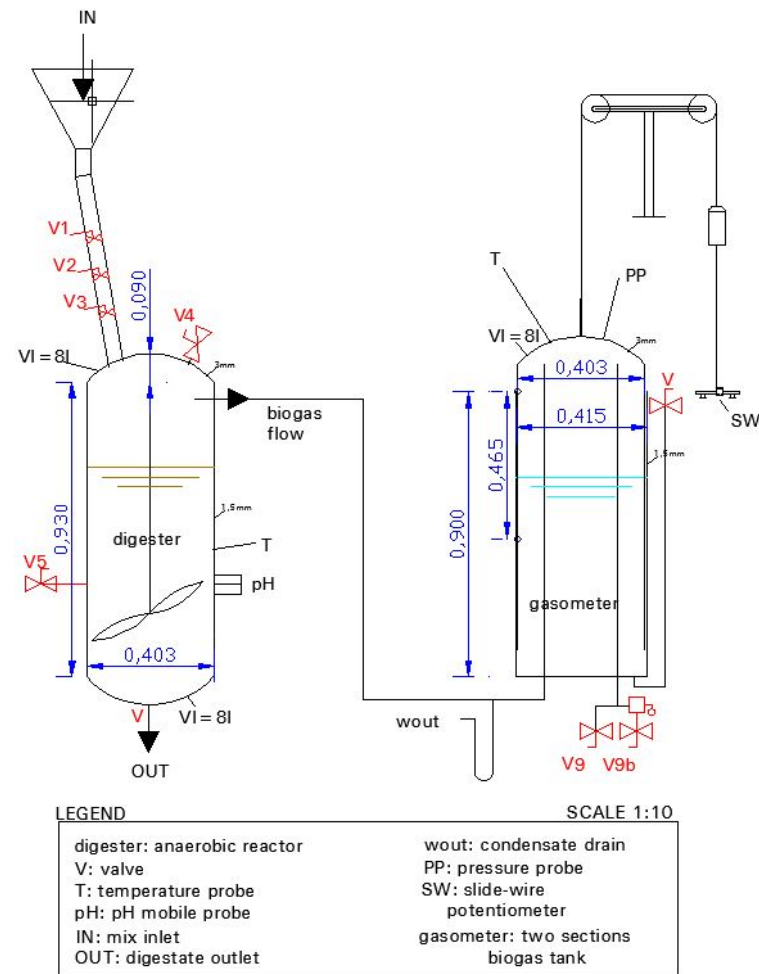


Figure 2

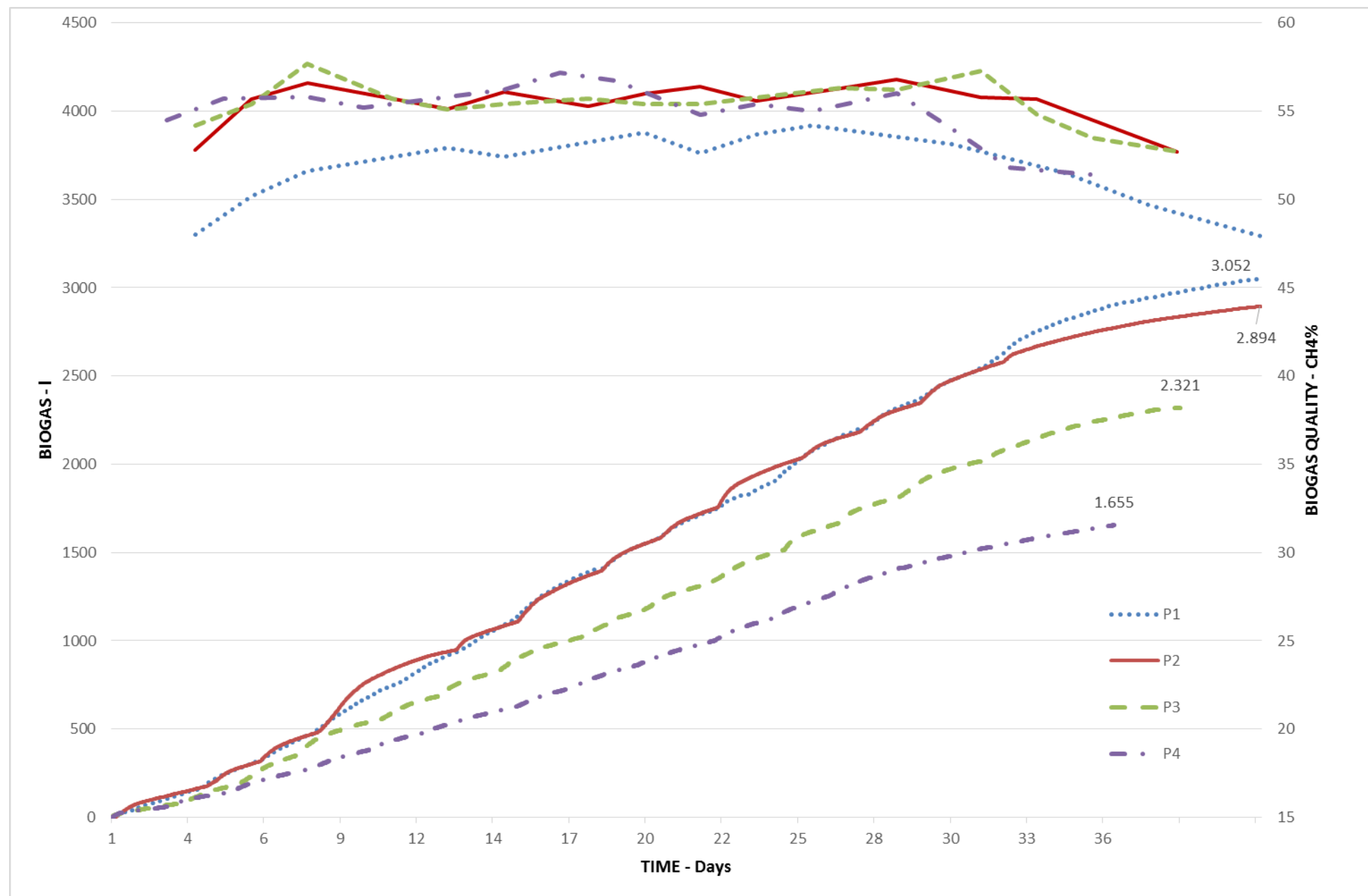
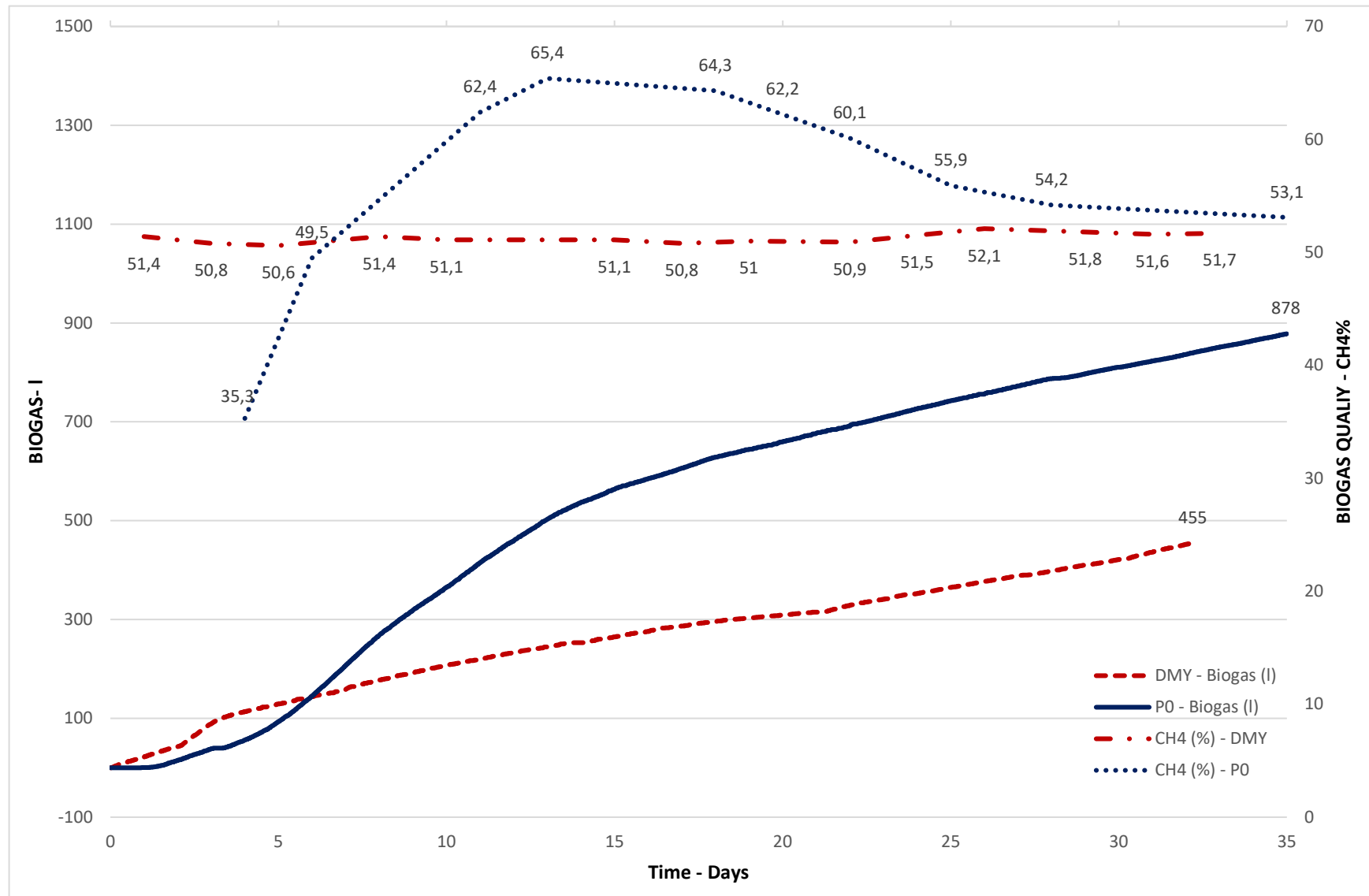


Figure 3



## 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.