

Full Length Research Paper

# Reuse of anaerobic reactor effluent on the treatment of poultry litter

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Received 2 November, 2016, Accepted 13 December, 2016

Anaerobic digestion of poultry litter was studied with reutilization of its effluent in the process by pumping into reactor feeding, contributing to the moisture content and making part of the feeding organic load: 0.5 and 1.0 kg VS/m<sup>3</sup>/day, at evaluations 1 and 2, respectively. The hydraulic residence time lasted 10 days for both evaluations and the useful volume of reactor was 35 m<sup>3</sup>, with a semi-continuous reactor feeding, under field conditions. The stability of anaerobic digestion was verified through Shewhart control chart. Average efficiency of biogas production was 0.0119 m<sup>3</sup>/(kg VS<sub>added</sub>) at evaluation 1 and 0.0429 m<sup>3</sup>/(kg VS<sub>added</sub>) at evaluation 2. In the second evaluation, the study revealed that biogas produced more energy as methane than spent with electric energy in reactor feeding. According to Lower Process Capability Index (C<sub>pl</sub>), measure developed for convenience engineering to quantify the performance of a process, the anaerobic digestion in the second evaluation was capable in its energy operations.

**Key words:** Biogas, Lower Process Capability Index, operational energy viability index, Shewhart control chart, statistical process control.

## INTRODUCTION

The intensive production system for broiler production has promoted poultry industry in Brazil, which is the world's third largest producer according FAOSTAT database (FAO, 2015), but also brought on generation of large amounts of waste, poultry litter (PL) and dead birds. PL is composed of animal waste and the material used as bed for broilers (e.g., wood shavings), dietary waste (Sharma et al., 2013) and broiler feathers. As there are high concentrations of poultry farms in producing regions,

it would be an attractive alternative to farmers finding different applications for such residue, despite its direct use as fertilizer on soil. In this context and considering current environmental problems related to global warming, anaerobic digestion of solid wastes has attracted more interest (Nasir et al., 2012). Anaerobic digestion has been successfully used in many applications and has conclusively demonstrated its ability to recycle biological wastes biomass (Dahiya and Joseph, 2015). Its scope

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has been spread in a wide range of operating conditions: the process is implemented at psychrophilic, mesophilic, and thermophilic temperatures, and even extreme conditions like high salt concentrations can currently effectively be tolerated in anaerobic reactors provided that adequate operational measures are taken (Kleerebezem et al., 2015).

According Labatut et al. (2014), the temperature and influent substrate may be the most important parameters determining performance and stability of the anaerobic digestion process. However, to heat the feedstock for anaerobic digestion is need the source of power.

The C/N ratio is an important indicator for controlling biological treatment systems (Wang et al., 2012). However, the optimum C/N range in feedstock for anaerobic digestion remains highly debated, although 20/1 to 30/1 is a most acceptable range (Zhong et al., 2012).

The methanogenic bacteria involved in AD have a low growth rate and are sensitive to inhibitors such as low pH caused by excessive concentrations of volatile fatty acids (VFA) (Brown et al., 2012; Jiang et al., 2012). The pH value increases by ammonia accumulation during degradation of proteins, while the accumulation of VFA decreases the pH value (Weiland, 2010). However, the pH also depends on the buffer capacity of the substrate.

There is also a wide variety of inhibitory substances are the primary cause of anaerobic digestion failure, since they are present in substantial concentrations in wastes, as ammonia, sulfide, light metal ions (Na, K, Mg, Ca e Al), heavy metals, and organics (Chen et al., 2008). However, such inhibitors are not controlled in most anaerobic digestion processes under field conditions because of the difficulty and complexity of the determination of these substances. Therefore, the process design must be well adapted to the substrate properties for achieving a complete degradation without process failure (Weiland, 2010).

A limitation for anaerobic digestion of PL is its low moisture content (about 20 to 40%), relative to the water amount required for the process (about 90-94%). This problem can be solved with the liquid waste anaerobic co digestion or with the mixture with fresh water. For example, studies have been reported on anaerobic co-digestion of PL and stillage (Sharma et al., 2013), on anaerobic co digestion of PL and carcasses of dead birds (Orrico Júnior et al., 2010), on anaerobic digestion from PL with water for biogas production (Espinosa-solares et al., 2009; Gangagni Rao et al., 2013; Markou, 2015). However, there are environmental concerns with the use of fresh water to treat waste.

Other alternative for this would be the process effluent reuse with PL into substrate mixture of the reactor feeding, which contributes also to recirculate the microorganisms and to take advantage of the organic load of effluent by the process of effluent recirculating in the reactor. So, to recirculate the effluent with PL for inlet

feedstock in reactor by pumping also allows circulating partially the slurry in reactor.

Thereby, this study aims at evaluating the effluent reuse of the PL anaerobic digestion in the process to dilute the PL in the reactor feeding, on a pilot scale.

## MATERIALS AND METHODS

### Poultry litter (PL)

The PL under study consists of wood shavings, saw dust, poultry manure and feathers remains, obtained from poultry houses and a result from 13 lots of 45 fattening days of broilers with an 11-day interval.

### Treatment system

This trial was carried out in a rural farm in Francisco Beltrão city, in Parana, Brazil, Latitude 25° 59'1.18" S and Longitude 53 ° 6'10.37" W.

The PL treatment system was formed by three units, according to Figure 1: Station 1, PL storage in a shed; station 2, PL anaerobic digestion; station 3, three effluent storage tanks. Each tank contained a hydraulic stirring system.

The horizontal reactor was formed by the union of two fiberglass boxes, with dimensions 3.60 m x 3.30 m x 2.60 m (largest diameter x smallest diameter x height) and then it was placed in horizontal direction within a 2.80 m-depth trench. PVC pipes of 200 mm diameter were connected on each side of the boxes for the inlet/outlet of the reactor.

### Inoculum

The anaerobic digestion was started with 3 m<sup>3</sup> of inoculum from reactor of swine wastewater plus 32 m<sup>3</sup> of PL diluted in water at 0.5% volatile solids (VS). The total and useful volumes of reactor were 40 and 35 m<sup>3</sup>, respectively.

### Operational procedures

Two feeding organic load were evaluated with the stabilized reactor, 0.5 and 1.0 (kg VS)/m<sup>3</sup>/day during 142 to 174 days and 210 to 241 days, forming evaluation 1 and 2, respectively. Since, the evaluation period was determined by period when anaerobic digestion was considered stable.

The reactor feeding volume was set at 3.5 m<sup>3</sup>/day and controlled by calibrated volumetric graduation in a flow control box, corresponding to 10 days of hydraulic residence time (HRT). Thus, according to feeding organic load and the feeding daily flow rate and the useful volume of reactor, the reactor was feeding daily with 17.5 and 35 kg VS/day, respectively.

The PL was used as complement of effluent during reactor feeding or to make part of the feeding organic load, since PL amount depended on VS effluent content. The effluent was reused to feed the reactor with a new amount of PL, according to Figure 2. So, for each reactor feeding, the effluent and PL stored an amount that could supply almost one reactor feeding were characterized, according to Figure 3.

Analyses regarding characterization were carried out in triplicate and daily obtained to determine the total solids (TS) and VS contents of effluent and PL. Prior to reactor feeding the stored effluent was stirred in order to prevent the supernatant build-up in

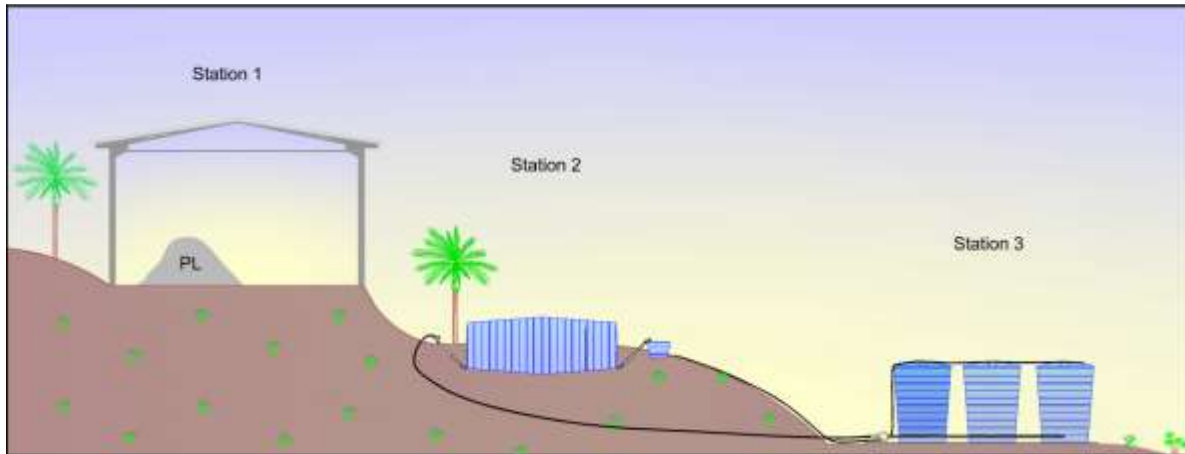


Figure 1. Poultry litter system treatment.

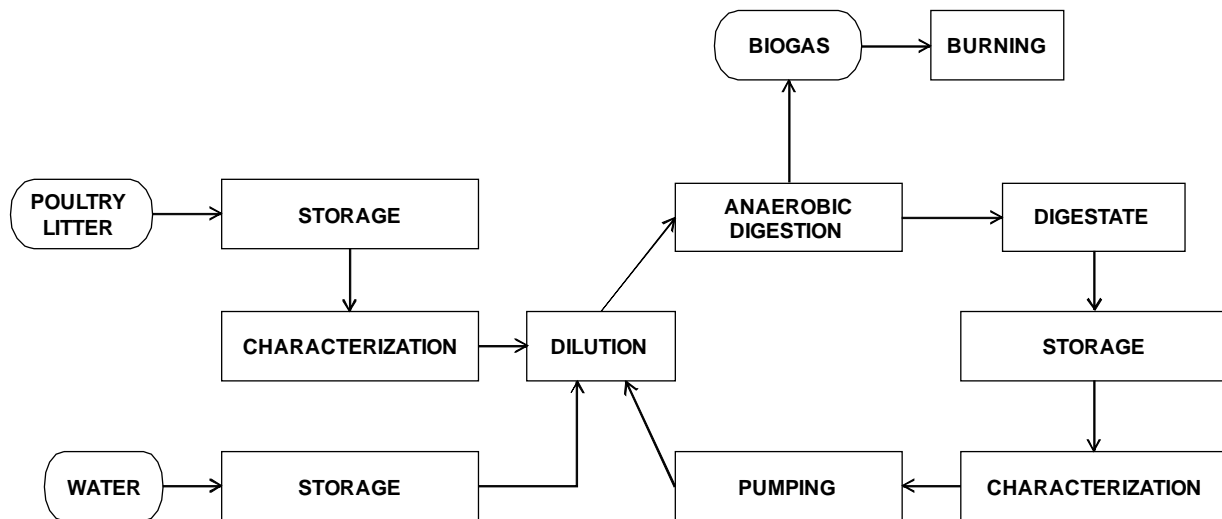


Figure 2. Flow chart of anaerobic digestion system.

the tank.

During the evaluations, data as pH and electrical conductivity of effluent, minimum and maximum room and anaerobic digestion temperatures as well as the TS and VS reduced content of effluent were periodically monitored.

The volume of the biogas was daily quantified by a gas meter LAO brand (model G 0.6) and corrected for Standard Temperature and Pressure of  $10^5$  Pa and  $0^\circ\text{C}$ . In each evaluation, biogas samples were tested with the analyzer GEM 5,000 Plus, Landtec brand, to investigate the concentrations of methane ( $\text{CH}_4$ )

#### Analytical methods

In order to analyze physicochemical parameters, the procedures described by APHA (1998) were applied for TS (2540B Method) and VS (2540E method) and by Silva (1977) to obtain volatile fatty acidity, total and partial alkalinity and pH.

#### Stability of anaerobic digestion

The reactor was stabilized according to biogas production and considered as so when it was under statistical process control by Shewhart control chart for individual measurements, with three average standard deviations, created in MINITAB® 17.1.0 (2013) software, according to Montgomery (2009). Prior to the creation of Shewhart control chart, its assumptions were tested in the variables analysis: Normality by Anderson Darling test (5% significance), sample independence by autocorrelation graph (5% significance and limits of two standard deviations) and sample randomness, observed in the Shewhart control chart.

Among the checking criteria of non-random patterns of control charts, some were chosen to determine the stability process: One or more points outside of the control limits (three average standard deviations); eight points in a row on both sides of the center line with none point inside one average standard deviations; and six points in a row steadily increasing or decreasing (Montgomery, 2009).

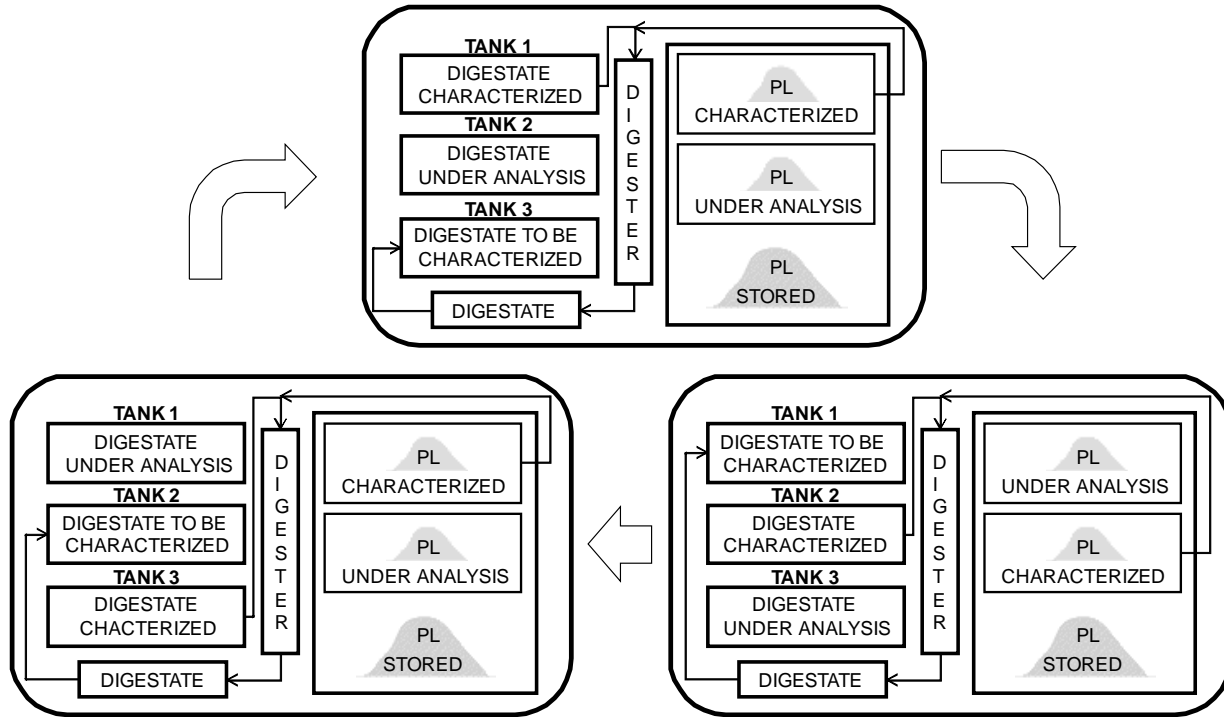


Figure 3. Reactor feeding system with poultry litter (PL) and effluent.

The ratio between VFA, total alkalinity (TA) and partial alkalinity (PA) were also monitored to obtain a better record of the process concerning the reactor potential withstand the evaluated loads.

**Data analysis**

As this process requires energy, a new index was created in order to relate the energy produced as methane ( $E_{produced}$ ) by the electric energy expended to stir and to pump the effluent into reactor feeding system ( $E_{expended}$ ): Average Index of Operational Viability (AIOEV) shown in Equation 1.

$$AIOEV = \frac{E_{produced}}{E_{expended}} = \frac{P_{biogas} \cdot [ ] CH_4 \cdot \rho \cdot c}{H \cdot P} > 1 = \text{feasible} \quad (1)$$

Where:  $P_{biogas}$  = average production of biogas ( $m^3/day$ );  $[ ] CH_4$  = average concentration of methane (percentage rate, volume);  $\rho$  = lower heating value of methane ( $CH_4$ ), equal to 50,156 J/g  $CH_4$  (Rendeiro et al., 2008);  $H$  = number of daily hours of pumping operation (h/day);  $P$  = power of effluent recirculation pump: 5 Hp \* 746 J/s/Hp = 3,730 J/s;  $c$  = constant,  $[16 \text{ g } CH_4/mol \cdot (1,000 \text{ L}/m^3 / 22.4 \text{ L}/mol) / 3,600 \text{ s}/h] = 0.1984 \text{ g}/m^3$ .

The Lower Process Capability Index ( $C_{pl}$ ) was also used to check the process capability in each evaluation for a Lower Specification Limit of biogas production ( $P_{biogas}$ ), which was determined prior to  $C_{pl}$  calculation with methane content value and the operating hours of the pump, respectively to the ones obtained during the evaluations, according to Equation 2.

$$LSL = \frac{P_{biogas}}{VS_{added}} = \frac{H \cdot P}{1 \cdot [ ] CH_4 \cdot \rho \cdot c \cdot VS_{added}} \quad (2)$$

Where: LSL = Lower Specification Limit to  $P_{biogas}$  ( $m^3/kg VS_{added}$ );  $P_{biogas}$  = average biogas production ( $m^3/day$ );  $VS_{added}$  = amount of added volatile solids (kg VS/day);  $[ ] CH_4$  = average methane concentration, percentage rate (volume);  $\rho$  = lower heating value of methane ( $CH_4$ ), equal to 50,156 J/g  $CH_4$  (Rendeiro et al., 2008);  $H$  = number of daily hours of pumping operation (h/day);  $P$  = power of effluent recirculation pump: (5 Hp \* 746 J/s/Hp = 3,730 J/s);  $c$  = constant,  $[16 \text{ g } CH_4/mol \cdot (1,000 \text{ L}/m^3 / 22.4 \text{ L}/mol) / 3,600 \text{ s}/h] = 0.1984 \text{ g}/m^3$ .

So, in order to determine  $C_{pl}$ , the Lower Specification Limit (LSL) was determined by  $P_{biogas}$  resulting in an AIOEV equal to one, value that relates the limit in which the process is feasible in its energy operations, according to Equation 3.

$$C_{pl} = \frac{\bar{X} - LSL}{k \sigma} \quad (3)$$

Where:  $C_{pl}$  = Lower Process Capability index;  $\bar{X}$  = sampling average to  $P_{biogas}$  ( $m^3/kg VS_{added}$ ); LSL = lower specification limit to  $P_{biogas}$  ( $m^3/kg VS_{added}$ );  $k$  = number of sampling standard deviations;  $\sigma$  = sampling standard deviation to  $P_{biogas}$  ( $m^3/kg VS_{added}$ ).

Finally, the classifications were associated to the process according to  $C_{pl}$  and AIOEV.

**RESULTS AND DISCUSSION**

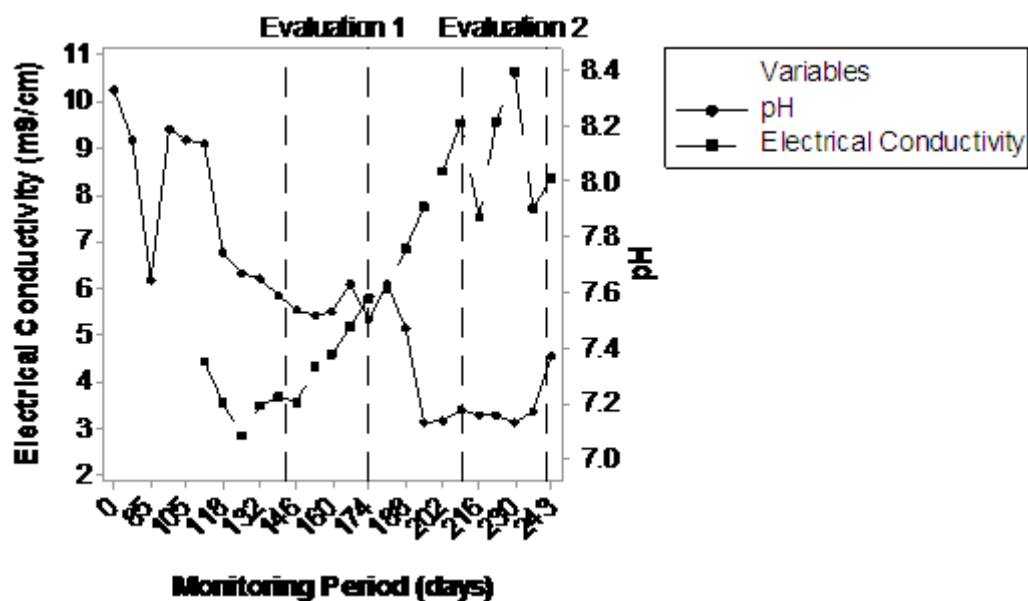
**Process monitoring**

Differences of pH, VFA and alkalinity between PL and inoculums were observed, according to Table 1.

However, this did not cause instability in process during

**Table 1.** Values of pH and of the ratio between volatile fatty acidity (VFA), total alkalinity (TA) and partial alkalinity (PA) from poultry litter and Inoculum.

Material	pH	VFA/PA	VFA/TA	PA/TA
Poultry litter	6.76	5.29	0.39	0.07
Inoculum	8.33	0.24	0.09	0.38



**Figure 4.** Values of pH and electrical conductivity of effluent.

its start because, for the following parameter concerning reactor operation, effluent pH remained similar to the inoculum (superior to 8.15) until 78 days, in accordance with Figure 4. The gradual increase of feeding organic load rate during the 113 initial days contributed to this answer. Methane production occurred at 135<sup>th</sup> day, fact observed by the biogas burning.

According Zuo et al. (2013), effluent recirculation from the methanogenic stage to the acidic stage can help buffer the rapidly produced VFA from hydrolysis and maintain a suitable pH, which was characteristic this process.

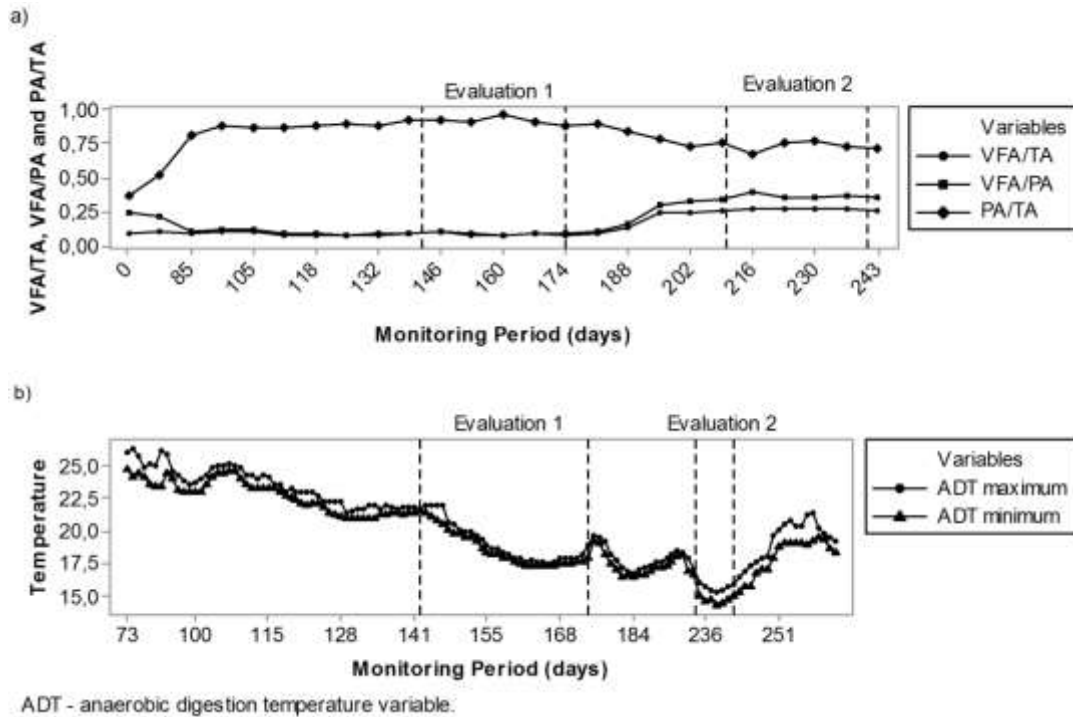
Unlike pH, electric conductivity tended to increase until the beginning of evaluation 2. The light metal ions including sodium, potassium, calcium, and magnesium are present in the influent of anaerobic reactors (Chen et al., 2008), therefore the increase of electric conductivity is due to effluent recirculation into reactor and by addition daily of PL in process, which contributes to the accumulation of salts inside the reactor. VFA/PA, VFA/TA and PA/TA rates presented the lowest fluctuations during the periods of evaluations 1 and 2. This fact has indicated a stable process during the evaluations. According to Zickefoose and Hayes (1976),

VFA/TA ratio can vary from less than 0.1 to almost 0.35 without any significant changes in digestion. Volatile fatty acidity and alkalinity rates are commonly used to verify the anaerobic digestion stability, however, in this study the rates do not express differences between the period that did have the feeding organic load increase (unstable) and the period that did have a single feeding organic load (stable), according to Figure 5a. This highlights the importance to use the Shewhart control chart to verify the anaerobic digestion stability.

According to variation range regarding daily values of maximum and minimum anaerobic digestion temperature, the highest answer was 2.3°C, so, there was a good thermal stability in the process. Reactor design kept stable the anaerobic digestion temperature, because daily room temperature varied in almost 20°C. Consequently, it can be pointed out that only seasonality influenced on greater ranges in anaerobic digestion temperature, according to Figure 5b.

### Stability

Statistical assumptions, based on Shewhart control chart



**Figure 5.** (a) Values of the ratio between volatile fatty acidity (VFA), total alkalinity (TA) and partial alkalinity (PA) from effluent; (b) Maximum and minimum temperature of anaerobic digestion.

for individual measurements, were met in evaluations 1 and 2. According to normality test, biogas production values showed normal distribution with 0.966 p-value for evaluation 1 and 0.192 p-value for evaluation 2.

Values of both evaluations are independent according to the chart of sampling autocorrelation function. Randomness was confirmed at Shewhart control chart since the values are nearby their average, without any trends. So, since statistical assumptions have been met, Shewhart control chart was drawn using biogas production values to check reactor stability in each evaluation, according to Figure 6.

Shewhart control charts met the criteria of non-random patterns of control charts, so, the process was considered stabilized during the reactor evaluation periods. The average efficiency of biogas production was  $0.0119 \text{ m}^3/(\text{kg VS}_{\text{added}})$  in evaluation 1 and  $0.0429 \text{ m}^3/(\text{kg VS}_{\text{added}})$  in evaluation 2.

### Energy production

Augusto (2011) recorded biogas production values close to the ones registered in this trial,  $0.0185 \text{ m}^3/(\text{kg VS}_{\text{added}})$ , when he evaluated a 10 L of PL batch reactor, diluted in water, for 50 days at 3.91% VS rate. Santos (2001) has also obtained production biogas average of  $0.0336 \text{ m}^3/(\text{kg VS}_{\text{added}})$  when he evaluated anaerobic digestion of

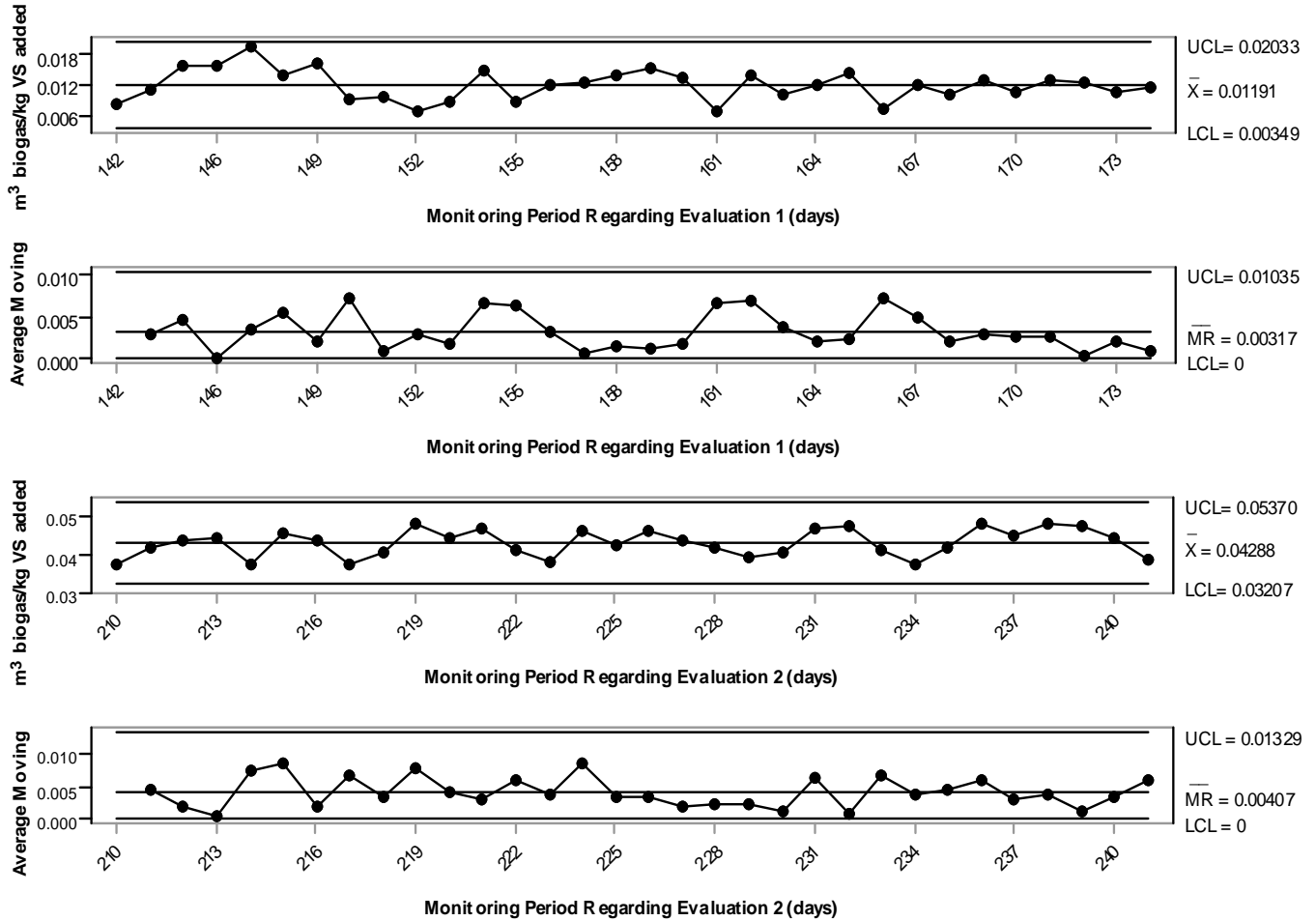
PL, diluted in water, in sequenced batch system with 9.5% TS rate over a period of 15 production days. By comparison, biogas production efficiency concerning evaluation 2 was higher and stood at the lowest HRT (10 days) and its feeding rate was only 1.0% VS.

Average methane content ( $\text{CH}_4$ ) was 49.25% in the evaluation 1 and 42.40% in the evaluation 2. So, the average efficiency of methane production was  $0.0059 \text{ m}^3/(\text{kg VS}_{\text{added}})$  in the first evaluation and  $0.0182 \text{ m}^3/(\text{kg VS}_{\text{added}})$  in the second evaluation.

Gangagni Rao et al. (2013) has also evaluated the anaerobic digestion of PL with effluent reuse in self-mixed anaerobic reactor under high-organic loading rate ( $4 \text{ kg VS}/\text{m}^3/\text{day}$  and 24 HRT days) and in conventional fixed dome anaerobic reactor ( $2.15 \text{ kg VS}/\text{m}^3/\text{day}$  and a 40 HRT days). They recorded the following answers to production of biogas and methane:  $0.23 \text{ m}^3/(\text{kg VS}_{\text{added}})$  and  $0.15 \text{ m}^3/(\text{kg VS}_{\text{added}})$ ,  $0.128 \text{ m}^3/(\text{kg VS}_{\text{added}})$  and  $0.083 \text{ m}^3/(\text{kg VS}_{\text{added}})$ , respectively. Nevertheless, the authors applied a higher feeding organic load as well as a higher HRT when compared to the one used in this trial, which contributed to its biogas production.

### AIOEV and $C_{PI}$

According to the results, AIOEV of each evaluation was calculated and has shown that both evaluations were



**Figure 6.** Values of biogas production from evaluations 1 and 2. Where: UCL = upper control limit; LCL = lower control limit;  $\bar{X}$  = sampling average to  $P_{biogas}$  ( $m^3/kgVS_{added}$ ) or estimate of the population means;  $\overline{MR}$  = estimate of the average moving range.

**Table 2.** Average index of operational energy viability (AIOEV).

Treatments	$P_{biogas}$ ( $m^3/day$ )	[ ] $CH_4$ (%)	H (h/day)	AIOEV
Evaluation 1	0.2084	49.25	0.26	1.05
Evaluation 2	1.5010	42.40	0.39	4.35

feasible. However, evaluation 2 stood out with energy production in methane form 4.35 times greater than electric power used in the treatment system operations with the pump (Table 2). Evaluation 1 was classified as feasible, though; the energy produced in methane form was only 1.05 times greater than the operational power expended.

According to Montgomery (2009), a process considered new (e.g., research on anaerobic digestion) is capable when its  $C_{pi}$  is greater than 1.45, according to Table 3. In this context, it is important to mention that AIOEV and  $C_{pi}$  indexes are related to the factors that affect biogas production, that is, factors that affect anaerobic digestion: temperature, C/N ratio, pH, volatile fatty acidity, alkalinity,

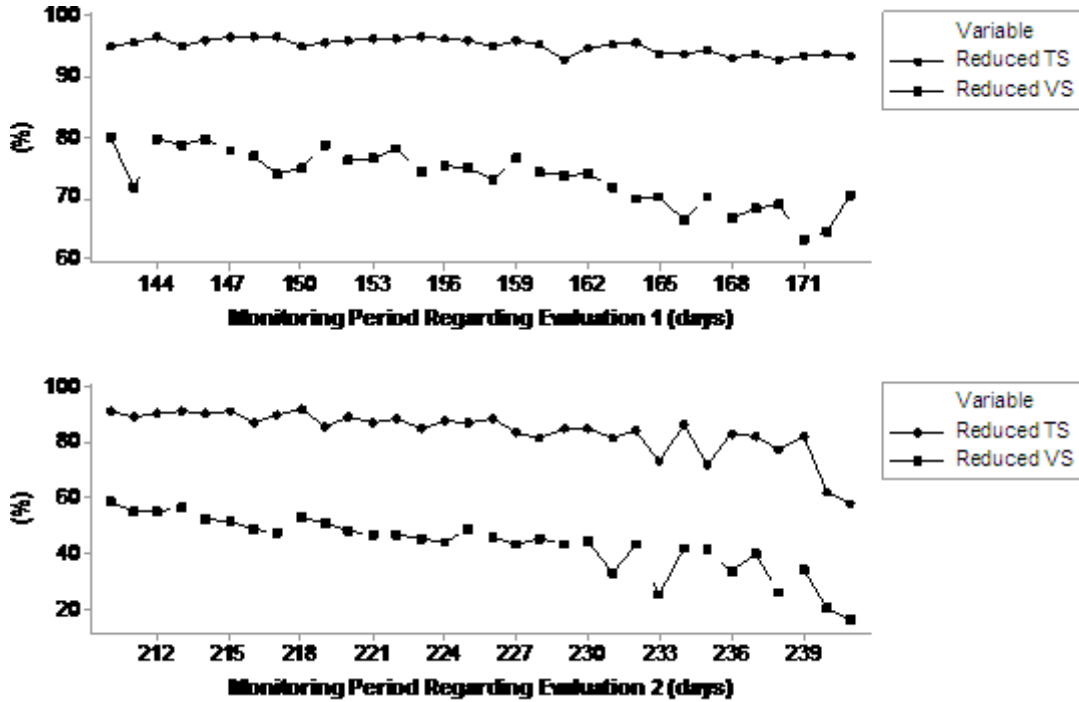
inhibitors, solids, HRT, volume reactor, others. Thus, for a larger useful volume of reactor is possible to obtain a larger production of biogas. However, in this case the pumping time to feed the reactor is also higher, because the feeding daily flow rate also increases to maintain the HRT. Thus, the AIOEV index relates the energy produced in the form of methane with the spent energy in the anaerobic digestion operations.

**Solids**

Solid load in effluent influenced on the time of pump use, which increased from 15.56 min in the first evaluation to

**Table 3.** Lower Process Capability Index ( $C_{pi}$ ) versus average index of operational energy viability (AIOEV).

Treatment	LSL ( $\frac{m^3 \text{ biogas}}{kg \text{ VS}_{added}}$ )	AIOEV	Classification	$C_{pi}$	Classification
Evaluation 1	0.0113	1.05	Feasible	0.07	Incapable
Evaluation 2	0.0099	4.35	Feasible	3.04	Capable



**Figure 7.** Values of total solids (TS) and volatile solids (VS) reduced for evaluations 1 and 2.

23.40 min in the second one, due to the need of a greater stirring of effluent and to the flow rate decrease of pump with the solids. After second evaluation, the effluent began to show greater solids content, which indicated solids' deposition into reactor. Since, the stirring and feeding time was 50 min and the pump had major reduction in its flow rate.

The averages on solids reduction were 95 and 73% in evaluation 1 and 84 and 43% in evaluation 2 for TS and VS, respectively, according to Figure 7. The high TS removed value can be attributed to the solid fraction of PL that has settled at the bottom of the digester because, according to Farias et al. (2012), the solid fraction of the bird waste is rapidly sedimented in the digester and its determination is always subject to be underestimated. This implies in use the volatile solids added ( $VS_{added}$ ) in the calculation of the specific biogas production instead of the volatile solids removed to avoid values not representative in the biogas production efficiency

analysis:  $m^3/kg \text{ VS}_{added}$ . So, from 1 to 239 days, solid content into effluent was low, and the values of TS reduction varied from 70 to 99%. Later on, TS reduction decreased by 20% at 289 days, showing that the reactor has gotten saturated by sludge, with higher output of solids in effluent.

**Conclusion**

The effluent of the PL anaerobic digestion can be reused in the own process to dilute the PL in the reactor feeding, on a pilot scale, contributing to the moisture content and making part of the feeding organic load.

**Conflicts of Interests**

The authors have not declared any conflict of interests.



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