Mesophilic anaerobic co-digestion of the sewage sludge with glycerine: Effect of solid retention time.

Zahedi, S.[ab]; Rivero, M. [a], Solera, R.[a]; Perez, M.[a*]

[a]Department of Environmental Technologies. University of Cadiz. Faculty of Marine and Environmental Sciences (CASEM) Pol, Río San Pedro s/n, 11510 Puerto Real (Cádiz), Spain. (soraya.zahedi@uca.es; maria.rivero@alum.uca.es; rosario.solera@uca.es; montserrat.perez@uca.es)

[b]Catalan Institute for Water Research (ICRA), Scientific and Technological Park of the University of Girona, 17003, Spain (zahedi.diaz@gmail.com)

*Corresponding author. montserrat.perez@uca.es;/ Tel.: +34 956016158

ABBREVIATION LIST

AD: anaerobic digestion

COD: chemical oxygen demand

GP: gas production

HRT: Hydraulic retention time

MP: methane production

OLR: organic loading rate

SMP: specific methane production

SRT. Solid retention time

TS: total solids

VFA: volatile fatty acids

VS: volatile solids

WWTP: wastewater treatment plant

Abstract

The main objective of this paper was to examine the effect of the increase of organic loading rates (OLRs) (by reducing solid retention time, SRT, from 20 d to 5 d) in single-phase mesophilic anaerobic co-digestion of the sewage sludge with glycerine (1% v/v). Experimentally, it was confirmed that anaerobic co-digestion of these biowastes in steady-state conditions can achieve 85±5% of volatile fatty acid (VFA) reduction at SRTs between 20 and 9 d, with a methane production around 0.8 l CH_4/l/d. Decreases in the SRT not only allow maintaining the sludge stability and the biogas production, but it also implies an increase in the waste that could be treated and lower cost. Therefore, mesophilic anaerobic co-digestion of sewage sludge and glycerin at SRT lower than 20 d is possible and preferable, due to it is more economical and environmental friendly.

Keywords: anaerobic co-digestion; biogas, SRT, mesophilic, sewage sludge, glycerine

1. Introduction

Disposal of sewage sludge as a main byproduct generated in wastewater treatment plants (WWTPs) is a major challenge which typically represents up to 50% of the overall operating costs of a WWTP [1,2]. Anaerobic sludge digestion as a reliable technology employed worldwide to stabilize organics and reduce solids, destroy pathogens and produce biogas as the source of energy [2–7]. Due to the advantages of anaerobic digestion (AD), many research studies have sought to optimize the AD of sludge, including the interesting option of the co-digestion process [8], which increases the load of biodegradable organic matter and produces a higher biogas yield. Recent studies have been demonstrated the efficacy of anaerobic co-digestion of municipal sludge or solid waste together with readily biodegradable organic substances, such as glycerol, a major by-product of biodiesel production [1,9–14]. Production of 100 kg of biodiesel yields approximately 10 kg of glycerin waste as a co-product. Numerous industries such as pharmaceutical, cosmetics and food processing, use refined glycerol as a raw input material. However, the glycerol generated, as a co-product of biodiesel production requires purification before being suitable for use in these industries. Therefore, the glycerol is often considered waste stream instead of a co-product [11], which makes its disposal a fundamental environmental concern. Most of the recent studies about AD of glycerin and sludge or municipal solid waste to improve the methane production (MP) have been focused to identify the optimal concentration (%, v/v) of glycerin that have to be added into the substrate [1,9–11,13,14] and very few have studied the effect on semi-continuous or continuous feeding regime [1,11] and none at optimization of solid retention time (SRT) during the anaerobic co-digestion of sludge and glycerol in a single phase reactor to MP. SRT optimization is important, since low SRT are preferred for real application, reducing the volume of the anaerobic digester and the WWTP cost. Based on these premises the present study has been developed. The experimental protocol was designed to examine the effect of the increase of organic loading rate (by reducing the solid retention time, SRT, from 20 d to 5 d) on the efficiency of stirred tank reactor treating sewage sludge and glycerin and to report on its steady-state performance. The experimental reactor was subjected to a program of steady-state operation over a range of solid retention times, SRTs, from 20 - 5 d and organic loading rates (OLRs), from 1.03 to up to 4.05 g COD/l/d in order to evaluate its treatment capacity.

2. Materials and Methods

2.1. Substrates and inoculum

Experimental work was carried out with sewage sludge samples (mixed primary sludge and activated sludge) from Cadiz-San Fernando WWTP (located in Cadiz-Spain, which handles more than 50,000 m^3 of wastewater daily). All the sludge samples were characterized on reception at the laboratory and were kept under refrigeration at 4 °C before they were used for the experiments so as to prevent biodegradation. This sludge was mixed with 1% v/v glycerol commercial household Panreac, which constituted the reactor feed. According to Fountoulakis et al. [14], the most appropriate concentration of glycerol in co-digestion with sewage sludge in anaerobic processes is 1%. The main characteristics of the sewage sludge are summarized in Table 1.

Regarding to the inoculum, it was collected from the mesophilic anaerobic digester present at the same WWTP. The pH, total solids (TS) and volatile solids (VS) were 7.5± 0.2; 32.0 ± 2.0 g TS/kg and 18.0 ± 0.2 g VS/kg, respectively.

2.2. Experimental equipment and operation conditions

The laboratory-scale reactor used for this study operates in a semi-continuous stirred tank reactor and in the mesophilic range (35ºC). The equipment consists of a reactor with a stainless steel vessel that is agitated and heated and that has a total volume of 5 L and a working volume of 4.5 L (Figure 1). No biomass recycling was used; the hydraulic retention time (HRT) and the Solid Retention Time (SRT) are equal. The reactor features a lid that allows it to be sealed to maintain anaerobic conditions within the reactor.

The stainless steel lid has three openings (one for the biogas outlet, a feed inlet and another opening for the stirring system). The bottom of the reactor has a release valve used for sampling the material inside the reactor, which is made possible by the sealing system between the vessel and the cap. The assembly includes an agitator that achieves the homogenisation of waste using stainless steel blade scrapers. To maintain the operating temperature (mesophilic, 35 ºC), the reactor is heated by recirculating water through a thermostatic jacket. Biogas is collected in 40-L Tedlar bags and a special syringe is used for sampling gases.

Regarding to the operation conditions, the reactor was fed with sewage sludge and glycerin (1%) without nutrients and pH correction once a day (semi-continuous regime). Based on information found in the literature and the previous experience of the group [15,16], SRTs of 20, 15, 9, 7 and 5 days were selected for study until the process breakdown. Figure 2 describe the SRTs and its corresponding OLRs applied to the reactor during the experiment. The overall duration of the experiments was 255 d.

2.3. Analytical methods

To characterise the waste and the inoculum, as well as to monitor the effluent of the process, the following were analysed: pH, alkalinity, volatile fatty acids (VFA), total chemical oxygen demand (TCOD), total solids (TS) and volatile solids (SV). These analyses were conducted in accordance with standard methods (APHA, 1995) and Zahedi et al. [9]. The gas volume produced in the reactors was directly measured using a high-precision flow gas meter: Ritter_ drum-type gas meter TG-01-Series (Wet-Type). VFA were determined by gas chromatography, using a gas chromatograph (Shimadzu GC-2010) equipped with a flame ionization detector (FID) and a capillary column filled with Nukol. The gas volume produced in the reactor was directly measured using a high-precision flow gas meter: TG-01-Series (Wet-Type) Ritter drum-type gas meter. The biogas composition was determined by gas chromatography separation (SHIMADZU GC-2010). H_2, CH_4, CO_2, O_2 and N_2 were analysed by means of a thermal conductivity detector (TCD) employing a Supelco Carboxen 1010 Plot column. Samples were taken using a 1 ml Dynatech Gastight gas syringe under the following operating conditions: split = 100; constant pressure in the injection port (70 kPa); 2 min at 40 ºC; ramped at 40 ºC/min until 200 ºC; 1.5 min at 200 ºC; detector temperature: 250 ºC; and injector temperature: 200ºC. Helium was used as carrier gas (266.2 ml/min). Commercial mixtures of H_2, CH_4, CO_2, O_2, N_2 and H_2S (Abelló Linde S.A.) were used to calibrate the system.

Gas volume and composition were measured daily; in the effluent, the pH was measured daily in all condition assayed. VS, TCOD, alkalinity and VFA were analysed approximately three times a week**.**

3. Results and discussion

This section discusses the evolution of the main variables during the semi-continuous mesophilic anaerobic digestion process, such as pH, VFA, alkalinity, TCOD, VS and biogas production and composition. All the values correspond to the analytical determinations in steady conditions (except at 5 d SRT, because destabilization was observed). The number of determinations in steady conditions considered to present the averages and standard values of biogas and pH were 35, 20, 12, 11 and 9 for 20, 15, 10, 9 and 7 d SRT, respectively; and the analyses (in steady conditions) considered to present the averages and standard values VFA, COD, alkalinity and VS were 9, 8, 12, 9 and 8 for 20, 15, 10, 9 and 7 d SRT. Date of 5 d SRT are unstable and they only are represented to show the system destabilization.

3.1. Process stability: pH evolution and VFA/alkalinity ratio

The stability of the process was evaluated based on pH and the VFA/alkalinity ratio at different SRT tested [9,18]. pH was used to evaluate the stability along the process and VFA/alkalinity ratio was used to establish under which conditions (SRT) the mesophilic anaerobic co-digestion of the sewage sludge with glycerine could operate without jeopardising its safety.

pH is a basic parameter for control of the anaerobic degradation process. Figure 3 shows the evolution of pH during the semi-continuous mesophilic study. In this figure, vertical lines are included to indicate the changes in SRT and red horizontal dashed line indicates the 7.0 pH. Initially, with an SRT of 20 d, 15 d and 12 d, pH values stabilise at approximately 7.3-7.8, the optimum pH for the activity of methanogenic microorganisms [9,19]. With an SRT of 9 d, the pH decrease during the first 5 d until it reached a value 6.44, as result of the increased OLR feed, but finally it was increase and stabilised at 7.3-7.5, without the addition of an external agent (the reactor was no feeding per two days and it was recovered). The initial decrease in pH when an increase in the OLR is applied into the reactor may be due to the initial imbalance between the metabolic activities of microbial groups. When the added load is increased, the acidogenic microorganisms respond quickly, given their high specific growth rate and generate more VFA. However, methanogenic archaea are slower and require more time to grow and reach the population size necessary to degrade the excess of VFA.

Finally, the decreases in the SRT at 5 d supposed the pH dropped to values bellow 6 and the reactor was not recovered. At this condition, the pH decreases as a result of the accumulation of VFA in the reactor due to methanogenic archaea were not able to degrade the excess of VFA produced by hydrolitic-acidogenic bacteria [15,16,20], indicating acidification of the medium and thus destabilize the process. In short, taking to account the pH values it could be said that single-phase mesophilic anaerobic co-digestion of the sewage sludge with glycerine (1% v/v) is totally steady at SRTs between 20 and 7 d.

As previously mentioned, VFA/alkalinity ratio was also considered, to establish under which conditions (SRT) the mesophilic anaerobic co-digestion of the sewage sludge with glycerine could operate without jeopardising its safety. This parameter have been used to evaluate the stability of the process during the AD of waste and glycerol [9]. The medium values of these ratios are shown Figure 4. Values between 0.1 and 0.4 (equiv. acetic acid/equiv. $CaCO_3$) indicate favourable operating conditions without the risk of acidification. In this figure, horizontal dashed line indicates the 0.4 value. At SRT higher than 7, this parameter was under 0.4 (equiv. acetic acid/equiv. $CaCO_3$) indicating a proper performance. At 7 d SRT, VFA/alkalinity ratio was slightly higher than the optimum values, indicating risk of acidification. Therefore, at 7 d of SRT while the pH values were maintained high so as to allow methanogenic activity, the acids generated during the acidogenic are not totally consumed and some of them are accumulated in the system, thus start to affect the activity of the anaerobic consortia and a reduction in the organic matter removal is observed, as will be explained later. At but at SRT of 5 d this parameter was too high, indicating total system destabilization. This effect (high values for both, pH and VFA/alkalinity ratio) has been also detected in under non-stable AD process of glycerin and biowastes [9]. In short, taking to account the pH and VFA/alkalinity ratios values it could be said that single-phase mesophilic anaerobic co-digestion of the sewage sludge with glycerine (1% v/v) is totally steady at SRTs between 20 and 9 d. At 7 d of SRT the acids generated during the acidogenic phase start to accumulate in the system. Therefore, although in this study the system has been able to maintain the pH values around 7.0-7.4, 7 d of SRT could be considered a critical time to operate to mesophilic anaerobic co-digestion of the sewage sludge with glycerine (1% v/v), especially in real-industrial WWTP digester.

3.2. Organic matter removal

Figure 5 shows the removal efficiencies of VFA, TCOD and VS (as %) in the mesophilic reactor for different SRTs. For 20 d SRT, VFA, TCOD reaches approximately 60% TCOD removal, 50% VS removal and 85% VFA removal. Similar values are found in the other stages of the operation until 7 d SRT. At 7 d a huge decrease in the organic matter was detected consumption was detected VFA, TCOD and VS reaches a small value (25%VFA removal; 15% TCOD removal and 30%VS removal). At 5 d the organic matters removals were lower that between 4-10%. Taking into account these parameters we can ensure that at 7 d STR the reactor is not degrading properly organic matter and at 5 d SRT the reactor is not able to assimilate the ORL feed and consequently the pH decreases in the system and produces an accumulation of VFA, and it implied an increase in VFA/alkalinity ratio, as seen in previous sections. In short, breakdown efficiency starts at 7 d SRT and retention times shorter than 7 d are insufficient for a stable digestion of mixed sludge and glycerin (1% v/v).

Logically, the best results for TCOD concentration and VFA, in terms of the quality of the effluent, were obtained in the range 20-9 d SRT. The total acidity, expressed as the total amount VFA represented by acetic acid and TCOD, exhibits stable values in the effluent from the mesophilic reactor in the range 395 - 155 mg acetic/l and 6-8 g O_2/l, respectively, at SRTs between 20 and 9 d. When the SRT is changed to 7 d, a significant difference in average total acidity is observed, with a value of 1640 mg acetic/l, due to the increased organic load supplied to the system. This trend illustrates the initial destabilisation caused by the reduction in the SRT, as discussed above. However, at the end of the 5 d SRT, the average acidity values are close to 12000 mg acetic/l of acetic acid indicating total destabilization in the system. The increase in the VFA when glycerine is added into the feed has been reported in several studies [9,11,14,21]. Holm-Nielsen et al. [21] studied the anaerobic digestion of a mixture of manure, waste from food industries and glycerol added to the reactor gradually. The authors observed the accumulation of volatile fatty acids and glycerol in the reactor with the addition of 3.5 to 6.5% of glycerol (v/v), from the 16th to the 19th day of the experiment, which caused the inhibition of the methanogenic phase. Razaviarani et al. [11] observed that the accelerated increase in VFA concentration in the test digester and decrease in the biogas CH_4 content suggest that methanogens inhibition occurred at supplementation of glycerin of 1.8% (v/v) were added to municipal sludge at SRT of 20 d. In a similar study, Fountoulakis et al. [14] reported that adding 3% (v/v) glycerine to sewage sludge resulted in VFA accumulation and process instability at 23–25 d of SRT. Zahedi et al. [9] study explores the effect of five different glycerol supplementations (0%, 0.1%, 0.25%, 0.5% and 1%) on effluent characteristics, anaerobic consortia and MP in batch mode and they observed that during the acidogenic phase of anaerobic VFA were accumulated at supplementation of glycerin of 0.5% (v/v). However the effect of tat different SRT and for a constant value of glycerin, as the present study is worked, has not been related yet. The destabilization in the present paper is not due to the high values of glycerin as the other researchers discussed above, since at AD of sewage sludge and glycerin (1% v/v) at SRT between of 20-9 d VFA were very low (395 - 155 mg acetic/l). The destabilization was due to over load produced by a decrease in the SRT.

3.3. Biogas

Fig. 6 shows the medium values of biogas production (l /l reactor/d) and methane production (l CH_4 /l reactor/d) at every condition tested from SRT of 20 d to 5 d. At SRT from 20 to 9 d SRT, the medium values of GP and MP were ranged between 1.21-1.43 l/l/d and between 0.6-0.9 l CH_4/l/d respectively. For SRT lower than 9 d, the tendency changes and a drop in both GP and MP are observed indicating overload or destabilization, especially at 5 d SRT in which an extremely decrease was noted, coinciding with the total destabilization of the system. It was in line with the other parameters (pH and organic matter removal decrease and VFA increase). For the higher SRT (20d, 15 d, 12 d and 9 d), the values of the MP and specific methane production (SMP, ml of methane per TCOD consumed) were ranged between 0.6-0.9 l CH_4/l/d and between 0.28-0.33 l CH_4,/ g TCOD, respectively and these results are in line with others about AD of sewage sludge and glycerine [1,11,12].

3.4 Optimal conditions of the mesophilic AD process of sewage sludge and glycerin

AD process of sewage sludge and glycerin for stabilizing sludge and for obtaining renewable energy was carried out at six different SRTs (from 20 d to 5 d) or six OLRs (from 1.03 g to 4.05 g COD/l/d) (Figure 2). As, the GP, MP and SMP were more or less constant (except to 7 d and 5 d), the quality of the effluent and the adaptive capacity were the parameters selected to determine the optimum operating condition for AD process of glycerin and sewage sludge from Cadiz-San Fernando WWTP.

Considering the pH values, the low values of VFA, the high organic matter removal it could be said that AD process of sewage sludge and glycerin at SRT of 15 d and 12 d can be as effective as SRT of 20d. 9 d of SRT seems to be very low because it is close to the SRT in which VFA start increasing and organic matter removal start decreasing (SRT of 7 d). Therefore digesters could operate at 15-12 d instead of 20 d without jeopardising its safety, since at supplementations of 1% (v/v) glycerin is stable up to 7 d SRT. It means a reduction in the reactor cost (initial cost) of the AD up to 25-40%, compared to AD process of sewage sludge and glycerin at SRT of 20 d. In addition, the operational cost of anaerobic co-digestion will be reduce (lower time to heat and mix the waste).

In short AD process of sewage sludge and glycerin at SRT lower than 20 d is possible and preferable, due to it is more economical and environmental friendly. Decreases in the SRT not only allow maintaining the sludge stability and the biogas production, but it also implies an increase in the waste that could be treated and lower initial (lower volume of reactor) operational cost (lower volume/time to heat and mix) in a real process. It is an important fact, due to sludge management is a serious issue since up to one-half of the costs of operating WWTPs is associated with sludge treatment and disposal [2,7,22] and it has been estimated that 4 billion gallons of crude glycerol will be produced each year by the biodiesel market reached [9,23]. Therefore every process to allow treat more waste in a shorter time or produce a reduction in the cost (initial cost or operating cost) of the WWTP have to be highlighted.

Conclusion

The effectiveness of the glycerin supplementation during the AD of sewage sludge at different SRT was assessed in this study. The following conclusions have been obtained:

- Co-digestion of glycerin and sludge is totally stable at SRT between 20 and 9 d. No significant differences in methane production and organic matter removal were detected under these conditions.
- AD process of sewage sludge and glycerin at SRT lower than 20 d is possible and preferable, due to it is more economical and environmental friendly. In a real WWTP operate at 15-12 d instead of 20 d could suppose a reduction in the WWTP cost of the AD of biowaste.

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Figure captions

Figure 1. The laboratory-scale reactor used for this study**.**

Figure 2. Experimental conditions applied during the assay (SRTs and OLRs).

Figure 3. pH evolution along the process at different SRTs (● 20 d, ◆ 15 d, ▼ 12 d, ▽ 9 d, □ 7 d, ✚ 5 d) .

Figure 4. VFA/alkalinity ratio (equiv. acetic acid/equiv. $CaCO_3$) at different SRTs tested.

Figure 5. Medium values of organic matter removal: VFA, TCOD and VS.

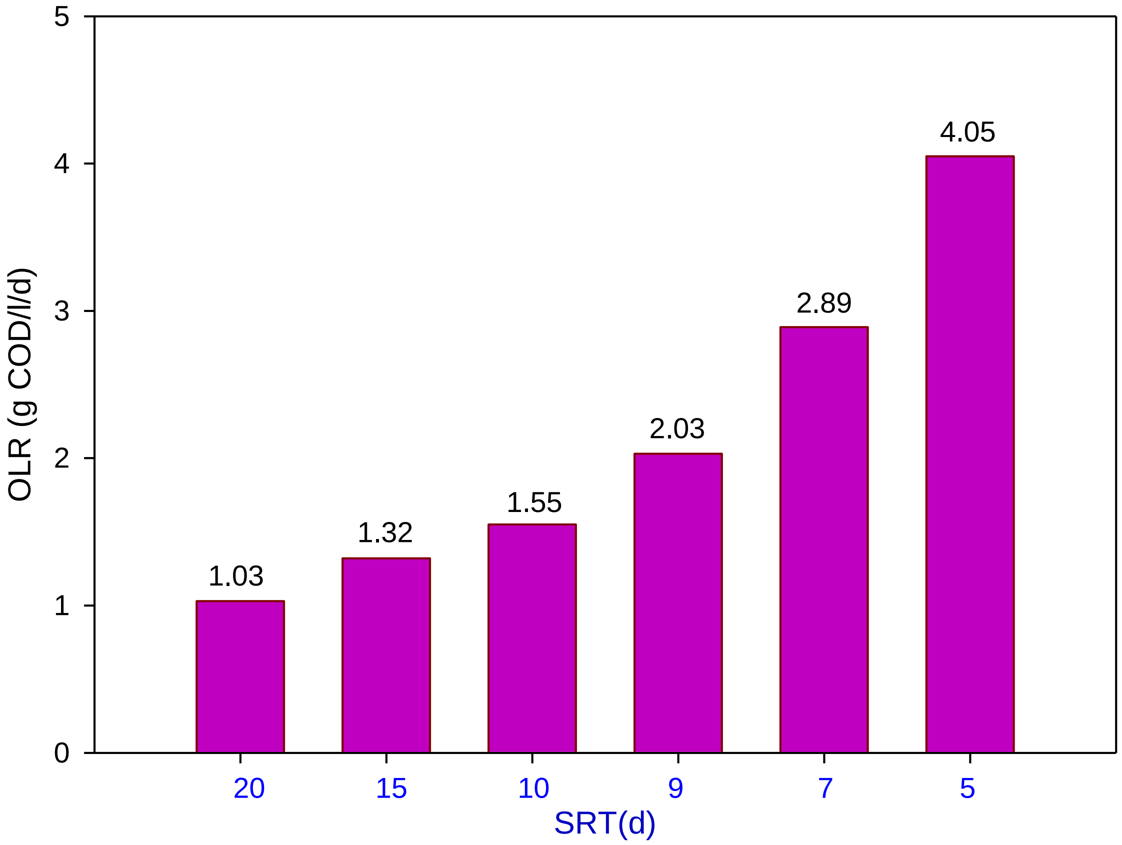
Figure 6. GP and MP at different SRTs tested.

Table 1. Main characteristics of sewage sludge.

Parameters	Sewage Sludge
pH	5.65±0.11
Conductivity (mS/cm)	9.88±1.25
TS (g/kg)	45.02±4.52
VS (g/kg)	34.59±5.05
TCOD (g O_2/l)	49.41±5.53
TOC (g/l)	15.83±2.36

Figure 1

Figure 2



392 **Figure 3**

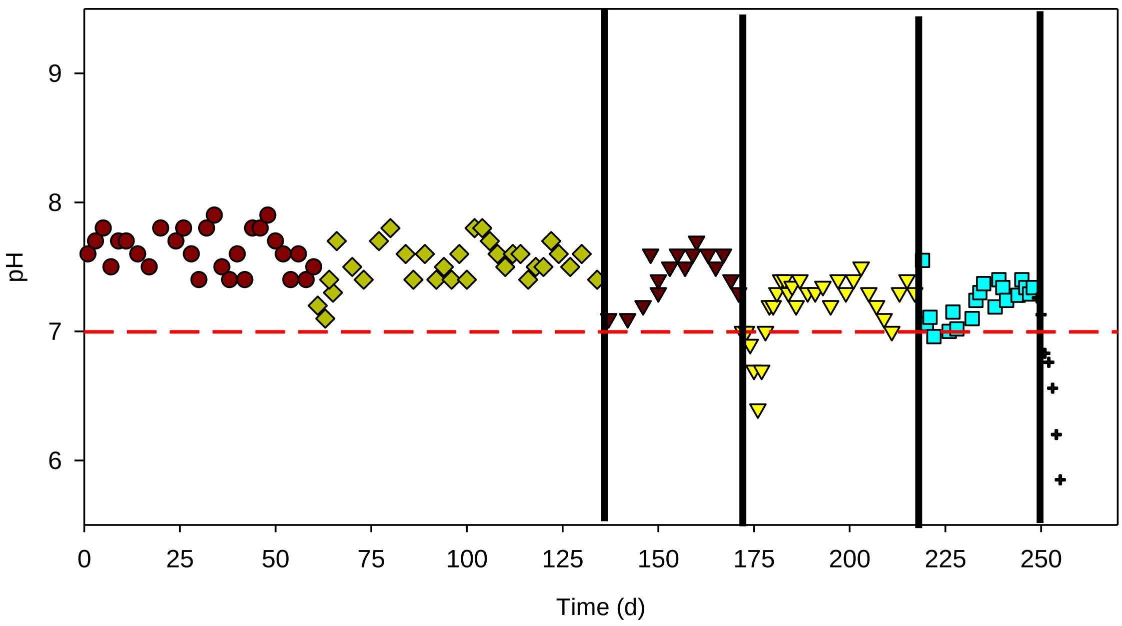


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Figure 4

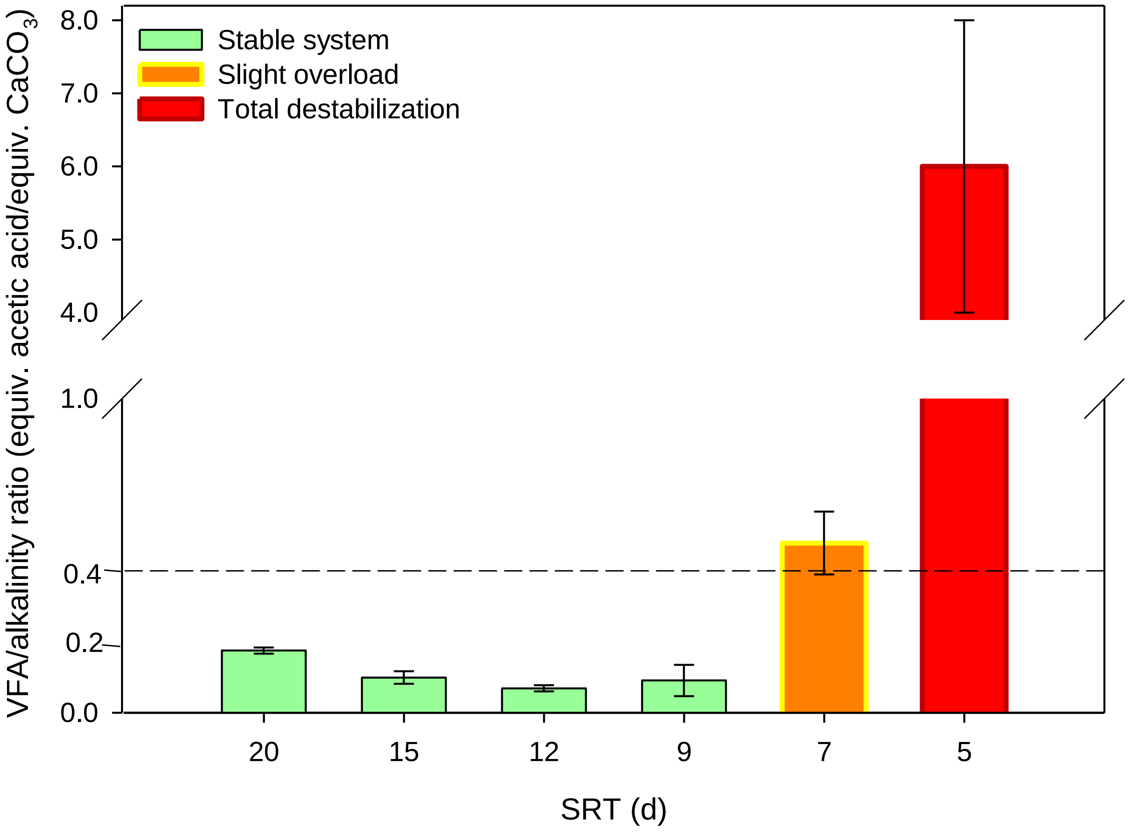

Stable system
Slight overload
Total destabilization
VFA/alkalinity ratio (equiv. acetic acid/equiv. $CaCO_3$)
8.0
7.0
6.0
5.0
4.0
1.0
0.4
0.2
0.0
20
15
12
9
7
5
SRT (d)

Figure 5

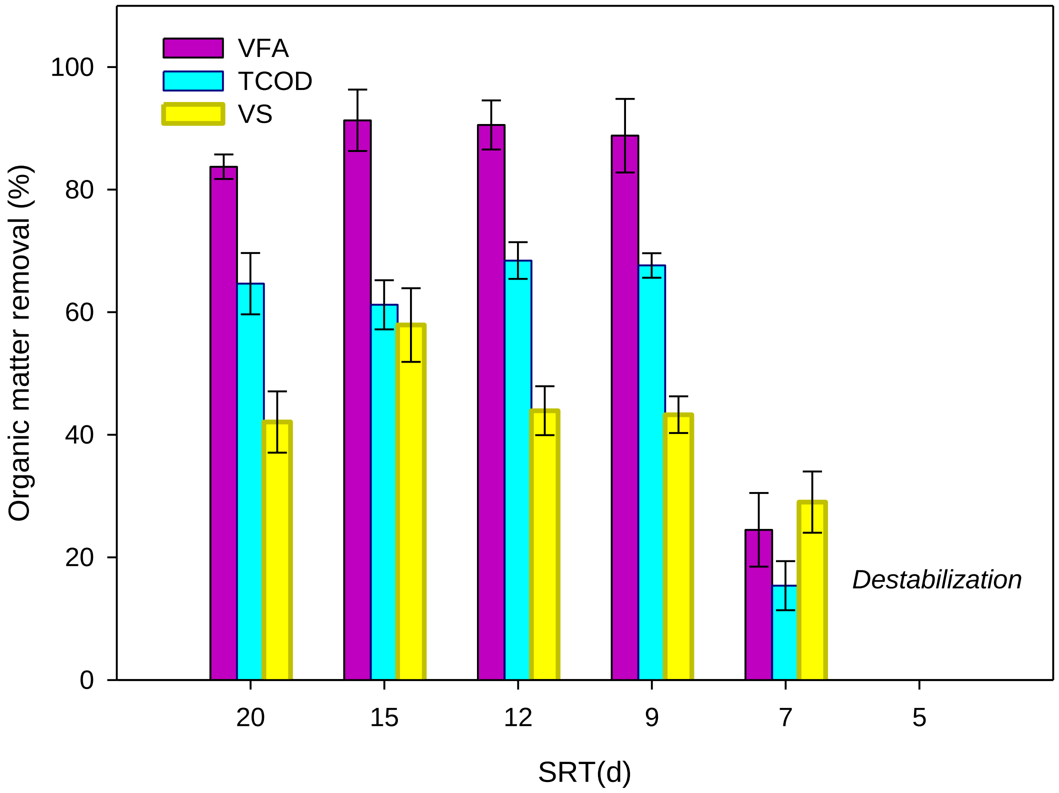
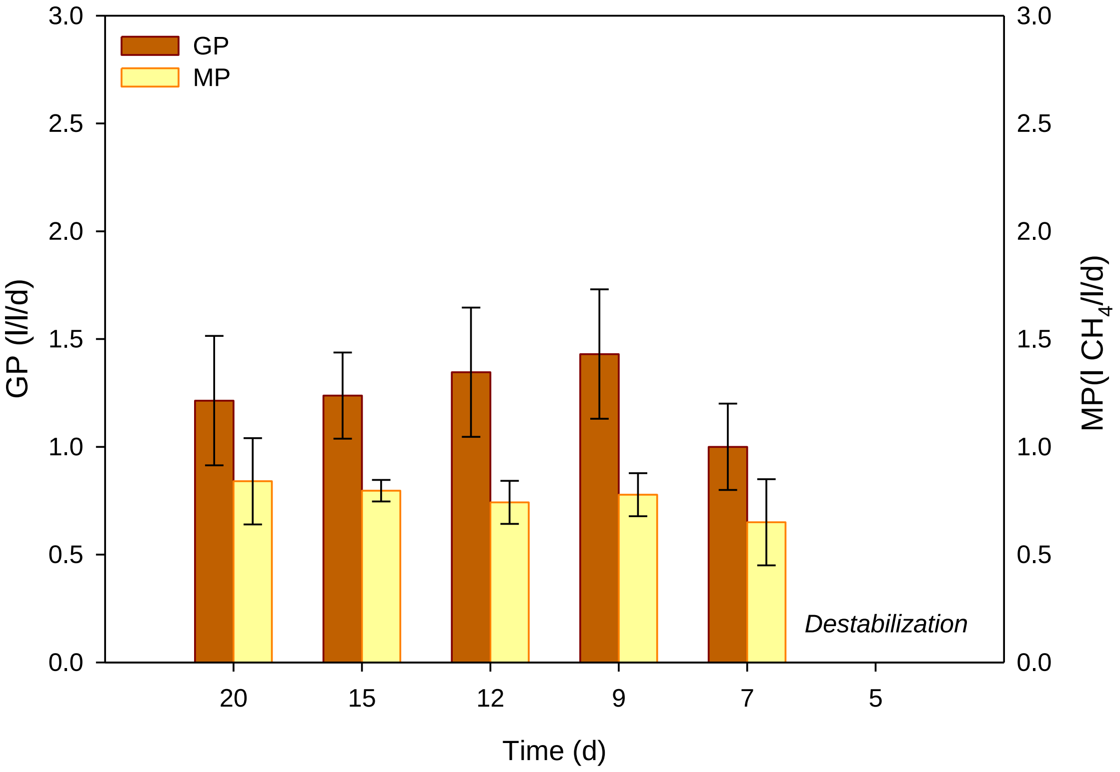

VFA
TCOD
VS
Organic matter removal (%)
100
80
60
40
20
0
Destabilization
20
15
12
9
7
5
SRT(d)

Figure 6



GP
MP
GP (l/l/d)
MP(l CH_4/l/d)
3.0
2.5
2.0
1.5
1.0
0.5
0.0
20
15
12
9
7
5
Time (d)
Destabilization