Two-stage anaerobic digestion of fruit and vegetable wastes

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The objective of this work was to determine the maximum organic load that can be treated in a two-stage anaerobic system for the stabilization of fruit and vegetable waste (FVW). The hydrolysis-acidogenesis step was carried out in a batch-operated stirred tank reactor (STR) and the methanogenic step was carried out in an up flow anaerobic sludge blanket reactor (UASB). This separation favored the environmental conditions of the microbial groups present in each stage and had a better control over the global variables of the process. The easily biodegradable fraction imposed (as volatile solid, VS) the maximum organic loading rate (OLR) of the overall process (13 g VS/L·d), in addition to conditioning the maximum input load of volatile fatty acids (VFA) to the methanogenic reactor 3.5 g/L·d. These conditions allow to highlight the benefit of separating the stages. The productivity reached was 3.0 L_{CH4}/L·d with a chemical oxygen demand (COD) removal of 80 %.

Keywords: methanogenesis; acidogenesis; hydrolysis; fruit and vegetable waste; UASB.

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Introduction

The generation of fruit and vegetable waste (FVW) increases with the growth of the population and the inadequate disposition of these FVW generates great problems of environmental pollution. It is necessary to invest in infrastructure for the collection and disposal of waste. In Mexico, investments have been made in the construction of landfills. However, biogas is not recovered in these sites due to the increase in initial investment costs. This way of operating sanitary landfills causes the loss of energy and the emission of greenhouse gases. That is why we should look for more sustainable alternatives for waste management. The anaerobic digestion (AD) of FVW generates biogas (methane and carbon dioxide). The capture of these gases represents important benefits: on the one hand, the emission of greenhouse gases is reduced and on the other, the waste is reassess transforming them into energy (methane) to satisfy the population's constant energy demand. Anaerobic digestion is a process that involves multiple stages (hydrolysis, acidogenesis, acetogenesis, and methanogenesis) differentiated by the types of microorganisms that intervene in them. Each of the stage requires different environmental conditions and grows at different speeds, which makes the study of the multistage process complex. Therefore, an alternative to study may be the physical separation of these stages.

Different anaerobic digestion processes are used, for example, batch processes, continuous one-stage, and two-stage processes. The AD can be carried out in different bioreactors, for example, continuous stirred tank reactors (CSTR), tubular reactors, anaerobic sequential batch reactors (ASBR), up flow anaerobic sludge blanked reactors (UASB), and anaerobic filter reactors. The anaerobic digesters differ mainly in the way in which the microorganisms are retained and by the type of flow of the materials.

Several processes and bioreactors have been implemented to anaerobically digest fruit and vegetable waste. The continuous one-stage reactors have better productivities than the batch reactors. It can produce up to 5 times more methane than batch reactors going from 0.2 to 1.1 L_{CH4}/L·d; and treat OLR from 0.98 g VS/L·d (reactors in batch) to 2.8 g VS/L·d (continuous reactors) [1, 2]. The obtained results from Aslanzadeh *et al*. [3] indicated that the systems of one-stage continuous could reach organic loads of up to 3 g VS/L·d before being destabilized (decrease in the production of methane). On the other hand, in two-stage continuous systems it has been possible to treat loads of up to 6 g VS/L·d obtaining mean productivities of 2.3 L_{CH4}/L·d [1, 2]. However, the standard deviation between the two-stage systems is up to 2 L_{CH4}/L·d. This difference is linked to several factors such as the type of bioreactor used in the two-stage system, the activity of the inoculum in the methanogenic reactor, the use and permanence of the inoculum in the acidic reactor, the hydraulic retention time, and the retention time of solids used [4-9].

In general, hydrolysis is the rate limiting step if the substrate is in particulate form. However, the anaerobic degradation of cellulose-poor wastes like FVW is limited by methanogenesis rather than by the hydrolysis. These wastes are very rapidly acidified to volatile fatty acids (VFA) and tend to inhibit methanogenesis when the feedstock is not adequately buffered. In one-stage systems all these reactions take place simultaneously in a single reactor while in two- or multistage systems the reactions take place sequentially in at least two reactors. Despite this knowledge in none of the anaerobic digestion systems described above, it has separated the easily biodegradable fraction from the difficult one which apparently causes the hydraulic retention times (HRT) in the one and two stage systems greater than 10 days in order to achieve good methane productivities. Therefore, in this study a two-stage system for the anaerobic digestion of FVW is proposed with the first stage allows separating easily biodegradable fractions (VS with particle sizes less than 105 µm) from the difficult biodegradation fraction (VS with particle sizes greater than 105 µm) with the intention of managing high VS loads in the second stage (methanogenic) to reduce the time of treatment.

Materials and methods

Configuration of the two-stage anaerobic system

The two-stage anaerobic digestion system consisted of a 120 L anaerobic stirred tank reactor (STR) and a 24 L UASB reactor with an area of 400 cm^2 and a height of 60 cm (methanogenic reactor). The STR was fed with domestic wastewater and mixtures of fruit and vegetable waste. The system had screens that allowed the separation of solids with a particle size greater than 105 µm. The liquid digestate (particle size less than 105 µm) from the STR is adjusted to pH 7 by adding 0.5 g of $NaHCO_3$/g chemical oxygen demand (COD) and 5 N NaOH. Once neutralized the liquid digestate was fed to the methanogenic reactor (Figure 1).

To determine the treatment time in the STR, a waste:water ratio of 1:2 was established, and the residues were mixed at 100 rpm intermittently (1 h every 12 h). The operation was performed in batch. To evaluate the efficiency of the reactor, 10 mL samples were taken every 12 h and were passed through a 105 µm screen. The quantities of COD, VS, VFA, and pH were determined. The treatment time was established as the time at

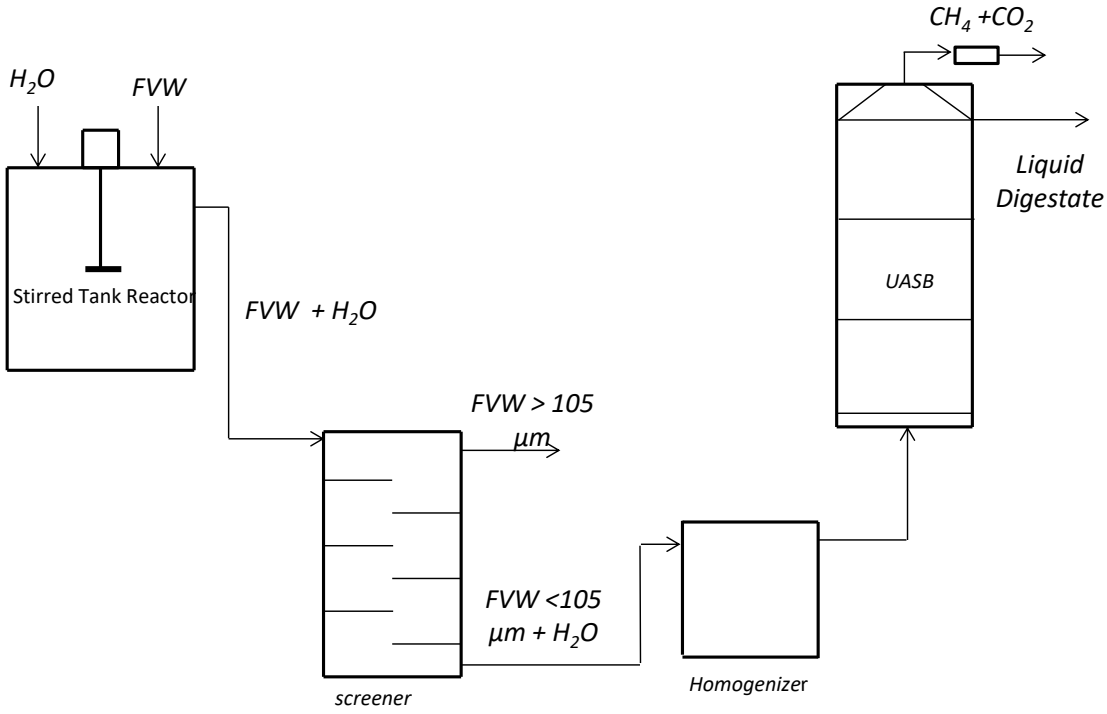

Figure 1. Two-stage anaerobic system.

which the maximum concentration of VFA and COD (water saturation by solids) was achieved.

The start-up of the methanogenic reactor was carried out using the procedure proposed by Van lier [10]. The sludge used as inoculum was a flocculent anaerobic sludge from a laboratory-level reactor fed with leachate blends of fruit and vegetable wastes composting. The initial organic load of operation was 0.37 g VS/L·d and this increased as the VS removal efficiency exceeds 80%. The maximum operating load was established based on maximum removal efficiency and maximum methane productivity. The organic load rate (OLR) is determined by using equation 1:

$$OLR = \frac{SQ}{V}24 \qquad \text{Equation 1}$$

where the OLR is the organic load rate in g VS/L·d; S is the volatile solids in g/L; Q is the feed flow in L/h; V is the reactor volume in L; and 24 is a conversion factor.

Characterization of fruit and vegetable residue mixtures in liquid and solid digestates

Fruit and vegetable residues were collected in an establishment where fruit cocktails and salads are made in the municipality of Ecatepec, metropolitan area of Mexico City, Mexico. After reaching the laboratory, they were classified according to the types, and then, were homogenized to 10 cm size of particle and weighed to determine their composition. FVW mixtures were characterized according to humidity, COD:N:P ratio, COD/VS ratio, and VS/TS ratio. The total solid (TS) and the volatile solid (VS) contents of the substrates were determined by drying the samples in a constant weight at 105°C and 550°C, respectively. The Kjeldahl nitrogen contents were determined by homogenizing 0.1 g of mixed dry FVW to a particle with the size smaller than 500 µm and analyzing it according to the NMX-AA-026-SCFI-2001 protocol (Water analysis - Determinationof total kjeldahl nitrogen in natural water, wastewaters and wastewaters treated - test method) [11]. The phosphorus concentration

Table 1. Composition of fruit and vegetable waste mixtures.

Mixture 1		Mixture 2		Mixture 3	
Waste	**Fraction**	**Waste**	**Fraction**	**Waste**	**Fraction**
Avocado	0.002	Orange	0.010	Strawberry	0.140
Peanut	0.000	Epazote	0.005	Peach	0.036
Pumpkin	0.006	Guava	0.012	Guava	0.013
Cane	0.008	Mango	0.132	Orange	0.053
Onion	0.003	Tangerine	0.023	Grape	0.031
Squash	0.003	Papaya	0.053	Lemon	0.048
Coriander	0.007	Avocado	0.009	Avocado	0.001
Strawberry	0.016	Bean	0.012	Cantaloupe	0.019
Pomegranate	0.001	Soursop	0.041	Pineapple	0.002
Soursop	0.045	Lemon	0.020	Mamey	0.005
Guava	0.025	Mamey	0.003	Guava	0.003
Jicama	0.081	Banana	0.025	Papaya	0.028
Tomato	0.003	Cantaloupe	0.091	Mango	0.259
Lemon	0.015	Apple	0.015	Watermelon	0.362
Tangerine	0.089	Watermelon	0.289		
Mango	0.113	Pineapple	0.195		
Cantaloupe	0.002	Strawberry	0.056		
Orange	0.093	Cane	0.008		
Pope	0.004				
Pear	0.047				
Pineapple	0.183				
Banana	0.074				
Watermelon	0.145				
Tomatillo	0.019				
Grape	0.001				
Carrot	0.016				
Mixture 4		**Mixture 5**		**Mixture 6**	
Waste	**Fraction**	**Waste**	**Fraction**	**Waste**	**Fraction**
Cucumber	0.125	Watermelon	0.631	Jicama	0.071
Beetroot	0.103	Tuna	0.216	Beetroot	0.074
Jicama	0.098	Orange	0.143	Carrot	0.025
Orange	0.080	Carrot	0.002	Tuna	0.067
Carrot	0.022	Mango	0.007	Lemon	0.055
Cantaloupe	0.168			Papaya	0.160
Watermelon	0.227			Radish	0.004
Papaya	0.085			Cucumber	0.145
Mango	0.093			Apple	0.124
				Orange	0.056
				Watermelon	0.219

was measured according to the NMX-AA-029-SCFI-2001 protocol (Waters analysis - Determination of total phosphorus in natural, wastewaters and wastewaters treated- test method; Vanadomolydophosphoric acid method) [12].

Table 2. Physicochemical characteristics of the samples.

	Mixture 1	Mixture 2	Mixture 3	Mixture 4	Mixture 5	Mixture 6
Humidity %	89	89	73	89	89	91
COD:N:P	350:6:7	350:2:6	350:4:12	350:6:7	350:5:13	350:4:9
g COD/g VS	1.9	2.1	1.6	1.8	1.9	1.4
g VS/g TS	0.98	0.93	0.97	0.97	0.98	0.91

Analytical method

Biogas volume was quantified by a column displacement of saturated NaCl (pH 3.5) in a 3.5 cm diameter glass tube. The biogas composition was determined by taking a sample from the biogas reactor collector and injecting 0.1 mL into a gas chromatograph (GOW-MAC Instrument Co, U.S.A.) with an SP-4290 integrator and a stainless-steel column packed with carbosphere. The operating temperature conditions of the gas chromatograph were: injector 170°C, column 140°C, detector 190°C. Helium was used as carrier gas with a flow of 30 mL/min. Filament current was 120 mA.

The volatile fatty acid (VFA) was determined in a gas HP 5890 chromatograph (Agilent, Santa Clara, CA, USA) with flame ionization detector and an AT 1000 capillary column using nitrogen as gas carrier at 5 mL/min. The temperature of the detector and injector were 200°C with a ramp of 25°C/min.

Results and discussion

Characteristics of fruit and vegetable waste mixtures

The FVW mixtures used are shown in Tables 1. The composition of the samples was used to determine the variability of the physicochemical characteristics of the different mixtures of fruits and vegetables. Table 2 shows the results of the physicochemical characterization of the used mixtures. The average humidity rate was 89% except for mixture 3, in which the proportion of fibrous material (> 25%, mango bone) caused a decrease in humidity to 70%. It was also observed that the C:N:P ratio is a function of the composition of the mixture. Therefore, it would be necessary to control this relationship to improve the performance of the anaerobic digesters. Four of the mixtures met the 350:5:1 ratio [13] necessary for accomplishing an efficient anaerobic digestion. This difference implied balancing in the C:N:P ratio for mixtures 3 and 6 to avoid affecting the AD of the mixtures of fruit and vegetable wastes.

Solubilization and hydrolysis of fruit and vegetable wastes.

The reaction time of two 180 L STR was set as 3 days, at which time the maximum concentration of COD was reached (particle size less than 105 µm). Two reactors of the same capacity were used to be able to operate continuously the UASB reactor, which operated with a feed of 57.6 L/d (HRT 10 h, V = 24 L). The COD for the first six batches of the STR were shown in Table 3. The average concentration of VFA in the reactor, after three days of reaction, was 3,000 ± 500 mg/L.

Table 3. COD in the STR.

Mixture	COD (g/L)
1	39 ± 3
2	42 ± 5
3	40 ± 4
4	41 ± 3
5	59 ± 6
6	30 ± 2

After loading the STR with FVW, the pH of the wastewater dropped to 3.7 in about 20 minutes. The mixing of the FVW and the residual water immediately caused the organic matter to dissolve with the concentration of 3 g COD/L.

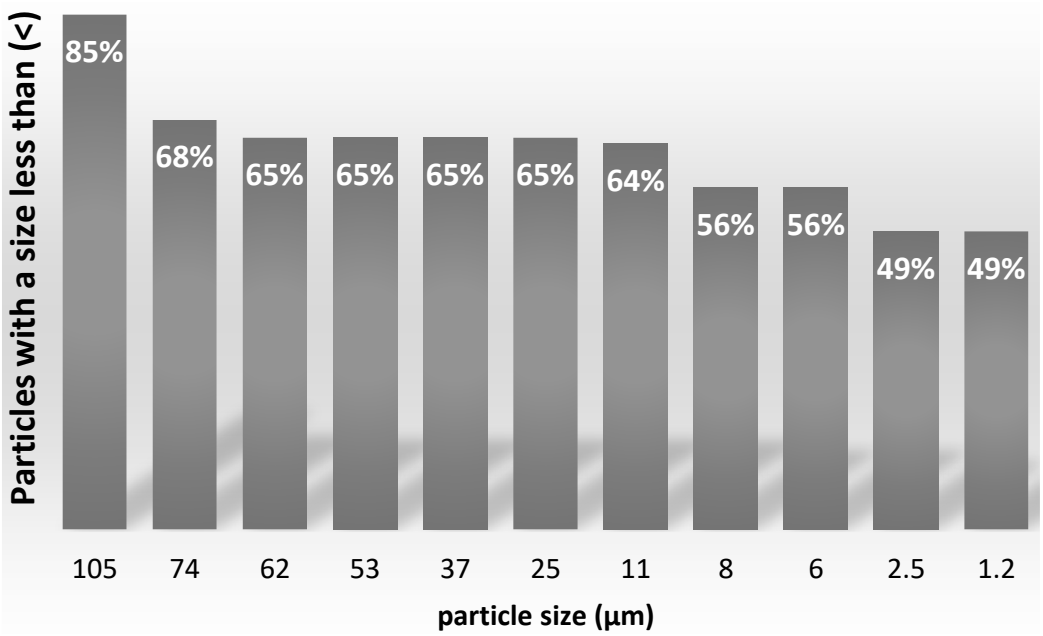

Figure 2. Particle size distribution at the effluent of the STR.

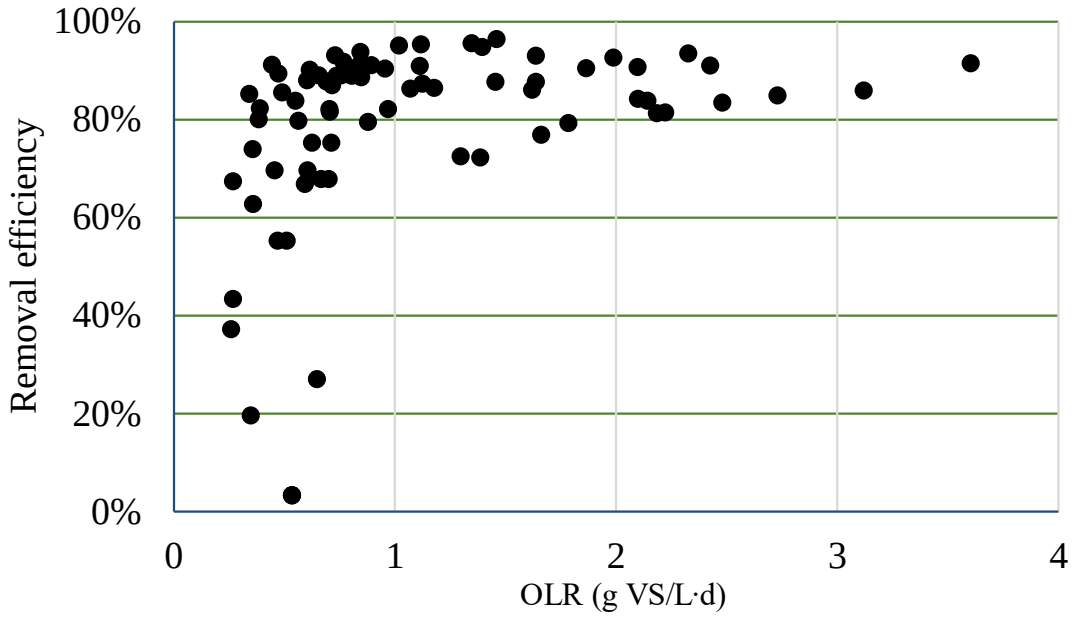

Figure 3. Effect of the organic loading rate on the efficiency of VS removal in the methanogenic reactor.

After three days of operation, the concentration increased with the average values of 42 ± 11 g/L (Table 3). The percentage of solids which size were greater than 1.2 µm at the end of each batch was 51% in average. The particle size distribution was shown in Figure 2.

Increase of the organic load in the UASB methanogenic reactor

The effects of increasing the organic load (OLR) on the performance of the methanogenic reactor were evaluated. The organic load fed was increased from 1 to 3.5 g VS/L·d in the first

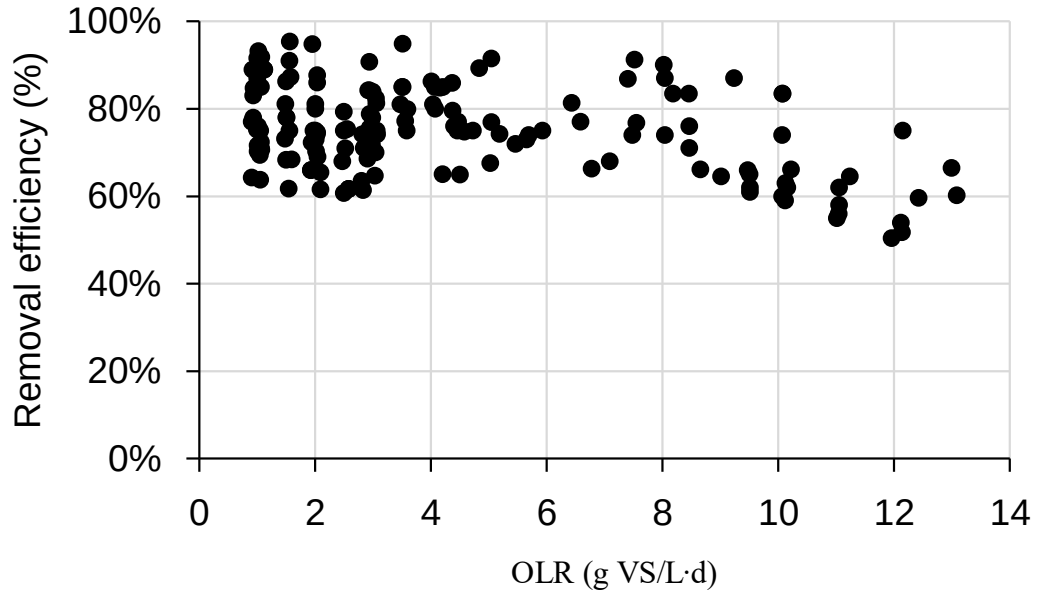

Figure 4. Effect of high organic loading rate on the efficiency of VS removal in the methanogenic reactor.

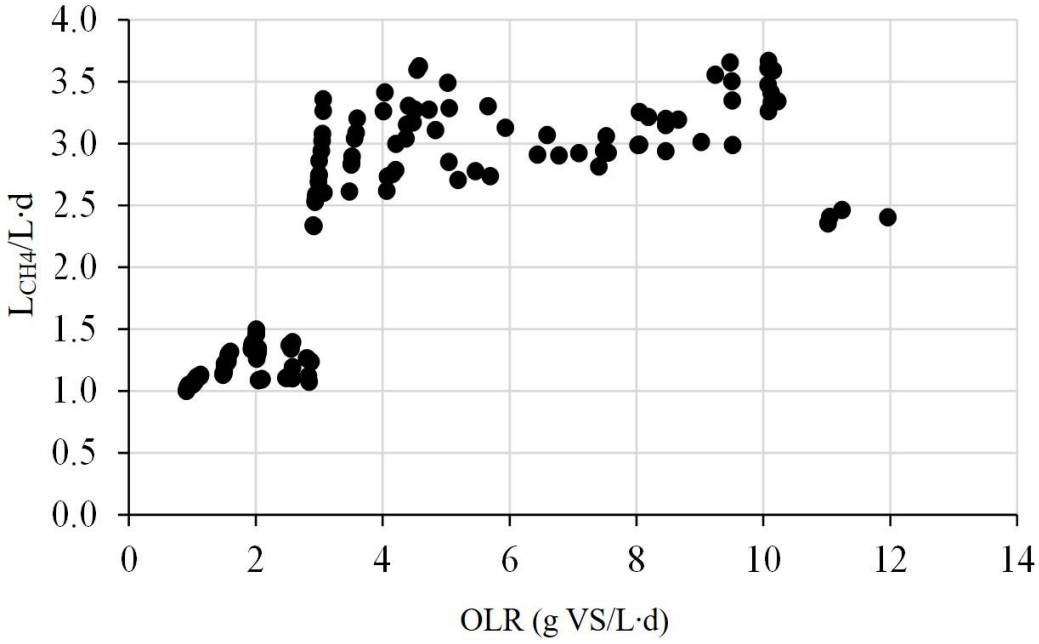

Figure 5. Effect of high organic loading rate on the methane productivity.

period to maintain a VS removal efficiency greater than 80% (Figure 3). In the second period, the organic loading rate was increased to 13 g VS/L·d, which caused a reduction in the VS removal efficiency (below 60%) (Figure 4).

Methane production for solids with particle sizes less than 105 µm from the STR increased with the increase of organic load (Figure 5) and reached the maximum value of 2.1 L_{CH4}/L·d, which was previously reported in the interval for the similar systems (1.5 L_{CH4}/L·d) [14]. However,

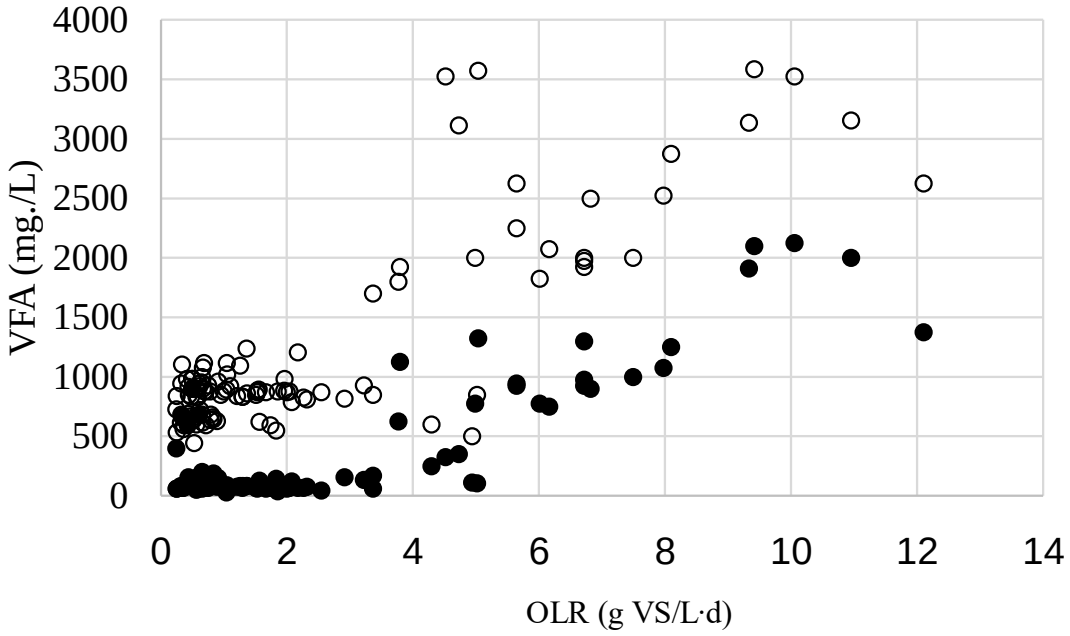

Figure 6. Concentration of VFA at the influent (o) and effluent (●) of the methanogenic reactor at different organic loading rate.

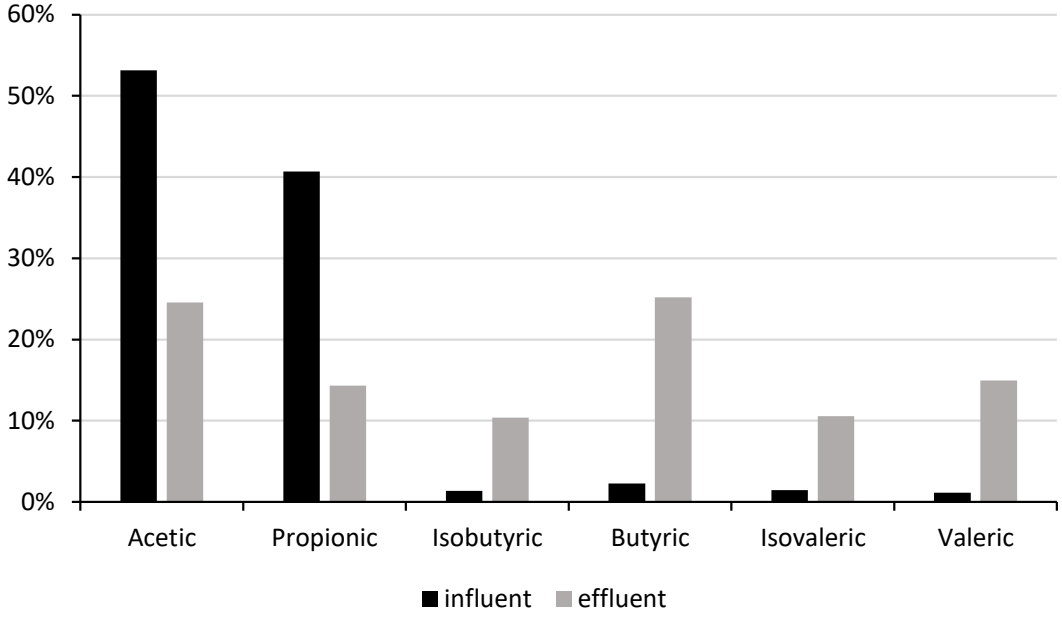

Figure 7. Percentage of VFA at the influent and effluent of the methanogenic reactor at high OLR (greater than 4 g VS/L·d)

the production of methane decreased 62% with respect to the theoretical value by increasing the organic load above 3.5 g VS/L·d (HRT 10 h), which behavior has been reported for loads above 2 g VS/L·d [2, 4]. The maximum values of methane production reported by previous studies before starting the decrease in productivity were 1.83 and 0.8 L_{CH4}/L·d, respectively. In this study the maximum values were 3.0 L_{CH4}/L·d, which could be due to the high concentration of VFA fed the methanogenic reactor.

The concentration of total VFA at the influent and effluent of the UASB reactor operating at low and high organic loads is presented in Figure 6. The concentrations of total VFA in the feed were 839 ± 190 mg/L in average for low loads (1-4 g VS/L·d) and 2,485 ± 748 mg/L for high loads (5-12 g VS/L·d). The VFA percentages including acetic, propionic, isobutyric, butyric, isovaleric, valeric in the influent were 53%, 40.7%, 1.3%, 2.3%, 1.4%, and 1.1%, respectively. The removal of VFA in the methanogenic reactor was 83 ± 20% at low loads and 56 ± 14% at high loads. The acids' percentages including acetic, propionic, isobutyric, butyric, isovaleric, valeric at the effluent of the reactor were 25%, 14.3%, 10.4%, 25.2%, 10.6%, and 15.0%, respectively (Figure 7). The organic acid with the highest concentration in both low and high organic loads was acetic acid, which may explain the high methane productivities. The fraction of butyric acid increased at high organic loads. This accumulation coincides with that reported by Shen, *et al*. [7] who found that butyric acid accumulated when the AD was carried out at organic loads greater than 3 g VS/L·d.

Conclusion

The system showed good stability mainly due to the VS concentration with particle size less than 1.2 µm delivered by the STR reactor.

The methanogenic reactor operated at organic loads between 5 and 13 g VS/L·d. Under these conditions the methane productivity was between 2 and 3.5 L_{CH4}/L·d.

The condition that can influence the increase of productivity is the high concentration of VFA in the effluent at high organic loads. The adequate acidification (3,000 mg/L of VFA) of the residues in the acidic reactor, the low input of suspended solids to the methanogenic reactor, as well as the neutralization of the feed prior to the methanogenic stage contributed to the adequate performance of the system.

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