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A new strategy to maximize organic matter valorization in municipalities: combination of urban wastewater with kitchen food waste and its treatment with AnMBR technology.

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ABSTRACT

The aim of this study was to evaluate the feasibility of treating the kitchen food waste (FW) jointly with urban wastewater (WW) in a wastewater treatment plant (WWTP) by anaerobic membrane technology (AnMBR). The experience was carried out in six different periods in an AnMBR pilot-plant for a total of 536 days, varying the SRT, HRT and the food waste penetration factor (PF) of food waste disposers. The results showed increased methane production of up to 190% at 70 days SRT, 24 hours HRT and 80% PF, compared with WW treatment only. FW COD and biodegradability were higher than in WW, so that the incorporation of FW into the treatment increases the organic load and the methane production and reduces sludge production (0.142 vs 0.614 kg VSS·kg removed COD^{-1}, at 70 days SRT, 24 hours HRT and 80% PF, as compared to WW treatment only).

KEYWORDS

Anaerobic wastewater treatment, kitchen food waste (FW), organic matter valorization, penetration factor (PF), submerged anaerobic membrane bioreactor (AnMBR), wastewater co-treatment

1. INTRODUCTION

Anaerobic treatment is increasingly recognized as the core method of an advanced technology for environmental protection and resource preservation. Combined with other methods, it represents a sustainable and appropriate wastewater treatment system (Seghezzo *et al.*, 1998) as it has the important potential of recovering energy by reducing the organic matter content of municipal and industrial wastewaters while producing biogas (Skouteris, 2012). Anaerobic wastewater treatment also has other advantages over conventional aerobic systems: (1) lower sludge production, (2) reduced pathogens, (3) lower energy demand and (4) the possibility of recovering nutrients from wastewater which can be reused for agricultural purposes (Robles, 2013). However, the main challenge of anaerobic biotechnology is to develop treatment schemes that prevent biomass loss and enable high solids retention times (SRT) to offset the low growth rates of anaerobic biomass at ambient temperatures (Lin *et al.*, 2010). Membrane technology applied to wastewater treatment by the so-called membrane bioreactors (MBR) is a promising alternative for obtaining high biomass and COD concentrations by decoupling both hydraulic retention time (HRT) and solids retention time. The complete retention of the microorganisms inside the MBR system allows high SRT to be obtained with reduced working volumes. In recent years, submerged MBR technology has been reported as a successful application for anaerobic wastewater treatment in the form of the Submerged Anaerobic Membrane Bioreactor (AnMBR) (see, for example, Giménez *et al.*, 2011, Huang *et al.*, 2011 and Robles *et al.*, 2013).

Wastewater in most developed countries is characterized by low organic matter concentration (Pons *et al*., 2004), so that the energy recovery potential through anaerobic processes is less than other streams highly enriched in organic matter. Bolzonella *et al*. (2003) found that the increase of the organic load content was possible mixing wastewater with the domestic organic fraction of municipal solid waste (OFMSW) by using food waste disposers. These disposers have been suggested as a practical way of separating food waste at source (Marashlian and El-Fadel, 2005).

Kujawa-Roeleveld and Zeeman (2006) pointed out the advantages of OFMSW co-treatment, the most important of which is that the influent is richer in organic matter, methane production is increased, with a subsequent increase in energy recovery. This treatment option can also help to accomplish the target of reducing by 2016 the quantity of organic waste going to landfill sites to 35% of the total amount of biodegradable municipal waste produced in 1995 in each Member State, according the 1999/31/CE Directive. Furthermore, it aligns with the 2008/98/CE Directive which considers that the recovery and re-use of waste materials should be encouraged in order to conserve natural resources.

Several authors have studied the co-digestion of food waste with wastewater treatment plant (WWTP) sludge (Iacovidou *et al*. 2012), with other organic wastes (Nayono *et al*., 2009), manure and other agricultural residues (Zhang *et al*., 2011). Preliminary studies carried out by Moñino et al. (2016) studied the chemical and physical characteristics of the kitchen FW after the grinding process, pointing out the potential benefits of mixing raw wastewater with FW and its treatment with the AnMBR technology, However, to date, only a few studies have been published on co-treating food waste with wastewater, and none of them used AnMBR, which is considered as an innovative technology within the 'waste-to-resource' philosophy. The main precedents of the present study are Kujawa-Roeleveld *et al*. (2006), Wendland *et al*. (2006), Luostarinen *et al*. (2007) and Rajagopal *et al*. (2012), which were on a lab-scale and without the AnMBR technology proposed in the present study, while the present study involved a continuous operation on an AnMBR at pilot-plant scale, treating the wastewater from a WWTP and FW from the restaurants of the Polytechnic University of Valencia. Real FW was used in order to reproduce the high variability factor of a real scenario. Therefore, despite increasing the complexity of the work, the use of real kitchen waste and AnMBR at industrial scale added considerable value from a technology-transfer point of view. Another important aspect of the joint treatment of WW and FW is the significant reduction of the transport cost and greenhouse gas emissions of the FW from the production site (households) to the final treatment site. Currently this transport requires specific infrastructure with important energy consumption and

CO_2 emissions (specifically with the classical surface transport with vehicles), meanwhile the joint treatment of WW and FW uses the current sanitation infrastructures, reducing considerably the energy consumption, the CO_2 emission, and valorises the energy resource contained in the FW thanks to the anaerobic treatment, such as the one studied in this work.

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The aim of this work was to study on a pilot-plant scale the feasibility of a new organic matter operation strategy in municipalities in order to maximize the energy recovery, treating the kitchen food waste (FW) jointly with urban wastewater (WW) in a wastewater treatment plant through anaerobic membrane bioreactor technology.

2. METHODS

2.1. Pilot-plant description

Figure 1(a) shows the process flow diagram of the AnMBR pilot-plant used in this study, which is located in the "Barranco del Carraixet" WWTP, Alboraya (València). The pilot-plant is fed with the effluent of the Carraixet WWTP pre-treatment (after the screening, degritter and grease removal). After further pre-treatment in a rotofilter (RF) of 0.5 mm screen size and homogenization in the regulation tank (RT), the wastewater is pumped to the anaerobic reactor (AnR). The pilot plant mainly consists of an anaerobic reactor of 1.3 m^3 total volume (0.4 m^3 head-space volume) connected to two membrane tanks (MT) of 0.8 m^3 total volume each (0.2 m^3 head-space volume). Each membrane tank includes one industrial hollow-fiber ultrafiltration membrane module (PURON® Koch Membrane Systems (PUR-PSH31), 0.05 µm pore size). In order to improve the stirring conditions of the anaerobic reactor and to favour the stripping of the gases produced in the liquid phase, a fraction of the produced biogas is recycled to this reactor (P2). The sludge is continuously recycled through the external membrane tanks , where the effluent is obtained by vacuum filtration and stored in a Clean-in-Place (CIP) tank. Another fraction of the biogas produced is also recycled to the membrane tanks from the bottom of each fibre bundle with the purpose of minimizing cake layer formation. In order to control the solids

retention time in the system, a fraction of the sludge is intermittently extracted from the anaerobic reactor throughout the day. Process temperature can be controlled if necessary, since the anaerobic reactor is jacketed and connected to a water heating/cooling system. The AnMBR membrane operation consists of a combination of different stages based on a filtration–relaxation (F–R) cycle and also considering back-flush, degasification and ventilation. The anaerobic reactor is only fed when the filtration phase of the membranes is taking place, in order to maintain the same reactor volume and according to the set HRT. Therefore, the WW regulation tank is necessary to guarantee the AnMBR feed requirements. The stirrer in this tank helps to ensure a homogenized sample when feeding the reactor. It is necessary to homogenize the wastewater in order to avoid solid sedimentation in the regulation tank. Further details of this AnMBR pilot-plant can be found in Giménez *et al*. (2011) and Robles *et al*. (2013).

Figure 1 (a) shows the elements involved in FW feeding (inside the red box). Pretreatment of the FW required a commercial food waste disposer and a 0.5 mm space screen rotofilter (CT RTF), followed by a co-substrate tank (CT) with a usable volume of 0.180 m^3, to store the FW and remove grease by manual scraping (see Figure 1(b)).

The FW feeding system is regulated by a three-way valve that connected both RT and CT with the AnR in order to alternate feeding wastewater from the RT with FW from the CT.

2.2. FW feeding procedure

FW was ground into small particles before being fed to the reactor, in order to reproduce the conditions of a real scenario. An experimental set-up simulating a household FW grinding system was used (see Figure 1b). This consisted of a kitchen sink with an InSinkErator Evolution 100 food waste disposer fitted underneath. In order to prevent damage to the membranes, the FW was filtered through a 0.5 mm sieve-size rotofilter. The filtered FW was stored in the CT, which was equipped with a stirrer and membrane diffusers for homogenization and fat removal, respectively.

To study the effect of different percentages of households with food waste disposers on increasing the organic load in the treatment plant, an FW control system was developed. The "penetration factor" (PF) was defined as the percentage of households that use food waste disposers. The control system maintained the desired PF by feeding a small percentage of the CT (5%) each time in such a way that the organic load was equally distributed throughout the day.

2.3. Operational conditions in the AnMBR demonstration plant

The pilot-plant was operated for 536 days, during which four different co-treatment periods can be distinguished according to the operational conditions (Periods 2 to 5) shown in Table 1. In addition, an initial period (Period 1) and a final period after co-treatment with FW (Period 6) were also included to evaluate the process performance with and without adding FW.

The values shown in Table 1 belong to the pseudo steady-state achieved in each period. The criteria followed to consider a pseudo steady-state period were the accomplishment of COD balances with low COD accumulation in the reactor, with the subsequent stability of solids concentration and methane production. Period 1 consisted of the month previous to the co-treatment experience.

The pilot plant was operated at three different SRT: 40 days (Periods 1 and 2), 70 days (Periods 4, 5 and 6), and at an extended SRT (Period 3), during which only a 0.5 L sample was withdrawn on a daily basis for analytical characterization. The HRT was fixed at 20 hours, although it was somewhat higher during Periods 1, 3 and 5 due to operational problems. Operational temperature was maintained around 28 ºC during the whole co-treatment experimental period.

The FW PF was set at 40% for P2, P3 and P4, considering that only a few households used food waste disposers. It was then raised to 80% in P5, keeping the rest of the operational conditions to their previous values, to assess how the increased organic load from a high percentage of households using a food disposer affected the system.

FW flow was determined by considering that an inhabitant equivalent (IE) generated 225 litres of wastewater and 0.63 kg of FW per day, as specified in the Spanish National Integral Waste Plan (2007-2015). After grinding, an average flow of 2.52 $L \cdot IE^{-1} \cdot d^{-1}$ of FW was experimentally obtained (Moñino *et al.*, 2016). There was a remarkable difference between the FW volume and the wastewater volume. Therefore, the flow increase due to the FW addition was negligible (around 1%).

Start up of co-treatment

To progressively adapt the microbial population to the FW, the substrate load was increased stepwise during the first weeks. To achieve the organic load of the 40% of FW PF tested in the first scenario, the daily food waste added was increased by one IE per day during the first week and 3 IE per day during the second week.

2.4. Analytical methods

To evaluate the performance of the biological process, the following parameters were analysed on a daily basis for the WW and FW influents, effluent, and anaerobic sludge: Total Solids (TS), Volatile Solids (VS), Total Suspended Solids (TSS), Volatile Suspended Solids (VSS), Volatile Fatty Acids (VFA) and alkalinity (Alk). Furthermore, twice a week, coinciding with CT feeding, total and soluble chemical oxygen demand (COD_T and COD_S, respectively); total nitrogen (TN) and phosphorous (TP), sulphate (SO_4–S), sulphide (S^{2-}), and nutrients (ammonium (NH_4–N) and orthophosphate (PO_4–P)) were measured. Solids, COD, sulphate, sulphide, and nutrients were determined according to Standard Methods (APHA, 2005). The carbonate alkalinity and VFA concentration were determined by titration according to the method proposed by WRC (1992). Specific Methanogenic Activity (SMA) tests were carried out by the Automatic Methane Potential Test System (AMPTS) [Bioprocess Control, Sweden], and performed as described in Ozgun *et al*. (2014). Duplicate analyses were performed on each sample.

Sulphide determination requires special care during sample collection, in order to ensure the minimum contact between the effluent and the oxygen present. An anaerobic environment must be ensured, to prevent the oxidation from sulphide to sulphate. Therefore, sample collection bottles must be completely filled, avoiding any volume of head space, where the oxidation of the sulphide could take place. Sulphide concentration was determined by using a commercial kit (Merck, 1.14779.0001), based on methylene blue method (Standard Methods, 4500-S2- D; APHA, 2012).

The results obtained during the experimental period were statistically analysed by one-way ANOVA in order to compare the different periods. ANOVA tests were performed using STATGRAPHICS Centurion XVI software (16.1.17 version). A p-value less than 5% was considered as statistically significant.

2.5. COD and solids mass balances

COD mass balances for each experimental period were carried out in order to track the COD distributions between the different streams (effluent, biogas and waste sludge) and components present in the effluent that contribute to the effluent COD measurements. Furthermore, the COD balances was performed to identify the pseudo-steady state for each experimental period. The COD mass balance was performed according to the methodology proposed by Giménez et al. (2012) (see Equation 1).

$$Q_{inf} \cdot COD_{inf} = Q_{eff} \cdot COD_{res-eff} + Q_W \cdot COD_{AnMBR} + Q_{CH4\,gas} + Q_{eff} \cdot COD_{CH4\,dis} + Q_{eff} \cdot COD_{SRB} + V_{AnMBR} \cdot COD_{Accum}$$

where, Q_{inf}, Q_{eff} and Q_W: are the flow rates of the influent, effluent and purged sludge of the pilot plant, respectively ($L \cdot d^{-1}$); COD_{inf}: COD concentration in AnMBR feed (WW+FW in different proportions corresponding to each experimental periods) (mg $COD \cdot L^{-1}$); COD_{AnMBR}: COD concentration in the reactor; $COD_{res\text{-}eff}$: COD in the effluent excluding the dissolved methane and the COD due to sulphide oxidation during the COD measurement (mg $COD \cdot L^{-1}$); $Q_{CH4\ gas}$: the COD equivalent for the production rate of methane gas from the reactor (mg

$COD \cdot d^{-1}$); $COD_{CH4\ dis}$: dissolved methane concentration in the effluent (mg $COD \cdot L^{-1}$); COD_{SRB}: COD removed by Sulphate Reducing Bacteria (SRB), calculated on the basis of the sulphate concentration removed in the process, taking into account that SRB consume 2 kg of COD in order to reduce 1 kg of SO_4-S (Lens *et al.* 1998) (mg $COD \cdot L^{-1}$); COD_{Accum}: the accumulation term of the balance calculated as the COD accumulation in the reactor, measured as the difference in the COD concentration in the reactor between the beginning and final of the pseudo-steady estate evaluated for each period (mg$COD \cdot L^{-1} \cdot d^{-1}$); V_{AnMBR}: AnMBR volume (L).

Solids mass balances were also performed in order to compare the solids removal efficiency between periods. The solids mass balance was calculated according to the methodology proposed by Giménez (2014) and is described in Equation 2:

$$Q_{inf} \cdot VS_{inf} - Q_W \cdot VS_{AnMBR} - H = V_{AnMBR} \cdot VS_{accum}$$

where, Q_{inf} and Q_W: are the flow rates of the influent, and purged sludge of the pilot plant, respectively ($L \cdot d^{-1}$); VS_{inf}: VS concentration in AnMBR feed (WW+FW in different proportions corresponding to each experimental periods) (mg $VS \cdot L^{-1}$); VS_{AnMBR}: VS concentration in the reactor; VS_{accum}: the accumulation term of the balance was calculated as the VS accumulation in the reactor, measured as the difference in the VS concentration in the reactor between the beginning and final of the pseudo-steady estate evaluated for each period (mg $VS \cdot L^{-1} \cdot d^{-1}$); V_{AnMBR}: AnMBR volume (L). H (mg $VS \cdot d^{-1}$) is the generation term, which, in this case, corresponds to the hidrolized solids and represents the solids removal efficiency when compared with the solids of the influent.

3. RESULTS AND DISCUSSION

3.1. Kitchen food waste and wastewater characterization

Table A1 (Appendix A) shows the characterization (mean value and standard deviation) of the FW and WW for each experimental period. As can be seen in this Table, a high variability in the composition of both streams can be observed during the different experimental periods. The

variability in FW and WW streams deeply affect to the experimental results obtained as will be discussed later.

3.2. COD and solids mass balances

The COD balance was performed according to the procedure explained in section 2.5. Figure 2 shows the influent COD, the effluent COD and its distributions between the different effluent streams (effluent, biogas and waste sludge). The time lapse evaluated in the COD balances correspond to the final days of each experimental period (marked as a grey zone in Figure 5). As can be seen in Figure 2, the COD balance is accurately closed during the different experimental periods with an error lower than 7% (maximum error corresponding to the period 2). Furthermore, the accumulation calculated at the end of each experimental period was lower than 10% of the COD balance for each experimental period confirming the achievement of the pseudo steady-state for each set of experimental conditions. However, for the experimental period P3 (PF 40% and with extended SRT) the steady state was not achieved (accumulation higher than 30%) due to the relative short duration of this period in comparison with the extended SRT set on it.

Solids mass balances were also performed in order to compare the solids removal efficiency between periods. The removal efficiency was obtained by comparing the hydrolysed solids and the influent solids fed into the reactor. The percentage of hydrolysed solids is shown in Table 3, and as can be seen, the higher hydrolysis is achieved in Period 3, as the biomass has unlimited time to degrade the substrate. Comparing Period 1 and 2, with the same SRT, the FW addition increases hydrolysis in a 20%. The Periods with 70 days of SRT, show that there was not a significant difference between hydrolysed solids at 40% PF and 80% PF, but there was a 58% decrease when comparing Period 5 with 80% PF of FW and Period 6, where only WW was treated in the AnMBR pilot plant. Therefore, it was demonstrated that FW contributes to a higher hydrolysis in the anaerobic process, seeming to be a synergy effect of the co-treatment. Moreover, when comparing Periods prior and after the co-treatment, it is shown that after treating FW and WW the percentage of hydrolysed solids was doubled, which means that the

effect of FW co-treatment has probably generated a new biomass population more capable of degrading complex molecules.

Figure 3 represents the solids generated due to the hydrolysis per day over the average methane production per COD kilogram removed by Methanogenic *Archaea* (MA). As can be seen in this figure, there is a lineal relation between methane production and the solids generated. Hydrolysis is the limiting step of anaerobic digestion processes, and, as it shown, it is directly related to the methane production by MA.

3.3. FW effect in organic loading rate and VFA accumulation

Figure 4 shows the total organic load rate (OLR) in the influent of the AnMBR pilot-plant (this figure considers only the available OLR for Methanogenic *Archaea* as will be discussed later), and the relative contribution (as a percentage) of the urban wastewater and the FW to OLR in the four co-treatment periods (periods 2, 3, 4 and 5). An ANOVA test demonstrated that there was significant differences between some of the periods, (p-value = 0.0010, see Table B.1 and B.2 and Figure B.3) due to the variability of the influent fed to the AnMBR pilot plant. The variability observed in the OLR was a consequence of different conditions, such as:

(1) Real WW load fluctuations and WWTP operation involved variations in COD concentration (e.g. different loads in dry and rainy periods, etc.)

(2) Variations in food waste composition (e.g. average COD: 59400±14000 $mg \cdot L^{-1}$; min: 44100 $mg \cdot L^{-1}$; max: 78200 $mg \cdot L^{-1}$, (see Moñino *et al.*, 2016 for further details).

The influent wastewater fed to the AnMBR pilot-plant had a high sulphate concentration (105±13 $mg\ S \cdot L^{-1}$) (Giménez *et al.*, 2011), due to the typical soil composition, rich in sulphates, of the Mediterranean basin. Sulphate concentration determines the competition between Sulphate Reducing Bacteria and Methanogenic *Archaea* for the available substrate (COD) in anaerobic processes. This competition depends on the COD/S-SO_4 ratio. SRB need 2 $g\ COD \cdot g^{-1}$ S-SO_4 for sulphate reduction, so if the ratio is higher than two, there is enough COD for the growth of both populations. The competition between MA and sulphate-reducing bacteria for

the available substrate thus affects methane production. Also, the presence of SRB can cause problems such as odours and corrosion, inhibition of MA, and a drop in the amount and quality of the biogas produced (Giménez *et al*., 2011). Table 2 shows the total OLR, the OLR consumed by SRB, the available OLR for MA, and the proportion of the available OLR due to the WW and the FW, respectively. According to laboratory measurements, most of the sulphate present in the influent was reduced to sulphide. Therefore, the OLR consumed in this process was subtracted from total OLR. As can be seen in Table 2, since the OLR consumed by SRB remained almost constant during the whole experimental period (the sulphate concentration did not change after the incorporation of the FW into the WW, as the ANOVA test shown in Appendix B, there are no significant differences between periods regarding the OLR available for SRB, see Tables B.3 and B.4 and Figure B.2), the available OLR for MA increases with FW, being expected a higher methane production (see section 3.6). It is worth mentioning that despite the increase in PF from 40 to 80 % between Period 4 and Period 5, the total OLR available for MA was similar (P4: 0.786 g COD·L^{-1}·d^{-1}; P5: 0.794 g COD·L^{-1}·d^{-1}), as a result of the reduced contribution of the wastewater organic load to the total organic load between Period 4 and Period 5. The Figure B.4 shows that the contribution of the WW to the OLR in Period 4 was higher than the rest of the periods due to the variability of the wastewater influent, and the Figure B.5 shows that the contribution of the FW to the OLR Period 5 was different to the rest of periods because of doubling the PF, but in Figure B.1 and B.3, it can be seen that Periods 4 and 5 have no significant differences between them.

Regarding the possible effect of FW addition over the anaerobic process performance by VFA accumulation, the VFA in the reactor showed an average value of 30 mg HAc·L^{-1}, which is significantly lower than the normal concentrations found in anaerobic digesters for this parameter (Zhao and Viraraghavan, 2004). The average value of the ratio VFA Alkalinity per Total Alkalinity (AlkVFA/AlkT), which is used as an indicator of the possible VFA accumulation in the reactor, was 0.05 during the whole experimental period. This parameter is considerably lower than the reference value of 0.3 despite the high PF and SRT fixed along the

experiments. This value ensures the operational stability of the process, whereas an increase above 0.3–0.4 would indicate stability problems requiring corrective actions (Martí et al. 2008).

3.4. FW effect on the solids concentration

Figure 5 shows the evolution of the solids concentration in the reactor during the entire study period. In this figure pseudo steady-state periods are shaded (as previously indicated in Section 2.3, the criteria followed to consider a pseudo steady-state period was the COD balance accomplishment). Table 3 shows the average results of total and volatile solids concentration of each period, percentage of hydrolysed solids, sludge production, influent COD/S-SO_4 ratio and effluent COD.

In Period 2, both total (TS) and volatile (VS) solids concentration remained stable regarding Period 1, at around 16 and 11 $g \cdot L^{-1}$, respectively. In Table B.12 it is shown that there was no significant differences between Period 1 and 2. In Period 3, sludge wasting was suppressed (only the sludge necessary for the daily laboratory analysis was purged) and TS concentration rose to 29 $g \cdot L^{-1}$. SRT was then set to 70 days in P4, resulting in a sharp decrease in solids concentration to 15.5 $g \cdot L^{-1}$.

As can be seen, TS concentration in P4 is slightly lower than in P2, despite having a notably longer SRT (70 d vs 40 d), which could have been due to a combination of different factors:

(1) Period 2 was carried out in the summer. Previous studies (Giménez, 2014) have demonstrated that the Carraixet WWTP influent has lower biodegradability during summer weather, resulting in a higher accumulation of non-biodegradable solids within the system.

(2) Extending SRT from 40 to 70 days caused a higher level of hydrolysis and further degradation of slowly biodegradable organic compounds, as a result of the longer contact time between the particulate fraction of the organic matter and the enzymes responsible for its hydrolysis. The higher hydrolysis level would lead to a higher amount of VFA available to be converted to methane by MA. In fact, the Specific Methanogenic Activity measured represents the maximum capacity of a reactor operating under ideal conditions to convert volatile fatty

acids into methane) increased from 10 to 50 mL $CH_4 \cdot g^{-1}$ VS·day^{-1} at 40 days and 70 days of SRT, respectively (see Table 5), resulting in higher methane production and lower solids concentration in the reactor at the longer SRT.

TS and VS concentrations dropped further in P5, as can be seen in Table 3, despite having a similar total OLR to P4 (see Table 2). This drop could have been due to the higher proportion of FW, which is more biodegradable than WW (Moñino *et al.*, 2016). In P6, the system was again fed with wastewater only, resulting in a marked reduction of OLR (from 0.794 to 0.458 g COD L^{-1} d^{-1}, see Table2), which led to reduced TS and VS concentrations.

The specific sludge production per COD removed (kg VS·kg^{-1} removed COD) in P2 was half that observed in P1 (see Table 3). The addition of FW as co-substrate increased OLR, which led to an increase in the COD removed (COD removal was higher than 90% in both periods, see Table 3). The high biodegradability of the co-substrate meant that the increased OLR was well accepted by the system, with no solids accumulation and a noticeable increase in methane production. This can also be seen by comparing P4 and P5, when PF was doubled (with similar total OLR), thus increasing the contribution of the FW (which is more biodegradable than WW) and yielding lower sludge production, which significantly increased in Period 6, because of the lower COD removed (see Figure B.7 and Table B.14, for statistic tests). Sludge production dropped from 0.316 kg VS·kg removed COD^{-1} in P2 to 0.179 kg VS·kg removed COD^{-1} in P4 (Table 3) due to the longer SRT (from 40 to 70 days). Sludge production was lowest during P3 (0.015 kg VS·kg removed COD^{-1}) because of the extended SRT.

3.5. FW effect in nutrient content

Nutrient concentrations (N and P) in both the influent and effluent of the AnMBR pilot-plant were similar both with and without FW, evidencing a similar composition of the co-substrate and the WW regarding nutrients content. Table 4 shows the concentration of NH_4-N and PO_4-P in the influent and effluent of the pilot-plant and also in the wastewater for comparison purposes. An increase in the concentration of soluble forms of the nutrients occur in the effluent

because of the degradation of the organic matter, which entails the solubilisation of the organic nitrogen and phosphorous to ammonium and phosphate, respectively.

Sulphates concentration in FW and WW was in the same range (Moñino *et al.,* 2016). Since the COD concentration was 100 times higher in the FW than in the WW, the COD/SO_4-S ratio increases significantly after blending both substrates. As explained in Section 3.1., if COD/SO_4–S ratio is higher than two, there is enough COD for the growth of both SRB and MA. Hence, the COD concentration available for MA is higher and the subsequent methane production is also expected to be higher. The COD/SO_4–S ratio of the influent for the different periods is shown in Table 3, and at the ANOVA test for the COD/S-SO_4 ratio shown in Appendix B, it is shown that the difference between Periods with FW and Periods treating only WW are significant (between Period 1 and 2, and between Period 5 and 6, see Table B.16).

3.6. FW effect in effluent characteristics

Effluent concentrations were similar in all periods and lower than the limit concentration allowed to accomplished the discharge requirements (125 mg COD ·L-1), according to Council Directive 91/271/EEC of 21 May 1991. The organic matter elimination is higher than 90% in all cases (see Table 3), despite the increase in the organic load from adding FW. The excellent retention capacity of the membranes made the system capable of achieving high effluent quality in all periods.

3.7. FW effect on methane production

Table 5 shows methane production for the entire study, including: (1) average flowrate of methane gas recovered in the gas stream in litres per day; (2) total methane volume produced per day in both the gas stream and dissolved in the effluent stream (the methane dissolved in the effluent was calculated by Henry's law, further details can be found in Giménez *et al.* (2012)); (3) the percentage of methane contained in the biogas (4) SMA; (5) methane yield in litres kg of COD removed by MA; (6) total methane production in litres per kg of COD removed (by SRB and MA) and (7) the increase in methane production compared with P1, the period prior to FW

co-treatment (calculated as the difference between a period's methane production and that obtained in Period 1, divided by the latter).

As can be seen in Table 5, methane production during co-treatment (Periods 2 to 5) is noticeably higher than the production obtained when the pilot-plant was fed with wastewater only (Periods 1 and 6), reaching an increase in methane production of up to 190% in P5 over P1. ANOVA test of the total methane production (see Appendix B, Tables B. 17 and B. 18 and Figure B.9), shows the increment between Periods and the higher production of Period 6 regarding Period 1. The joint treatment of different substrates leads to the benefit of synergies between them, as reported by many authors (Macías-Corral *et al.*, 2008; Silvestre *et al.*, 2015). As indicated by these authors, co-digestion promotes higher biomass population and its activity, and as a consequence, higher removal rates and gas production. The increment in the percentage of solids hydrolysed, methane yield and SMA values (see Table 3 and Table 5, respectively) suggests the enhancement of biomass population. Therefore, this huge increment in the methane production is probably thanks to the co-treatment of FW and WW.

As expected, the longer the SRT the higher methane production with the same PF, because the substrate is retained in the system for a longer time so that hydrolysis is promoted (limiting stage in anaerobic process), allowing the degradation of slowly biodegradable organic compounds (Martí, 2007). Higher methane production can be observed between 40 and 70 days of SRT (P2 and P4). The results also show that operating the AnMBR at an SRT longer than 70 days does not yield higher methane production, as the production between 70 days and extended SRT (P3 and P4, respectively) was similar, while the solids concentration at extended SRT doubled its value (see Table 3).

Doubling PF increased methane production by 30% (P4 vs P5) and by nearly 200% between P5 and P1 (from 0 to 80% PF). Although the FW PF in P5 was twice that of P4, total OLR was similar in both periods (as shown in Table 2). Therefore, the difference in methane production between both periods is due to the higher FW biodegradability. The higher the PF, the higher the biodegradability of the influent, allowing improved hydrolysis of the substrate, increasing

VFA availability for MA (higher SMA was observed (see Table 5)) and leading to higher methane production and lower solids concentration in the reactor.

In the periods when only WW was treated (P1 vs P6), the higher methane production observed in P6 was due to several factors: the higher SRT, and the higher SMA (see Table 5) in P6. Methane production in P6 is relatively high, with a drop of only 37% when compared with the previous period at 80% FW PF, which could be attributed to the enrichment in the MA population, which made the sludge more active. Note the higher SMA value in P6 over P1. Nevertheless, further research is needed regarding the microbial population dynamics in order to analyse the different genera or species present with and without FW.

The methane content of the biogas increased significantly, from 43.5 to 74.7% (P1 vs P5) probably because of the increased substrate availability for MA, as the SRB have almost the same concentration of sulphates in the influent in all periods (see section 3.3). Adding FW to the AnMBR leads to higher MA activity, generating CH_4 and CO_2, while SRB produces only CO_2, so that there is a higher proportion of methane in the biogas obtained.

Since the presence of sulphates is a peculiarity of the region in which this experimental study was carried out, methane production was estimated in the absence of sulphates, by calculating the methane production expected from the anaerobic degradation of the COD utilised by the SRB for sulphate reduction (2 g COD·g^{-1} S-SO_4). The results are shown in Figure 6, which compares the actual and estimated methane production in the absence of sulphates. On average, the absence of sulphates would increase methane production by around 155±23 more litres of methane per day.

The results obtained in this study show that joint co-treatment of FW and urban wastewater in anaerobic conditions is a good and feasible alternative to the WW conventional treatment and reduces the amount of FW in line with the 1999/31/CE Directive, with the waste being converted to methane. The addition of the FW as a co-substrate increased OLR and the high biodegradability of the FW led to an increase in the COD removed. The increased OLR did not

entail acidification or overloads in the system, with no significant solids accumulation and a noteworthy increase in methane production. This was also seen in the sludge produced, which was half as much in P2 as in P1, with 40% of added FW. When PF was doubled between P4 and P5 (with similar total OLR) the contribution of the FW was increased and resulted in lower sludge production. FW biodegradability is higher than that of WW, so that increasing the penetration factor by adding FW to the treatment boosts methane production and reduces sludge production, which decreased between P2 and P4 (Table 3) due to the longer SRT (from 40 to 70 days).

3.8. Filtration Process

Pretel et al. (2016) evaluated the filtration process performance during the experimental period. The gas sparging intensity for membrane scouring (measured as specific gas demand per square metre of membrane area, SGDm) was set to 0.10 $m^3 \cdot h^{-1} \cdot m^{-2}$ on the basis of previous experimental results (Pretel et al., 2016a). Previous studies showed that operating at critical filtration conditions resulted in minimum filtration costs (Pretel et al., 2016). Therefore, the 20 ºC-standardised transmembrane flux (J_{20}) was established for meeting critical filtration conditions depending on the total solids concentration (TS) reached in the anaerobic reactor. The operating J_{20} resulted in values from 15.8 to 19.4 LMH.

As a result of the different operating conditions no meaningful differences were observed in membrane fouling rate when feeding UWW in comparison with treating WW jointly with FW. Indeed, the fouling rate remained at low values even when treating WW jointly with FW. For instance, the fouling rate when operating at TS concentration of around 17 $g \cdot L^{-1}$ and PF of 0% was approx. 0.14 $LMH \cdot bar^{-1} \cdot m^{-3}$, while the fouling rate when the ST concentration was 16 $g \cdot L^{-1}$ and the PF was 40% resulted in 0.17 $LMH \cdot bar^{-1} \cdot m^{-3}$. Nevertheless, other operating periods showed that the fouling rate could increase up to 200% (from 0.21 to 0.38 $LMH \cdot bar^{-1} \cdot m^{-3}$) when the PF was set to 80%. Therefore, further research should be done to properly determine the effect of treating WW jointly with food waste in the AnMBR plant.

This experimental study jointly the previous study published by Pretel et al. (2016) (where were studied the energetic and economical implications of the treatment studied in the present work), clearly demonstrate the feasibility and the interest of treating jointly WW and FW with the AnMBR technology. This treatment accomplishes with the novel regulations which promote the resources recovery, reduction of CO_2 footprint and reduction of landfill Food Waste deposition.

4. CONCLUSIONS

Treating FW jointly with urban wastewater through anaerobic membrane technology was found to be feasible. The results show a marked increase in methane production, lower sludge production (0.614 to 0.316 kg VS·kg removed COD^{-1}) and the accomplishment of COD discharge limits. The higher COD concentration and anaerobic biodegradability of the FW than the WW boosted methane production by 57 % at 40% PF of the FW, 124% when SRT was increased to 70 days and nearly 200% when the PF of the FW was doubled. This experimental study jointly the previous study published by Pretel *et al.* (2016), clearly demonstrate the feasibility and the interest of treating jointly WW and FW with the AnMBR technology in order to maximize the organic matter valorisation in municipalities.

5. ACKNOWLEDGEMENTS

This research work was possible thanks to financial support from Generalitat Valenciana (project PROMETEO/2012/029) which is gratefully acknowledged. Besides, support from FCC Aqualia participation in INNPRONTA 2011 IISIS IPT-20111023 project (partially funded by The Centre for Industrial Technological Development (CDTI) and from the Spanish Ministry of Economy and Competitiveness) is gratefully acknowledged.

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Figure 1. Process flow diagram of the AnMBR pilot-plant with the elements for the FW incorporation marked in red. (b) Photo of the experimental grinding set-up: (l to r) disposer, rotofilter and co-substrate tank.

Figure 2. COD balance for each experimental period.

Figure 3. Relation between Methane production and Hydrolysed solids

Figure 4. OLR fed in the AnMBR pilot-plant and relative contribution (%) of the FW to the total OLR during co-treatment (Period 2: day 78 to 106; Period 3: day 227 to 252; Period 4: day 344 to 379; Period 5: day 406 to 428). Average and standard deviation of the total OLR, OLR due to wastewater, and % of OLR due to FW for each period shown are included in the textbox.

Figure 5. Solids evolution during the co-tretament research. Shaded areas correspond to the identified steady-state periods.

Figure 6. Real measured methane production and estimated methane production if sulphates were not present in the influent of the AnMBR pilot-plant.

Table 1. Operational conditions in AnMBR pilot-plant in the six periods studied.

Table 2. Average and standard deviation of the MA available OLR fed in AnMBR pilot-plant during the entire study (g COD· L^{-1}· d^{-1}).

Table 3. Average solids concentration, sludge production, COD/SO_4–S ratio in the influent, COD concentration in the effluent and COD removal (%) during the entire study.

Table 4. Nitrogen and phosphorous concentrations (mg·L^{-1}) in the influent and effluent of the pilot-plant and in wastewater during the entire study (Average ± Standard Deviation)

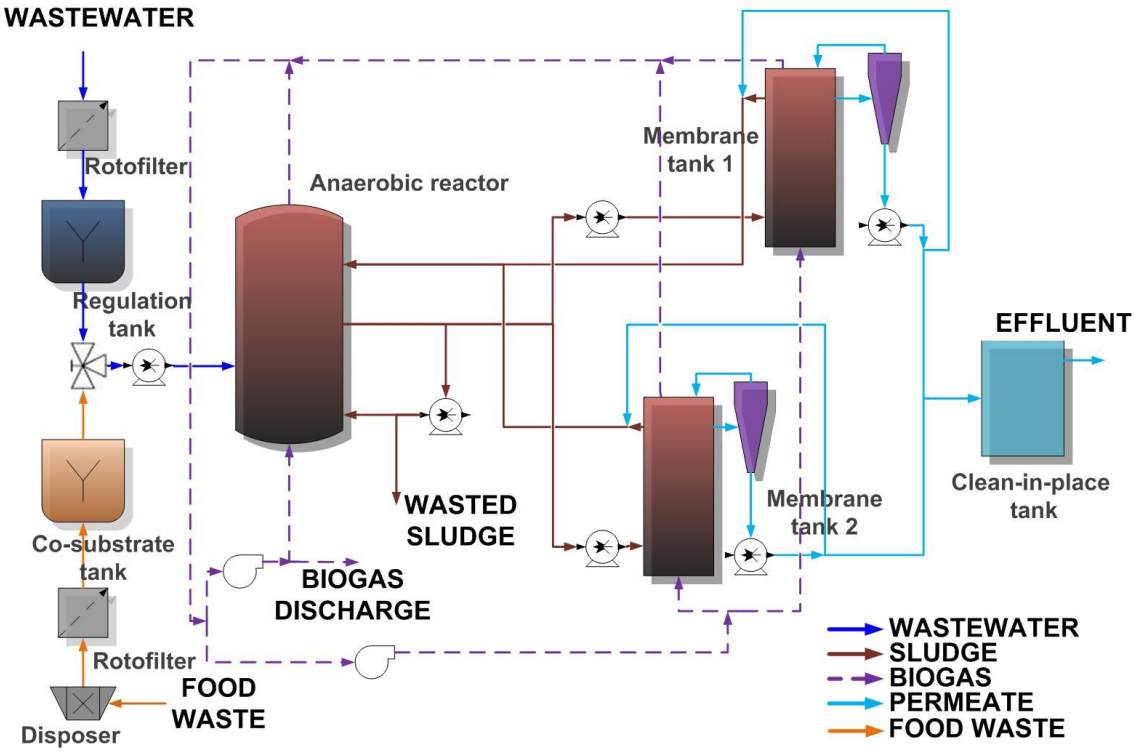
Table 5. Methane production during the entire study.

***Highlights**

Research Highlights:

- Food waste valorization by wastewater co-treatment in AnMBR technology is feasible.
- Methane production increased up to 190% compared with WW treatment only.
- Incorporation of food waste increases hydrolysis from 29% to 70%, at 70 d of SRT.
- Sludge production at 40d of SRT is reduced from 0.614 to 0.316 kg $VS \cdot Kg^{-1}$ removed COD

Figure 1



(a)

(b)

Figure 1. Process flow diagram of the AnMBR pilot-plant with the elements for the OFMSW incorporation marked in red. (b) Photo of the experimental grinding set-up: (l to r) disposer, rotofilter and co-substrate tank.

Figure 2

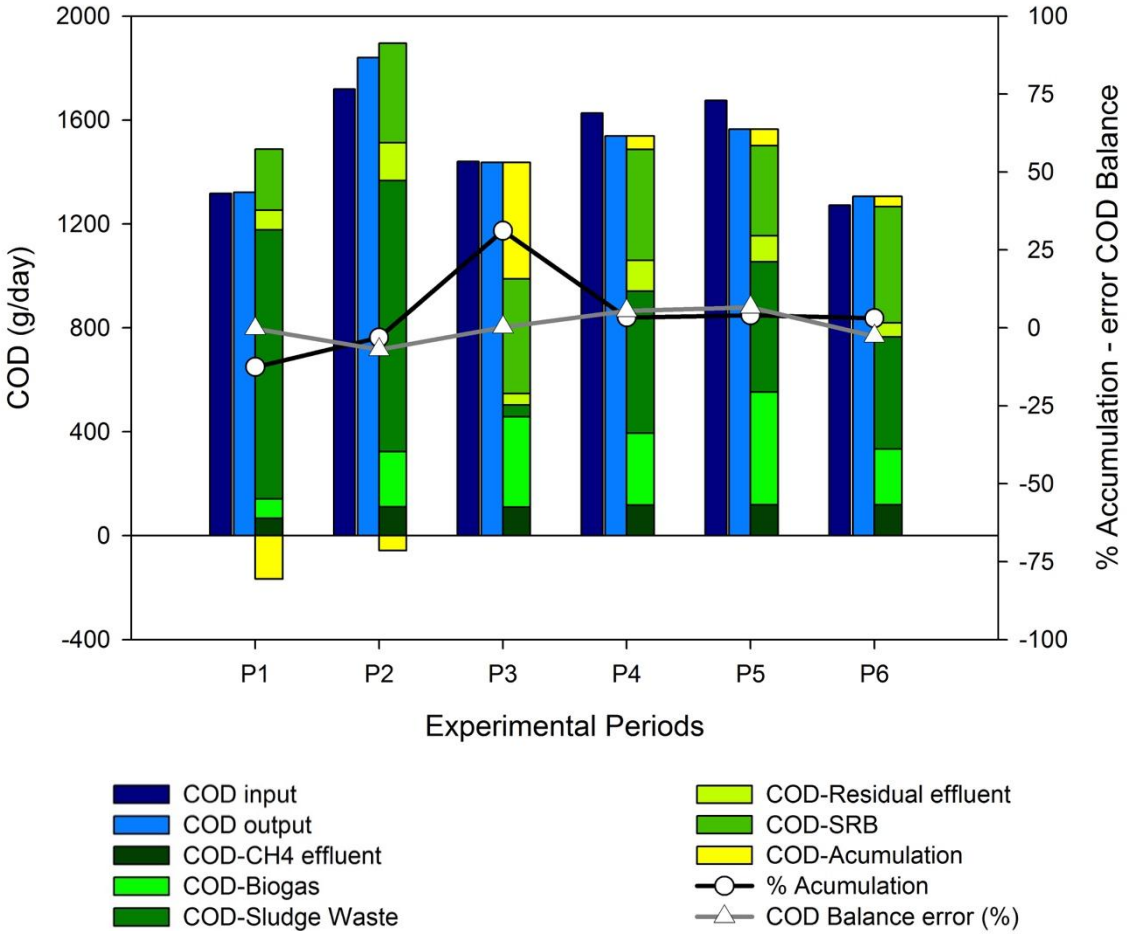


Figure 2. COD balance for each experimental period.

Figure 3

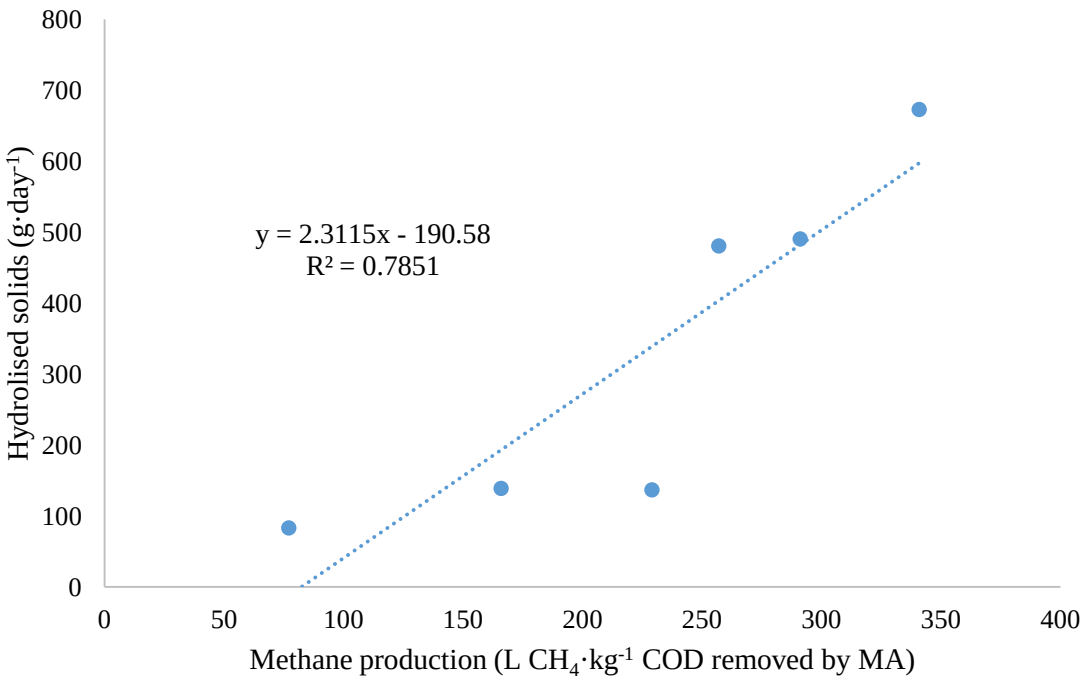


Figure 3. Relation between Methane production and Hydrolysed solids

Figure 4

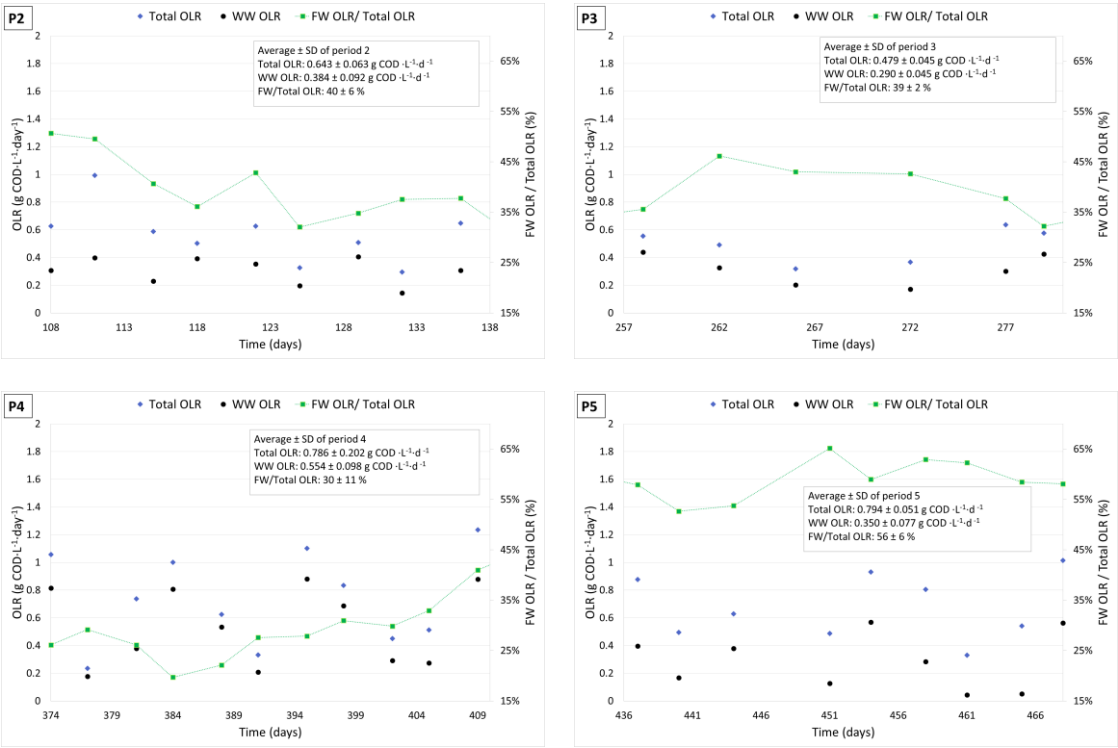


Figure 4. OLR fed in the AnMBR pilot-plant and relative contribution (%) of the FW to the total OLR during co-digestion (Period 2: day 108 to 136; Period 3: day 257 to 282; Period 4: day 374 to 4099; Period 5: day 436 to 468). Average and standard deviation of the total OLR, OLR due to wastewater, and % of OLR due to FW for each period shown are included in the textbox.

Figure 5

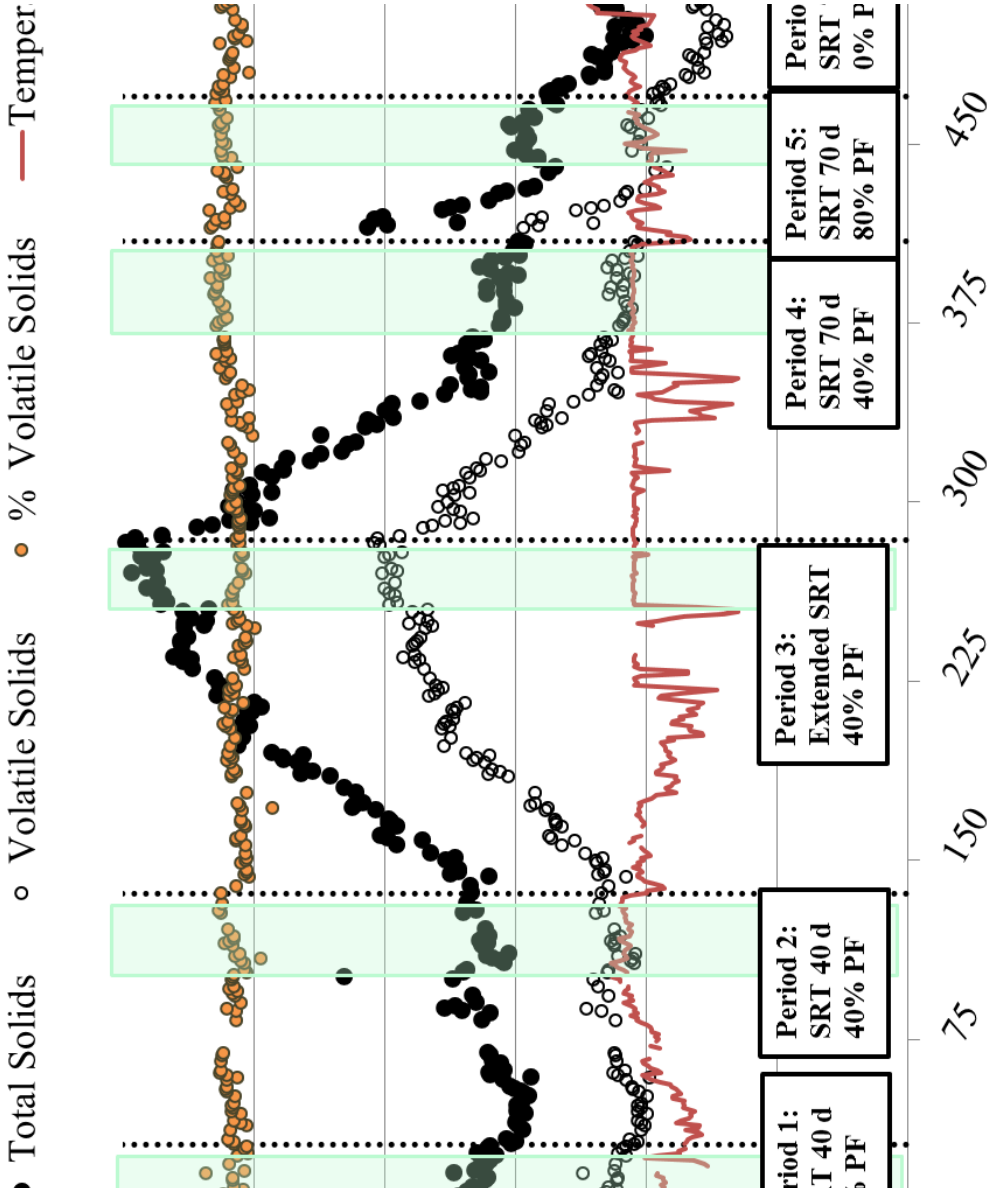


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Figure 6

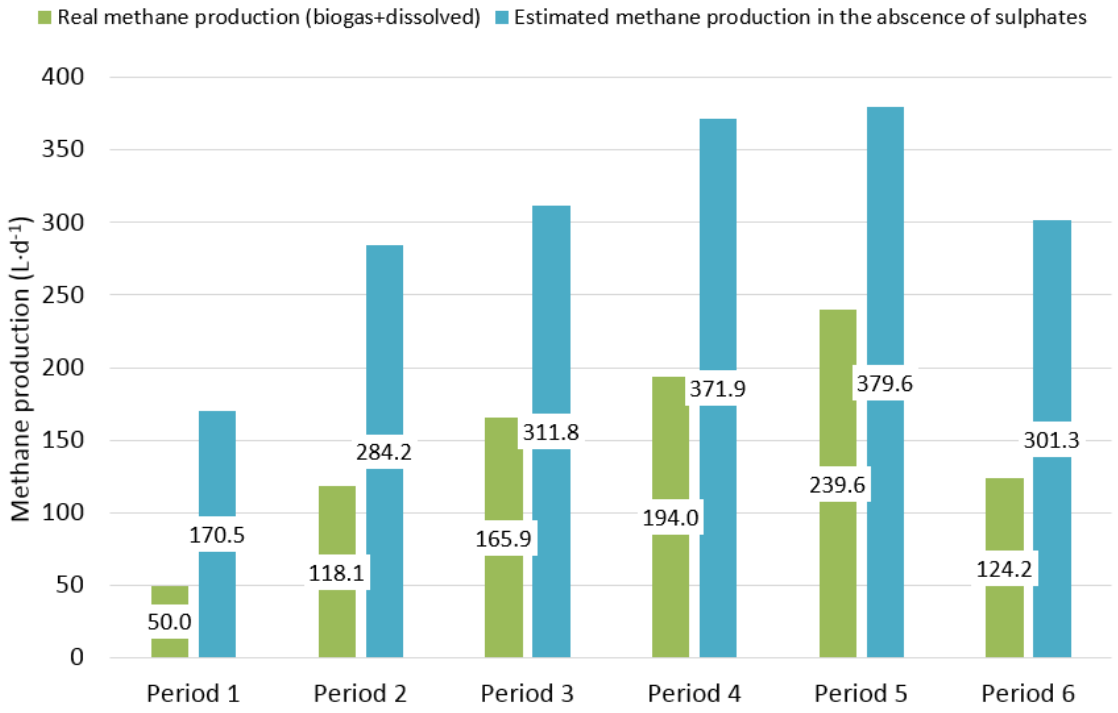


Figure 6. Real measured methane production and estimated methane production if sulphates were not present in the influent of the AnMBR pilot-plant.

Table 1

Table 1. Operational conditions in AnMBR pilot-plant in the six periods studied.

	SRT (d)	T (ºC)	HRT (h)	PF (%)
Period 1	42±2	25±2	30±4	0
Period 2	41±9	28±1	18±4	37±8
Period 3	Extended SRT*	28 ±0	26 ±3	39±7
Period 4	70±11	28±1	22±6	38±9
Period 5	69±6	27±1	24±5	78±9
Period 6	70±2	28±3	22±4	0

***Throughout Period 3, only the sludge necessary for the daily laboratory analysis was purged (hereinafter called 'extended SRT').**

Table 2

Table 2. Average and standard deviation of the MA available OLR fed in AnMBR pilot-plant during the entire study.

	Total OLR (g COD· L^{-1}· d^{-1})	SRB Available OLR (g COD· L^{-1}· d^{-1})	MA Available OLR (g COD· L^{-1}· d^{-1})	OLR WW (g COD· L^{-1}· d^{-1})	%	OLR FW (g COD· L^{-1}· d^{-1})	%
P1	0.605 ± 0.020	0.213 ± 0.110	0.392 ± 0.108	0.392 ± 0.108	100		
P2	0.871 ±0.092	0.225 ± 0.112	0.643 ± 0.063	0.384 ± 0.092	60	0.259 ± 0.061	40
P3	0.712 ± 0.066	0.233 ± 0.080	0.479 ± 0.045	0.290 ± 0.045	61	0.189 ± 0.023	39
P4	1.045 ± 0.255	0.257 ± 0.125	0.786 ± 0.202	0.554 ± 0.098	70	0.232 ± 0.113	30
P5	1.014 ± 0.066	0.22 ± 0.083	0.794 ± 0.051	0.350 ± 0.077	44	0.444 ± 0.057	56
P6	0.717 ±0.078	0.259 ±0.090	0.458 ± 0.044	0.458 ± 0.044	100		

Table 3

Table 3. Average solids concentration, solids hydrolysed, sludge production, COD/SO_4–S ratio in the influent, COD concentration in the effluent and COD removal (%) during the entire study.

	TS reactor ($mg \cdot L^{-1}$)	VS reactor ($mg \cdot L^{-1}$)	%VS	% Hydrolised solids in the reactor	Sludge production (kg $VS \cdot Kg^{-1}$ removed COD)	Influent COD_{total}/SO_4–S ratio	Effluent COD ($mg \cdot L^{-1}$)	COD removal (%)
P1	16556	11444	69	16	0.614	5.10	49.0	93
P2	16254	11215	69	20	0.316	7.69	51.6	91
P3	28943	19788	68	88	0.015	6.93	22.7	97
P4	15484	10873	70	65	0.179	6.99	54.3	85
P5	14373	9997	70	70	0.142	7.97	51.9	94
P6	12834	8907	70	29	0.245	5.57	25.7	97

Table 4

Table 4. Nitrogen and phosphorous concentrations ($mg \cdot L^{-1}$) in the influent and effluent of the pilot-plant and in wastewater during the entire study (Average ± Standard Deviation)

	NH_4 WW ($mg\ N \cdot L^{-1}$)	NH_4 influent ($mg\ N \cdot L^{-1}$)	NH_4 effluent ($mg\ N \cdot L^{-1}$)	PO_4 WW ($mg\ P \cdot L^{-1}$)	PO_4 influent ($mg\ P \cdot L^{-1}$)	PO_4 effluent ($mg\ P \cdot L^{-1}$)
P1	35.6 ± 8.9	35.6 ± 8.9	49.6 ± 11.1	3.9 ± 1.0	3.9 ± 1.0	6.0 ± 0.8
P2	29.1 ± 5.2	28.7 ± 5.0	40.8 ± 5.5	3.8 ± 0.5	3.5 ± 0.5	5.0 ± 0.5
P3	47.3 ± 7.1	48.3 ± 5.7	69.6 ± 11.1	5.0 ± 0.6	5.6 ± 0.5	7.9 ± 1.3
P4	43.7 ± 6.1	43.7 ± 6.1	69.1 ± 27.7	4.8 ± 0.5	5.1 ± 0.7	7.3 ± 1.1
P5	38.0 ± 9.5	40.0 ± 9.3	53.5 ± 4.8	5.3 ± 0.8	4.9 ± 0.6	7.9 ± 0.4
P6	39.6 ± 9.9	39.6 ± 9.9	44.5 ± 20.7	3.8 ± 0.6	3.8 ± 0.6	5.4 ± 0.8

Table 5

Table 5. Methane production during the entire study.

	(1)	(2)	(3)	(4)	(5)	(6)	(7)
	CH_4 in the gas stream ($L \cdot d^{-1}$)	CH_4 total ($L \cdot d^{-1}$)	% CH_4 in biogas	SMA (mL $CH_4 \cdot g^{-1}$ $VS \cdot day^{-1}$)	CH4 yield ($L \cdot kg^{-1}$ removed COD by MA)	Total CH_4 ($L \cdot kg^{-1}$ removed COD)	% Total methane production increase in relation to P1
P1	18.2	50.0	43.5	10	77.1	51.2	-
P2	76.0	118.1	47.9	10	229.1	80.4	57
P3	129.9	165.9	67.5	36	257.0	121.1	137
P4	145.7	194.0	72.9	49	291.1	114.9	124
P5	194.3	239.6	74.7	51	340.9	148.7	190
P6	69.3	124.2	62.0	43	165.9	93.9	83

Appendix A

Table A1. Characterization of the influent waste water stream (WW) and food waste stream (FW) for each experimental period (n indicates the number of analysis performed)

Period 1			**WW**	
Parameter	**units**	**n**	**average**	**SD**
Flow	$L \cdot day^{-1}$	27	1630	154
COD	$mg\ COD \cdot L^{-1}$	4	560	64
Soluble COD	$mg\ COD \cdot L^{-1}$	4	103	22
Alkalinity	$mg\ CaCO_3 \cdot L^{-1}$	20	246	95
VFA	$mg\ HAc \cdot L^{-1}$	20	6.8	7.8
$N\text{-}NH_4$	$mg\ N \cdot L^{-1}$	17	35.6	8.9
$P\text{-}PO_4$	$mg\ P \cdot L^{-1}$	18	3.9	1
$S\text{-}SO_4$	$mg\ S \cdot L^{-1}$	17	98	26
Suspended Solids (SS)	$mg\ SS \cdot L^{-1}$	19	248	138
Volatile SS (VSS)	$mg\ VSS \cdot L^{-1}$	18	195	102
% VSS		18	79%	

Period 2			**FW**			**WW**			**Influent**	
Parameter	**units**	**n**	**average**	**SD**	**n**	**average**	**SD**	**n**	**average**	**SD**
Flow	$L \cdot day^{-1}$	30	6.89	2.15	101	2710	784	101	2717	786
COD	$mg\ COD \cdot L^{-1}$	21	80646	22	24	472	161	22	675	280
Soluble COD	$mg\ COD \cdot L^{-1}$	21	20980	17	18	102	40	17	155	90
Alkalinity	$mg\ CaCO_3 \cdot L^{-1}$	21	160	56	63	330	69	56	330	81
VFA	$mg\ HAc \cdot L^{-1}$	21	2910	56	63	9	4	56	16	23
$N\text{-}NH_4$	$mg\ N \cdot L^{-1}$	10	30	26	39	37	7	26	37	7
$P\text{-}PO_4$	$mg\ P \cdot L^{-1}$	10	78	26	39	5	4	26	5	4
$S\text{-}SO_4$	$mg\ S \cdot L^{-1}$	21	195	28	39	98	17	28	98	19
SS	$mg\ SS \cdot L^{-1}$	21	31630	17	68	223	116	17	303	186
VSS	$mg\ VSS \cdot L^{-1}$	21	31080	23	63	166	102	23	244	139
% VSS			98%	15	62	74%		15	74%	

Period 3			**FW**			**WW**			**Influent**	
Parameter	**units**	**n**	**average**	**SD**	**n**	**average**	**SD**	**n**	**average**	**SD**
Flow	$L \cdot day^{-1}$	37	4.46	1.92	128	1877	514	128	1881	516
COD	$mg\ COD \cdot L^{-1}$	33	83602	24929	36	497	150	34	694	188
Soluble COD	$mg\ COD \cdot L^{-1}$	33	22426	7986	30	93	23	30	146	42
Alkalinity	$mg\ CaCO_3 \cdot L^{-1}$	33	190	59	73	351	72	61	351	95
VFA	$mg\ HAc \cdot L^{-1}$	33	5780	2310	68	3	4	64	17	10
$N\text{-}NH_4$	$mg\ N \cdot L^{-1}$	10	32	5	45	37	8	32	37	8
$P\text{-}PO_4$	$mg\ P \cdot L^{-1}$	10	83	35	45	4	1	32	4	1
$S\text{-}SO_4$	$mg\ S \cdot L^{-1}$	33	156	28	47	102	21	33	102	33

SS	mg SS·L^{-1}	33	38743	12694	71	245	81	39	336	109
VSS	mg VSS·L^{-1}	33	38630	12365	69	198	61	37	289	87
% VSS		33	99.7%		69	81%		37	81%	

Period 4		**FW**			**WW**			**Influent**		
Parameter	**units**	**n**	**average**	**SD**	**n**	**average**	**SD**	**n**	**average**	**SD**
Flow	L·day^{-1}	33	5.79	2.62	124	2217	513	124	2223	516
COD	mg COD·L^{-1}	28	69455	20130	35	618	185	35	797	205
Soluble COD	mg COD·L^{-1}	28	19438	6035	35	121	29	34	171	35
Alkalinity	mg $CaCO_3$·L^{-1}	28	162	90	58	297	68	42	297	79
VFA	mg HAc ·L^{-1}	28	1725	939	56	6	5	50	10	11
N-NH_4	mg N·L^{-1}	20	36	9	39	43	7	31	43	7
P-PO_4	mg P·L^{-1}	20	82	39	41	4	1	32	4	1
S-SO_4	mg S·L^{-1}	28	224	96	43	114	13	35	114	15
SS	mg SS·L^{-1}	28	39484	16400	70	302	107	31	404	123
VSS	mg VSS·L^{-1}	28	39480	14500	68	248	88	30	350	106
% VSS		28	100%		68	82%		30	82%	

Period 5		**FW**			**WW**			**Influent**		
Parameter	**units**	**n**	**average**	**SD**	**n**	**average**	**SD**	**n**	**average**	**SD**
Flow	L·day^{-1}	10	12.15	3.15	48	2026	546	48	2038	549
COD	mg COD·L^{-1}	9	71872	16518	9	564	182	9	989	206
Soluble COD	mg COD·L^{-1}	9	21795	7982	8	117	25	8	246	54
Alkalinity	mg $CaCO_3$·L^{-1}	9	120	50	18	301	74	14	300	65
VFA	mg HAc ·L^{-1}	9	2820	1781	18	8	3	15	25	10
N-NH_4	mg N·L^{-1}	9	28	9	7	38	8	6	38	8
P-PO_4	mg P·L^{-1}	9	89	26	7	6	2	6	6	2
S-SO_4	mg S·L^{-1}	9	140	55	8	123	16	8	123	18
SS	mg SS·L^{-1}	9	30015	14220	22	316	96	7	493	84
VSS	mg VSS·L^{-1}	9	29760	14350	20	262	58	7	438	56
% VSS			99%		22	83%		7	83%	

Period 6		**WW**		
Parameter	**units**	**n**	**average**	**SD**
Flow	L·day^{-1}	79	2223	359
COD	mg COD·L^{-1}	9	541	188
Soluble COD	mg COD·L^{-1}	9	101	33
Alkalinity	mg $CaCO_3$·L^{-1}	27	366	83
VFA	mg HAc ·L^{-1}	27	5.3	6
N-NH_4	mg N·L^{-1}	7	38	10
P-PO_4	mg P·L^{-1}	6	4.9	2

$S\text{-}SO_4$	mg $S \cdot L^{-1}$	9	124	19
SS	mg $SS \cdot L^{-1}$	39	223	70
VSS	mg $VSS \cdot L^{-1}$	32	174	87
% VSS		32	78%	

Statistical test for the Total OLR results

Table B.1. ANOVA Table for Total OLR

Source	*Sum of Squares*	*Df*	*Mean Square*	*F-Ratio*	*P-Value*
Between groups	0.998035	5	0.199607	5.29	0.0010
Within groups	1.32172	35	0.0377635		
Total (Corr.)	2.31976	40			

The ANOVA table decomposes the variance of OLR total into two components: a between-group component and a within-group component. The F-ratio, which in this case equals 5.28571, is a ratio of the between-group estimate to the within-group estimate. Since the P-value of the F-test is less than 0.05, there is a statistically significant difference between the mean OLR total from one Period to another at the 95.0% confidence level.

Table B.2. Multiple Range Tests for Total OLR

Method: 95.0 percent LSD

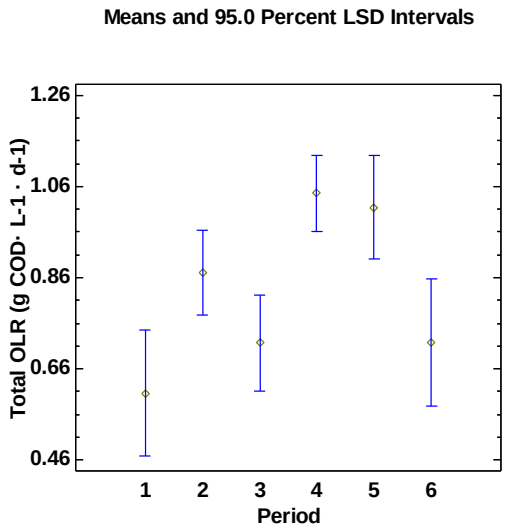
Contrast	*Sig.*	*Difference*	*+/- Limits*
1 - 2	*	-0.264566	0.23707
1 - 3		-0.109808	0.247271
1 - 4	*	-0.438615	0.230343
1 - 5	*	-0.40711	0.254654
1 - 6		-0.110236	0.27896
2 - 3		0.154758	0.198814
2 - 4		-0.174049	0.177318
2 - 5		-0.142544	0.207924

Contrast	*Sig.*	*Difference*	*+/- Limits*
2 - 6		0.15433	0.23707
3 - 4	*	-0.328807	0.190742
3 - 5	*	-0.297302	0.219484
3 - 6		-0.000427999	0.247271
4 - 5		0.0315047	0.200221
4 - 6	*	0.328379	0.230343
5 - 6	*	0.296874	0.254654

* denotes a statistically significant difference.

This table applies a multiple comparison procedure to determine which means are significantly different from which others. The output shows the estimated difference between each pair of means. An asterisk has been placed next to 7 pairs, indicating that these pairs show statistically significant differences at the 95.0% confidence level. The method currently being used to discriminate among the means is Fisher's least significant difference (LSD) procedure. With this method, there is a 5.0% risk of calling each pair of means significantly different when the actual difference equals 0.

Figure B.1. Table of Means for Total OLR (LSD procedure)



Statistical test for the SRB OLR results

Table B.3. ANOVA Table for SRB OLR

Source	*Sum of Squares*	*Df*	*Mean Square*	*F-Ratio*	*P-Value*
Between groups	0.0111237	5	0.00222475	0.41	0.8400
Within groups	0.190882	35	0.00545378		
Total (Corr.)	0.202006	40			

Since the P-value of the F-test is greater than or equal to 0.05, there is not a statistically significant difference between the mean OLR SRB from one level of P to another at the 95.0% confidence level.

Table B.4. Multiple Range Tests for SRB OLR

Method: 95.0 percent LSD

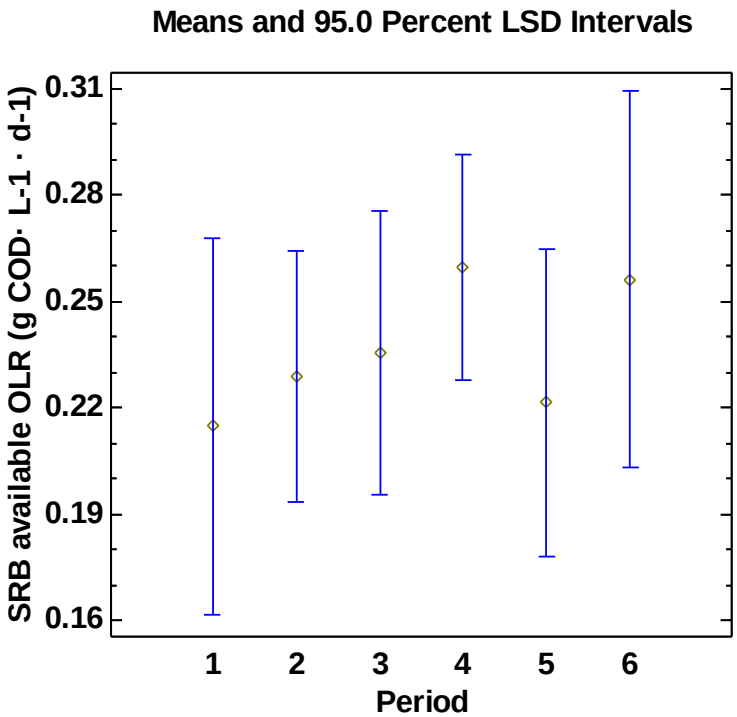
Contrast	*Sig.*	*Difference*	*+/- Limits*
1 - 2		-0.0140278	0.0900927
1 - 3		-0.0206786	0.0939694
1 - 4		-0.0449773	0.0875363
1 - 5		-0.00675	0.096775
1 - 6		-0.0415	0.106012
2 - 3		-0.00665079	0.0755542
2 - 4		-0.0309495	0.0673855
2 - 5		0.00727778	0.0790165

Contrast	*Sig.*	*Difference*	*+/- Limits*
2 - 6		-0.0274722	0.0900927
3 - 4		-0.0242987	0.072487
3 - 5		0.0139286	0.0834096
3 - 6		-0.0208214	0.0939694
4 - 5		0.0382273	0.0760889
4 - 6		0.00347727	0.0875363
5 - 6		-0.03475	0.096775

* denotes a statistically significant difference.

This table applies a multiple comparison procedure to determine which means are significantly different from which others. The output shows the estimated difference between each pair of means. There are no statistically significant differences between any pair of means at the 95.0% confidence level.

Figure B.2. Table of Means for SRB OLR (LSD procedure)



Statistical test for the MA OLR results

Table B.5. ANOVA Table for MA OLR

Source	*Sum of Squares*	*Df*	*Mean Square*	*F-Ratio*	*P-Value*
Between groups	0.926686	5	0.185337	7.34	0.0001
Within groups	0.883349	35	0.0252386		
Total (Corr.)	1.81004	40			

Since the P-value of the F-test is less than 0.05, there is a statistically significant difference between the mean OLR MA from one level of P to another at the 95.0% confidence level.

Table B.6. Multiple Range Tests for MA OLR
Method: 95.0 percent LSD

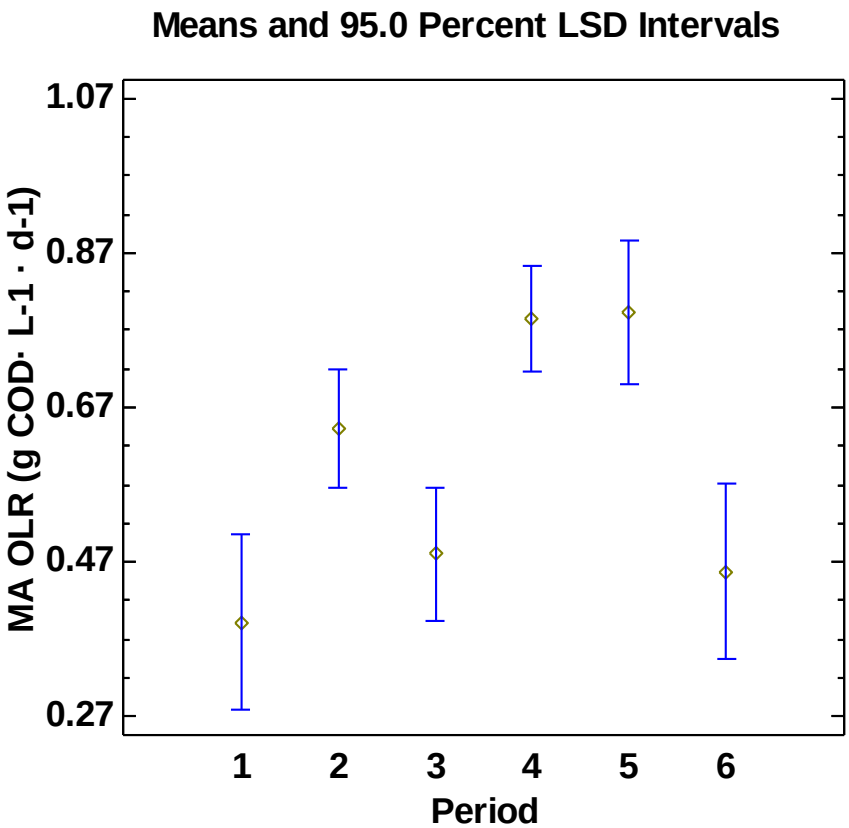
Contrast	*Sig.*	*Difference*	*+/- Limits*
1 - 2	*	-0.250556	0.193808
1 - 3		-0.089	0.202148
1 - 4	*	-0.393545	0.188309
1 - 5	*	-0.4005	0.208184
1 - 6		-0.0655	0.228054
2 - 3		0.161556	0.162533
2 - 4		-0.14299	0.14496
2 - 5		-0.149944	0.169981

Contrast	*Sig.*	*Difference*	*+/- Limits*
2 - 6		0.185056	0.193808
3 - 4	*	-0.304545	0.155935
3 - 5	*	-0.3115	0.179432
3 - 6		0.0235	0.202148
4 - 5		-0.00695455	0.163683
4 - 6	*	0.328045	0.188309
5 - 6	*	0.335	0.208184

* denotes a statistically significant difference.

An asterisk has been placed next to 7 pairs, indicating that these pairs show statistically significant differences at the 95.0% confidence level.

Figure B.3. Table of Means for MA OLR (LSD procedure)



Statistical test for the WW OLR results

Table B.7. ANOVA Table for WW OLR
ANOVA Table for OLR WW by P2

Source	*Sum of Squares*	*Df*	*Mean Square*	*F-Ratio*	*P-Value*
Between groups	0.348194	3	0.116065	11.56	0.0000
Within groups	0.291238	29	0.0100427		
Total (Corr.)	0.639431	32			

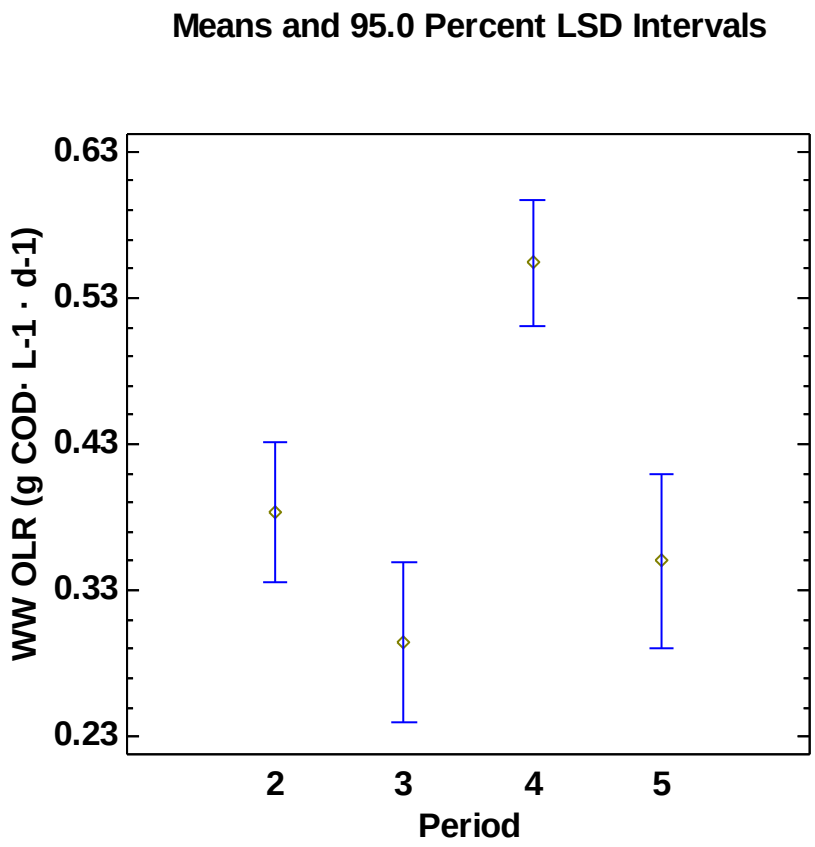
Since the P-value of the F-test is less than 0.05, there is a statistically significant difference between the mean OLR WW from one level of P2 to another at the 95.0% confidence level.

Table B.8. Multiple Range Tests for WW OLR

Contrast	*Sig.*	*Difference*	*+/- Limits*
2 - 3		0.0890159	0.10329
2 - 4	*	-0.170465	0.0921224
2 - 5		0.0332778	0.108023
3 - 4	*	-0.259481	0.0990966
3 - 5		-0.0557381	0.114029
4 - 5	*	0.203742	0.104021

* denotes a statistically significant difference.

Figure B.4. Table of Means for WW OLR (LSD procedure)



Statistical test for the FW OLR results

Table B.9. ANOVA Table for FW OLR

Source	*Sum of Squares*	*Df*	*Mean Square*	*F-Ratio*	*P-Value*
Between groups	0.243674	3	0.0812248	10.77	0.0001
Within groups	0.218728	29	0.00754234		
Total (Corr.)	0.462402	32			

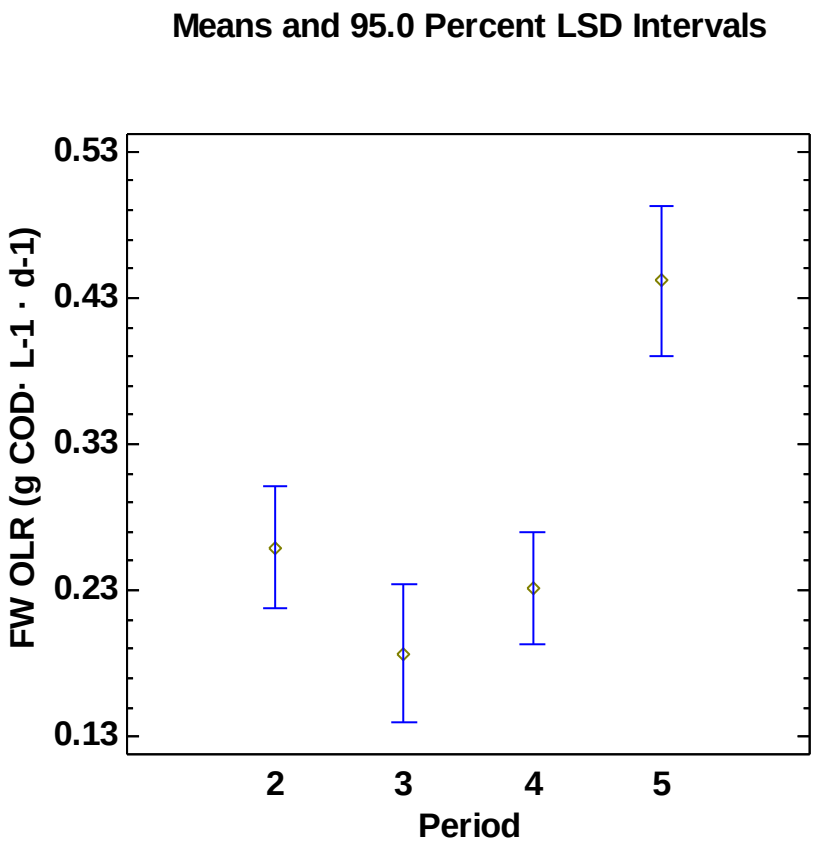
Since the P-value of the F-test is less than 0.05, there is a statistically significant difference between the mean OLR FW from one level of P2 to another at the 95.0% confidence level.

Table B.10. Multiple Range Tests for FW OLR

Contrast	*Sig.*	*Difference*	*+/- Limits*
2 - 3		0.0725397	0.0895129
2 - 4		0.0274747	0.079835
2 - 5	*	-0.183056	0.0936149
3 - 4		-0.0450649	0.085879
3 - 5	*	-0.255595	0.0988196
4 - 5	*	-0.21053	0.0901465

* denotes a statistically significant difference.

Figure B.5. Table of Means for FW OLR (LSD procedure)



Statistical test for the TS reactor results

Table B.11. ANOVA Table for TS reactor

Source	*Sum of Squares*	*Df*	*Mean Square*	*F-Ratio*	*P-Value*
Between groups	2.30418E9	5	4.60837E8	1392.48	0.0000
Within groups	3.0778E7	93	330946.		
Total (Corr.)	2.33496E9	98			

Since the P-value of the F-test is less than 0.05, there is a statistically significant difference between the mean TS reactor from one level of P ST to another at the 95.0% confidence level.

Table B.12. Multiple Range Tests for TS reactor

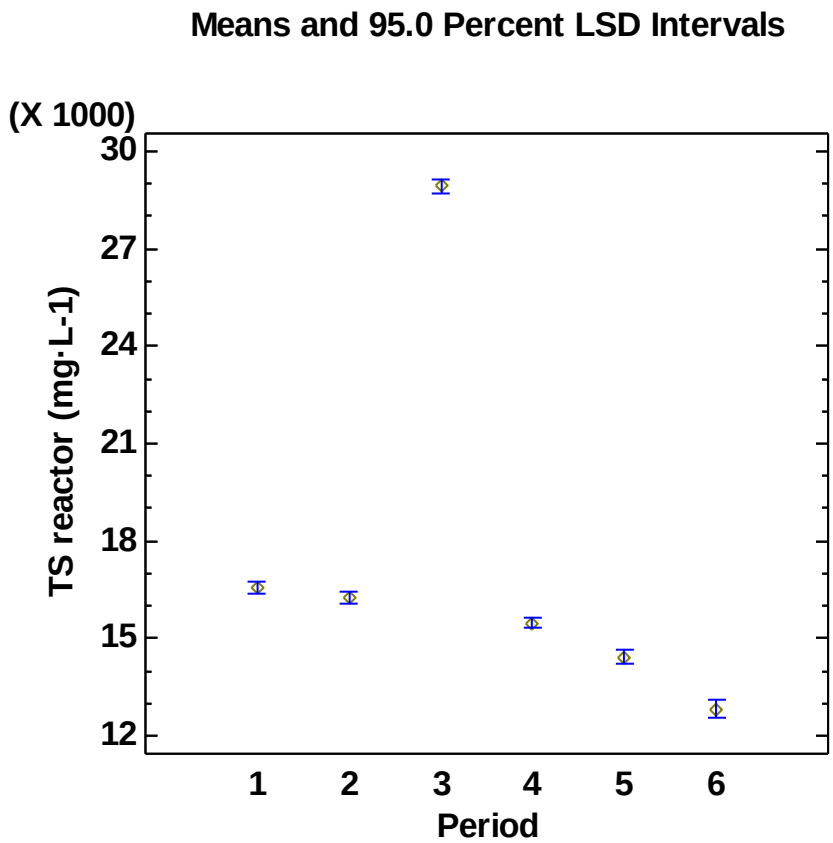
Method: 95.0 percent LSD

Contrast	*Sig.*	*Difference*	*+/- Limits*
1 - 2		302.167	376.008
1 - 3	*	-12387.0	384.183
1 - 4	*	1072.44	337.192
1 - 5	*	2138.7	376.008
1 - 6	*	3725.5	446.525
2 - 3	*	-12689.1	424.527
2 - 4	*	770.273	382.525
2 - 5	*	1836.53	417.143

Contrast	*Sig.*	*Difference*	*+/- Limits*
2 - 6	*	3423.33	481.675
3 - 4	*	13459.4	390.563
3 - 5	*	14525.7	424.527
3 - 6	*	16112.5	488.084
4 - 5	*	1066.26	382.525
4 - 6	*	2653.06	452.026
5 - 6	*	1586.8	481.675

* denotes a statistically significant difference.

Figure B.6. Table of Means for TS reactor (LSD procedure)



Statistical test for the Sludge production results

Table B.13. ANOVA Table for Sludge production

Source	*Sum of Squares*	*Df*	*Mean Square*	*F-Ratio*	*P-Value*
Between groups	0.717873	5	0.143575	18.28	0.0000
Within groups	0.180676	23	0.0078555		
Total (Corr.)	0.89855	28			

Since the P-value of the F-test is less than 0.05, there is a statistically significant difference between the mean Sludge prod from one level of P Sludge to another at the 95.0% confidence level.

Table B.14. Multiple Range Tests for Sludge production

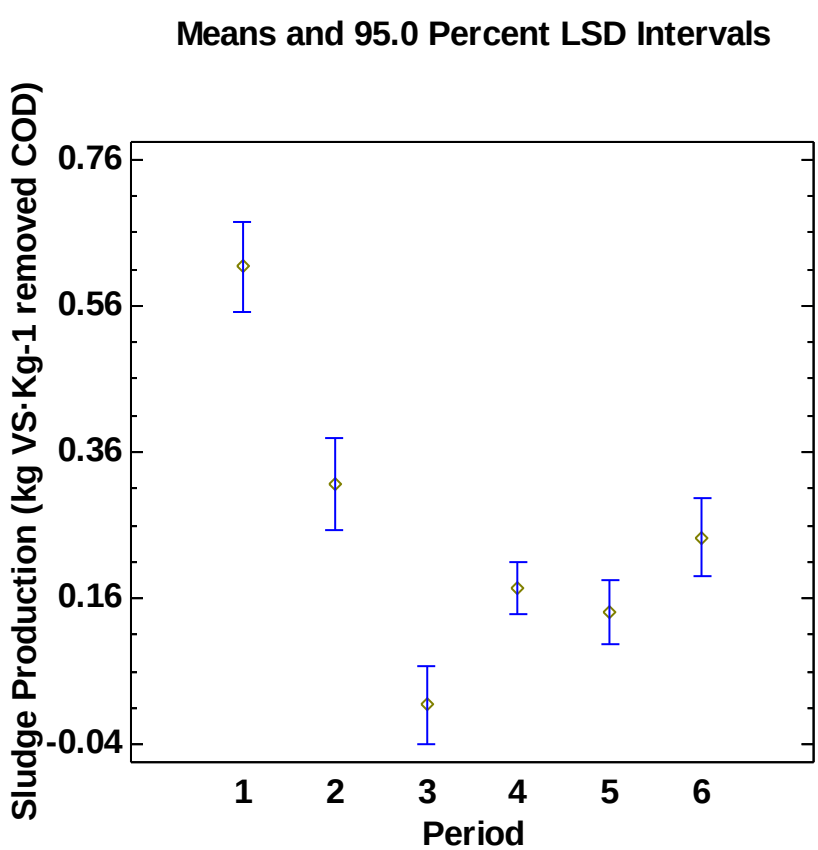
Method: 95.0 percent LSD

Contrast	*Sig.*	*Difference*	*+/- Limits*
1 - 2	*	0.297242	0.149703
1 - 3	*	0.599097	0.140034
1 - 4	*	0.438796	0.122232
1 - 5	*	0.471483	0.129647
1 - 6	*	0.370303	0.140034
2 - 3	*	0.301855	0.140034
2 - 4	*	0.141553	0.122232
2 - 5	*	0.17424	0.129647

Contrast	*Sig.*	*Difference*	*+/- Limits*
2 - 6		0.0730607	0.140034
3 - 4	*	-0.160302	0.110178
3 - 5	*	-0.127615	0.118351
3 - 6	*	-0.228794	0.129647
4 - 5		0.0326869	0.0966329
4 - 6		-0.0684927	0.110178
5 - 6	*	-0.10118	0.118351

* denotes a statistically significant difference.

Figure B.7. Table of Means for Sludge production (LSD procedure)



Statistical test for the COD/S-SO_4 ratio results

Table B.15. ANOVA Table for COD/S-SO_4 ratio

Source	*Sum of Squares*	*Df*	*Mean Square*	*F-Ratio*	*P-Value*
Between groups	27.9032	5	5.58064	1.81	0.1382
Within groups	98.4379	32	3.07618		
Total (Corr.)	126.341	37			

Since the P-value of the F-test is greater than or equal to 0.05, there is not a statistically significant difference between the mean Ratio from one level of P ratio to another at the 95.0% confidence level.

Table B.16. Multiple Range Tests for COD/S-SO_4 ratio

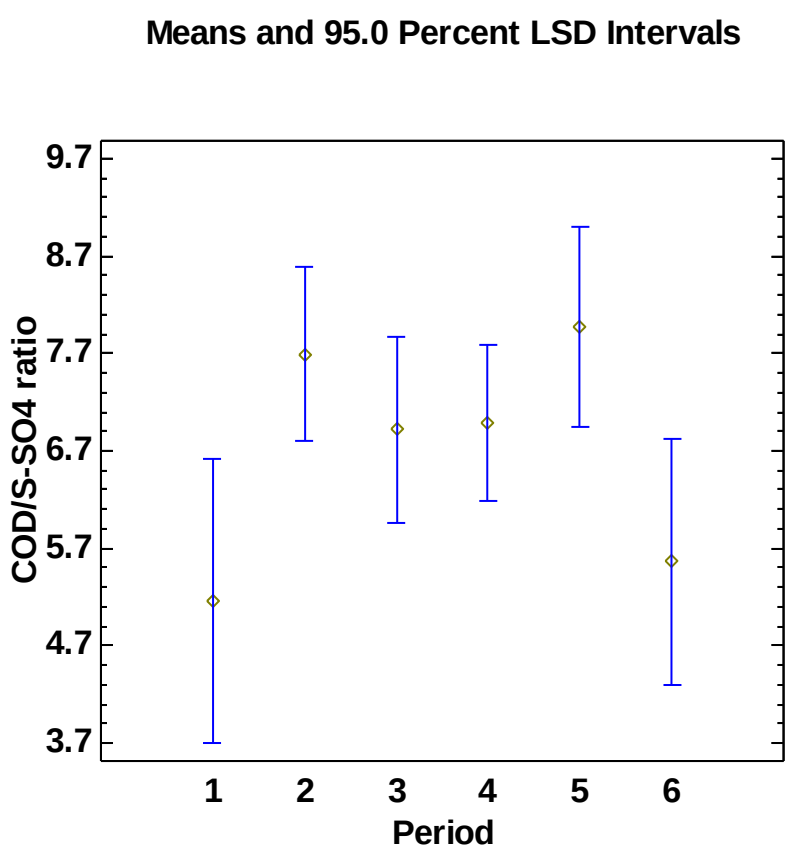
Method: 95.0 percent LSD

Contrast	*Sig.*	*Difference*	*+/- Limits*
1 - 2	*	-2.52594	2.41866
1 - 3		-1.75826	2.46533
1 - 4		-1.82519	2.35177
1 - 5	*	-2.81042	2.52621
1 - 6		-0.399901	2.72861
2 - 3		0.767687	1.84899
2 - 4		0.700755	1.69463
2 - 5		-0.284475	1.92942

Contrast	*Sig.*	*Difference*	*+/- Limits*
2 - 6		2.12604	2.18776
3 - 4		-0.0669318	1.76059
3 - 5		-1.05216	1.98761
3 - 6		1.35836	2.23924
4 - 5		-0.98523	1.84488
4 - 6		1.42529	2.11358
5 - 6	*	2.41052	2.3061

* denotes a statistically significant difference.

Figure B.8. Table of Means for COD/S-SO_4 ratio (LSD procedure)



Statistical test for the Total methane production ratio results

Table B.17. ANOVA Table for Total methane production

Source	*Sum of Squares*	*Df*	*Mean Square*	*F-Ratio*	*P-Value*
Between groups	512824.	5	102565.	40.23	0.0000
Within groups	346690.	136	2549.19		
Total (Corr.)	859514.	141			

Since the P-value of the F-test is less than 0.05, there is a statistically significant difference between the mean CH4 (L) from one level of Peri to another at the 95.0% confidence level.

Table B.18. Multiple Range Tests for Total methane production

Method: 95.0 percent LSD

Contrast	*Sig.*	*Difference*	*+/- Limits*
1 - 2	*	-67.9797	29.2747
1 - 3	*	-115.903	26.7534
1 - 4	*	-144.016	26.4866
1 - 5	*	-189.616	30.9094
1 - 6	*	-74.2137	27.344
2 - 3	*	-47.9229	30.1352
2 - 4	*	-76.0363	29.8987
2 - 5	*	-121.637	33.8789

Contrast	*Sig.*	*Difference*	*+/- Limits*
2 - 6		-6.23404	30.6608
3 - 4	*	-28.1134	27.4348
3 - 5	*	-73.7136	31.7256
3 - 6	*	41.6889	28.2634
4 - 5	*	-45.6002	31.501
4 - 6	*	69.8023	28.0111
5 - 6	*	115.403	32.2252

* denotes a statistically significant difference.

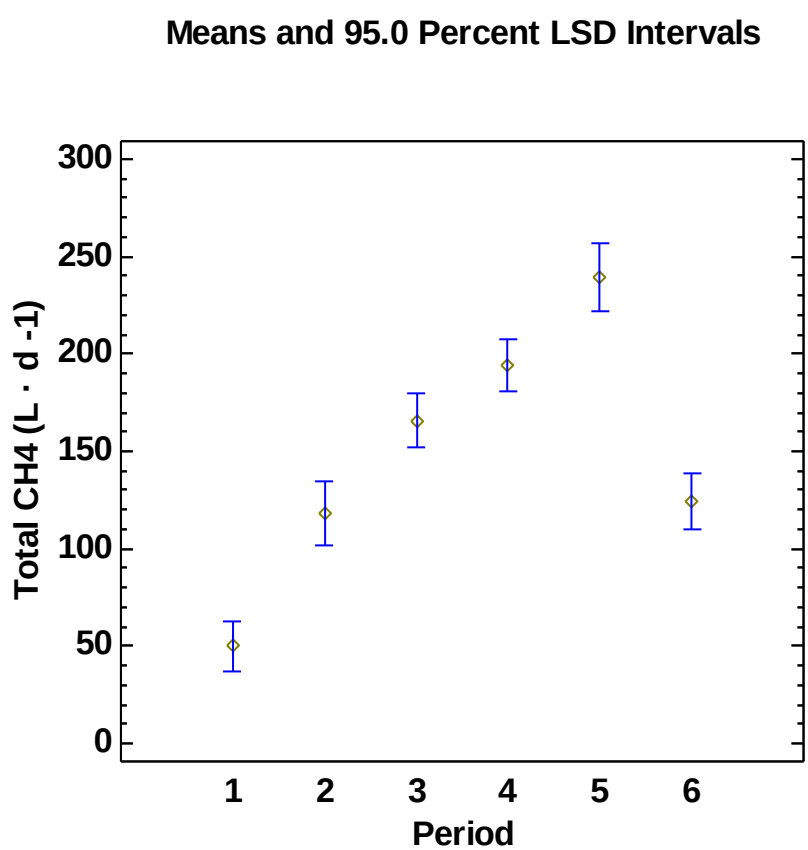



Figure B.9. Table of Means for Total methane production (LSD procedure)