TELEMAC IST - 2000 - 28156
TELEMonitoring and Advanced teleControl of high yield wastewater treatment plants

Experimental data sets at laboratory (D1.3a)

Deliverable Type: Database
Number: D.1.3a
Contractual Date of Delivery: 31 August 2002
Actual Date of Delivery: 23th August 2002
Task: WP1
Name of responsible: J.M. Lema
E. Roca

Author : USC
Colexio de San Xerome
Praza do Obradoiro, s/n
15782, Santiago de Compostela
Spain
Email: ***jmlema@usc.es***

Abstract
This report summarizes the work done by USC (WP1) in the last twelve months of the project, regarding the study at lab scale. The experiments at lab scale were defined and developed in coordination with industrial partners (Sauza, Agralco and Domecq), with the aim of taking into account the characteristics of the industrial wastewater treatment processes. Thus industrial wastewater and a lab scale CSTR were used. UASB was also used due to its wide application in the industrial wastewater treatment. The wastewater employed comes from an alcoholic distillery plant, which has a higher protein content than those coming from a wine distillery, as can be seen in its composition.
In this report the differences between the proposed protocol and the finally applied protocol for CSTR and UASB reactors are commented. Finally, the main experimental data are presented including two organic overloads of the CSTR and data from the start-up of the UASB.
The database with the experimental data set obtained at laboratory scale is published in the BSCW web page.

Keyword list: Experimental data sets, lab scale, start up, overload, CSTR, UASB.

Index

Experimental set-up

A CSTR of 2 liters of useful volume is employed. It is equipped with pH and temperature probes, a stirrer to provide 200 r.p.m. and a condensation system in the gas outlet. A flow meter measures the biogas production every 15 minutes. The temperature is fixed by a thermostatized jacket at 37 ºC. The reactor is fed by charges of fresh wastewater in a semicontinuous mode.’’
A picture of the overall experimental set-up is shown in Figure 1. The inoculum of the reactor comes from a pilot-scale plant treating dextrose with a specific methanogenic activity of 0.27 kg COD/m^3·d, and its initial concentration in the reactor was 6 g VSS/l. The biogas composition is measured via thermal conductivity gas chromatography (HP 5890 SerieII), with helium as carrier gas.

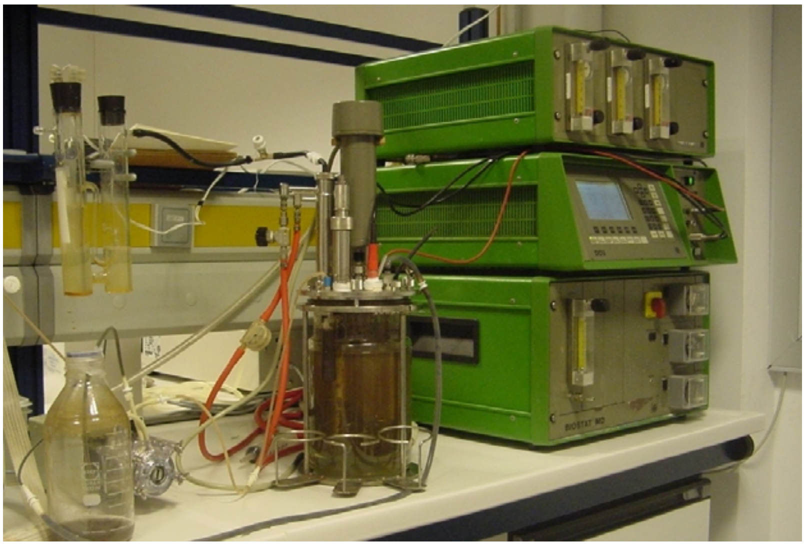

Figure 1. CSTR reactor

An UASB reactor of 4.8 l of useful volume was inoculated with the same biomass as the CSTR, but in this case with a concentration of 15 g VSS/l. A picture of the experimental set-up is also presented in Figure 2. It is equipped with pH, temperature and gas flow sensors and the biogas composition is measured on-line by a mass spectrometer. The reactor is fed continuously by a precision pump, and the recycling flow is adjusted to obtain 0.5 m/h of upflow velocity. Data from this process is acquired and stored on-line every 15 minutes by a program developed at the University of Santiago de Compostela (Ruiz *et al.*).

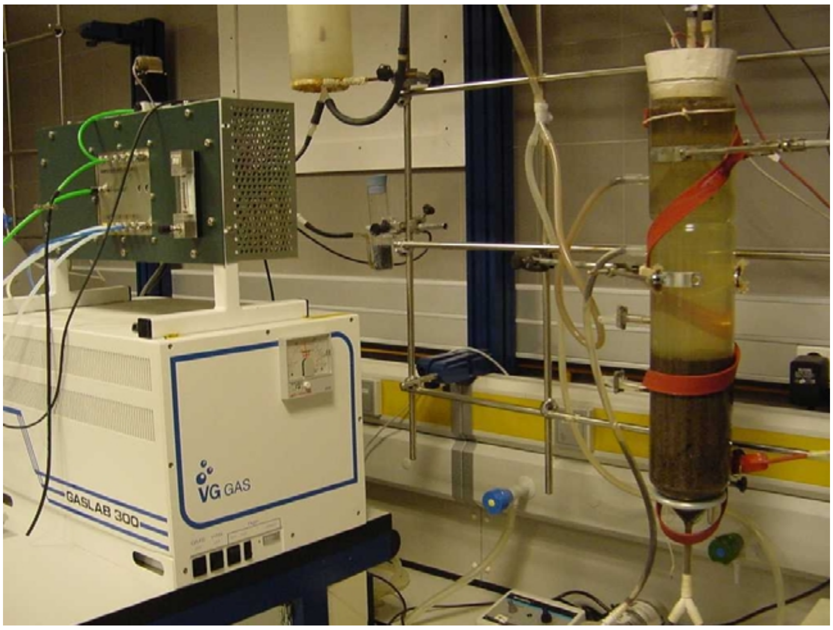

Figure 2. Experimental set-up of the UASB reactor.

Wastewater composition

The wastewater used for lab experiments comes from a local alcoholic distillery plant called "Aguardientes de Galicia, S.L.". The effluents from alcoholic distillery presents a high content in TKN, reaching in some cases values of 6000 ppm (Fernández *et al.*, 2001), in comparison to wine distillery wastewater which does not contain any protein. The main characteristics of wastewater employed by WP1 at lab scale are presented in Table 1.

Total COD (g/l)	75
Soluble COD (g/l)	71
TOC (g/l)	29.2
COD/TOC	2.6
AcH (g/l)	6.39
PrH (g/l)	0.07
nBuH (g/l)	0.47
TSS (g/l)	1.7
VSS (g/l)	1.63
Protein (g/l)	4.0
NH_4^+-N (ppm)	126
NO_x^- (ppm)	0
SO_4^{2-} (ppm)	709
PO_4^{3-} (ppm)	1142
Sugars (g/l)	6.4

Table 1. **Wastewater composition.**

According to this, the wastewater employed for carry out the experiments in the USC had a quite high concentration of protein.

Experimental protocol and results

The experimental data sets obtained in CSTR and UASB reactors at lab scale up to the date are reported. The start up phase was finished in both reactors. Three organic overload were carried out in the CSTR reactor. One of them was an accidental overload which however gave some interesting results which are also reported. The UASB is now working at normal conditions and the first organic overload will be carried out in the near future.

UASB

The start-up of this reactor was lower than the expected, because of the low quality of the sludge and the low biodegradability of wastewater, and several problems of the gas flow and gas composition sensors. Table 2 shows the main difference between the proposed and the applied protocol.

	OLR ($kgCOD/m^3 \cdot d$)	
t (days)	**Proposed**	**Applied**
0	1	1
6	1	1
12	2	1.8
18	3.5	1.85
25	5.5	2.5
29	11	3
37	5.5	3.45
41	11	3.6
48	5.5	3.68

Table 2. Comparison between the OLR values of the proposed and the applied protocol

The evolution of the Organic Loading Rate (OLR) of the influent, effluent and gas during the 48 days of experiment is presented in Figure 3. OLRin and OLRout are the organic loading rate corresponding to the wastewater fed to the reactor and to the outlet flow, respectively. OLRin and OLR out can be calculated from the follwing equations:

$OLRin = CODin \cdot D$

$OLRout = CODout \cdot D$

where D is the dilution rate.

OLRgas is the equivalent organic loading rate removal which leaves the reactor in the gas phase (principally methane), it can be calculate as it is explained below:

Considering the combustion reaction for the methane:

$CH_4 + 3/2O_2 \rightarrow CO_2 + 2H_2O$

the yield factor for calculating methane COD, considering an ideal gas behaviour at a presure of 1 atm is:

Temperature	mLCH4/gCH4 dry	mLCH4/gCH4 wet
10	363	367
15	369	376
20	376	385
25	382	394
30	388	405
35	395	418
40	401	433
45	408	450
50	414	471

Then OLRgas is the following:

ORLgas= [Qgas·(%CH4/100)]/[f·Vr]

where:

Qgas: gas flowrate (mL/d)
%CH4: percentage of methane in gas phase (%)
f: yield factor for methane (mLCH4/gCH4)
Vr: reactor volume (L)

It can be seen in this figure that between days 18 and 34, the COD balance does not fit correctly in the system, because of a failure in the gas flow meter.

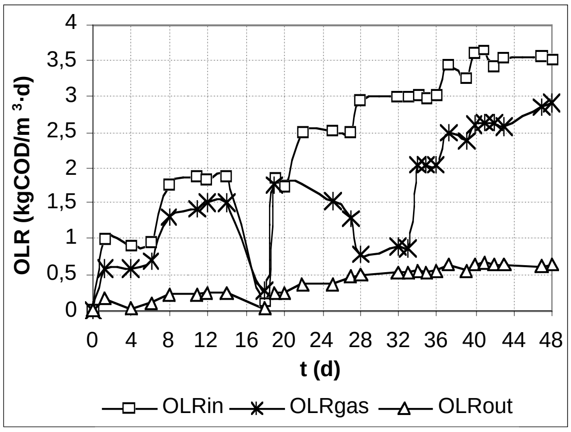

Figure 3. Organic Loading Rate (OLR) of influent, effluent and gas.

The alkalinity and gas composition are presented in Figures 4 and 5.

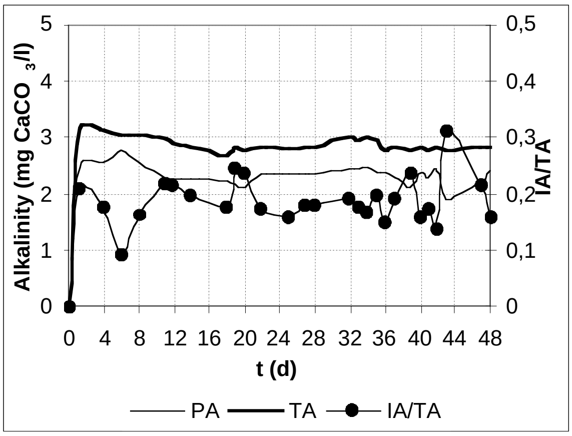

Figure 4. Partial alkalinity (PA), total alkalinity (TA) and alkalinity ratio (IA/TA)

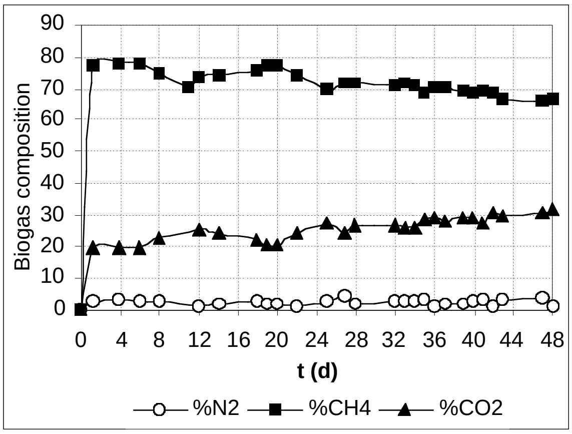

Figure 5. Biogas composition

These graphs, with the percentage of COD removal and the percentage of methanization (Figure 6), show the stability of the process during the experiment.

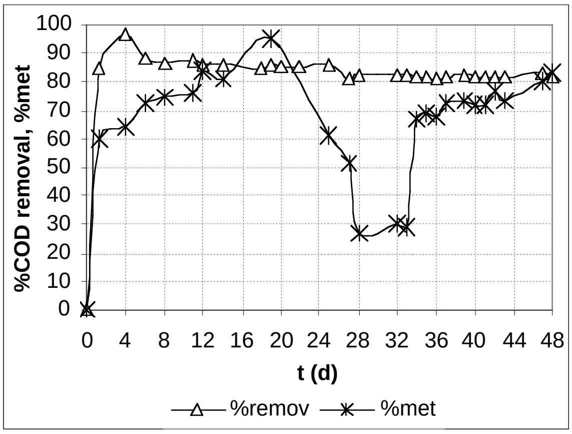

Figure 6. Percentage of COD removal and methanization

An example of on-line values for the gas flow, pH and biogas composition are presented in Figures 7 and 8.

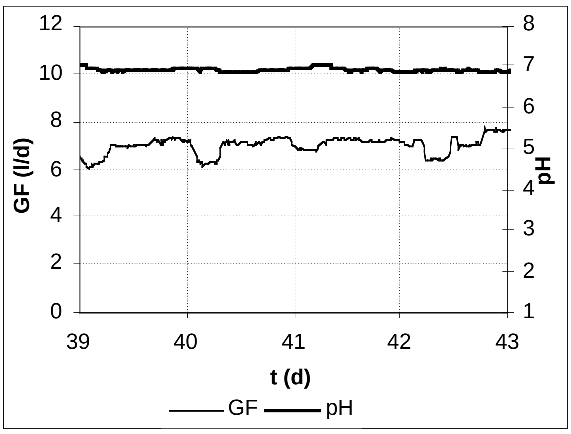

Figure 7. On-line values for pH and gas flow

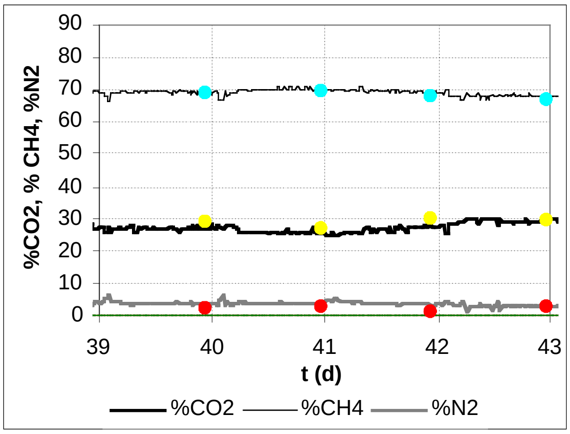

Figure 8. On-line values of the biogas composition (the points represent off-line values obtained by chromatography).

CSTR

The differences between the proposed and the applied protocols are shown in table 3.

	OLR (kgCOD/m³·d)		HRT (d)		COD (g/l)	
t (days)	**Proposed**	**Applied**	**Proposed**	**Applied**	**Proposed**	**Applied**
0	0.75	0.75	20	20	15	15
6	0.88	1.26	17	11.6	15	14.56
12	1.07	1.64	14	9.31	15	15.27
18	1.5	1.98	10	7.38	15	14.6
28	2	2.18	7.5	7.12	15	15.51
43	3	2.44	5	6.53	15	15.92
60	5	1.78	5	4.26	25	7.59
72	3	1.85	5	4.16	15	7.67
84	5	1.72	5	4.21	25	7.23
96	10	3.5	5	4.08	50	14.27
93	10	1.9	5	4.17	50	7.92
133	10	3.3	5	4.17	50	13.79
142	10	6.30	5	4,00	50	25.2

Table 3. Difference between the OLR, HRT and COD values of the proposed and the applied protocols.

An accidental organic overload on day 32, when the OLR was increased from 2.6 to 3.2 kg COD/m^3·d because of an increase in the COD content of the influent from 15.4 to 17.8 g/l (Figure 9), forced us to follow a new protocol as shown in table 3.

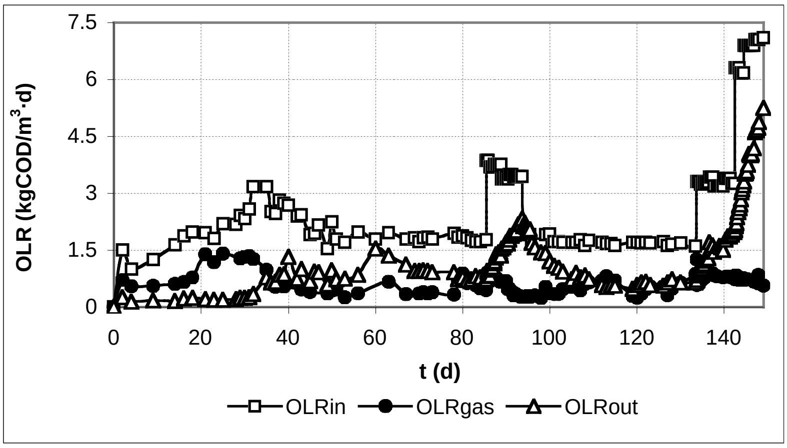

Figure 9. OLR of influent, effluent and gas

This overload was maintained during 4 days, but the alkalinity ratio (Figure 10) increased up to 0.5, and the OLR was decreased to the initial value of 2.5 kg COD/m3·d. Two pulses of bicarbonate (90ml of a solution of 53 g/L for the first one and of 50 ml with a concentration of 80g/L for the second one) were added to try to recover the process, however they only produce an instantaneous increase of pH as it can be seen in Figure 11. Afterwards a pH controller was installed from day 45 to day 73. The pH control was carried out by automatic addition of NaOH solution. 200mL of 1M NaOH solution were added from day 45 to day 53 and 140 mL of 3M NaOH solution were added from day 53 to day 73. The effect of the pH controller can also be seen in the biogas composition, since a decrease on the percentage of CO_2 was obtained (Figure 12).

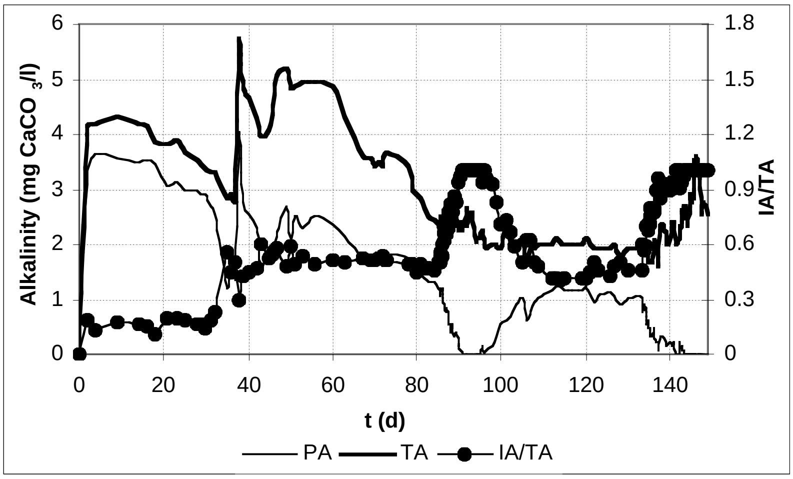

Figure 10. Partial alkalinity (PA), Total alkalinity (TA) and alkalinity ratio (IA/TA)

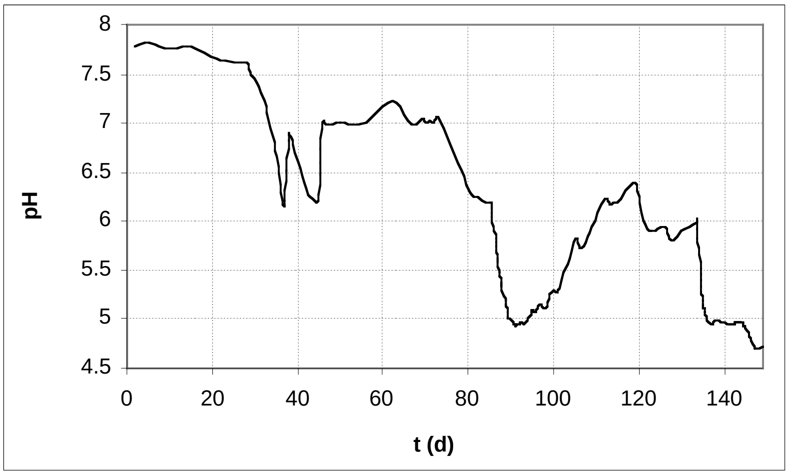

Figure 11. pH evolution during the experiment

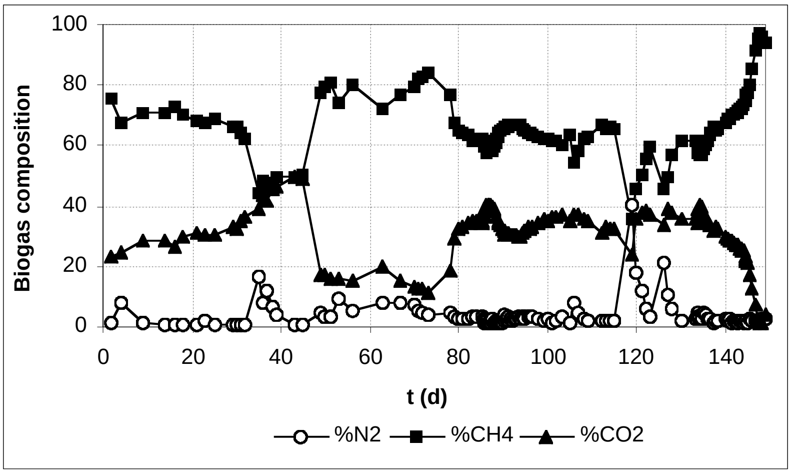

Figure 12. Biogas composition

Otherwise, on day 59 the feed flowrate was doubled and the concentration of COD reduced in a 50% in order to produce a dilution effect because of the high concentration of VFA (Figure 13) present in the reactor due to its bad performance since the organic overload on day 32.

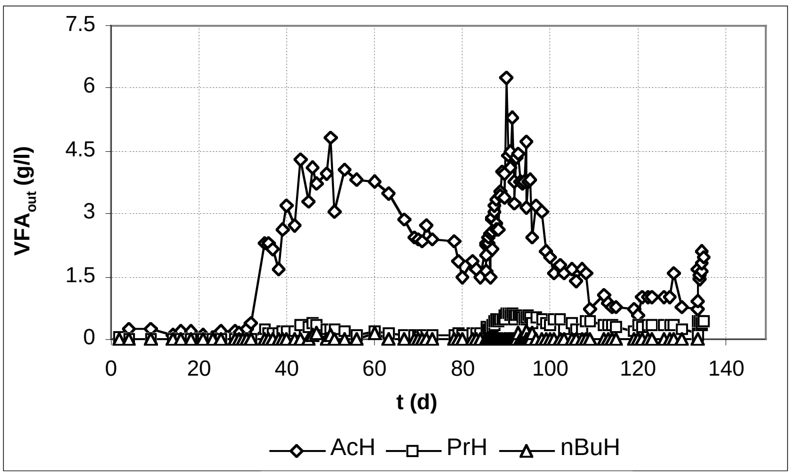

Figure 13. VFA concentration in the reactor

Between days 73 and 78, the reactor was stopped due to a failure in the stirring and temperature control system which stopped the biogas production. When restarting the stirring, the release of the biogas trapped in the liquid (Figure 14) generated an increase in the gas flow.

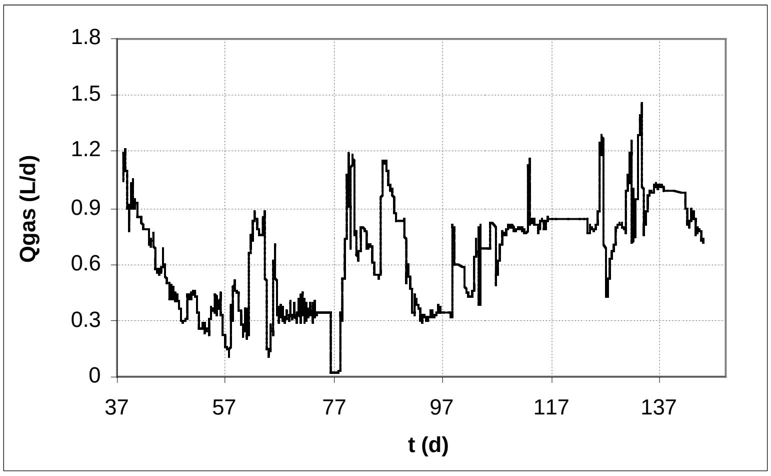

Figure 14. On-line values for biogas production.

On day 85 the first organic overload has been made, increasing the OLR from 1.8 to 3.9 kg COD/m^3·d. This state has been maintained until day 93, when the normal operation conditions where reestablished.

On day 133 the second organic overload was started. It was made in two steps, by a first increase in the OLR from 1.6 to 3.3 kg COD/m^3·d, followed by a second increase up to 6.3 kg COD/m^3·d on day 142.

The effects of the overloads are also shown in figures 15, 16, 17 and 18, where it can be pointed out the drastically decrease in COD removal, the annulment of the partial alkalinity and the trend of the alkalinity ratio to reach the value of one.

.

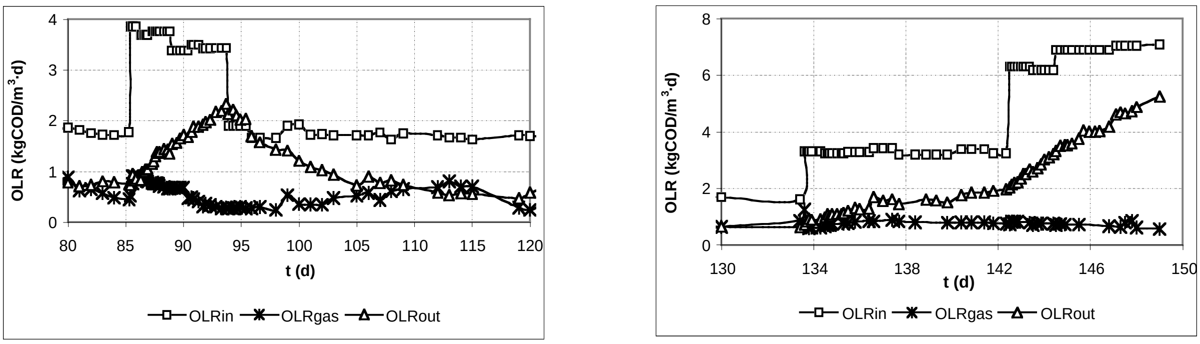

Figure15. OLR of influent, effluent and gas during the organic overloads

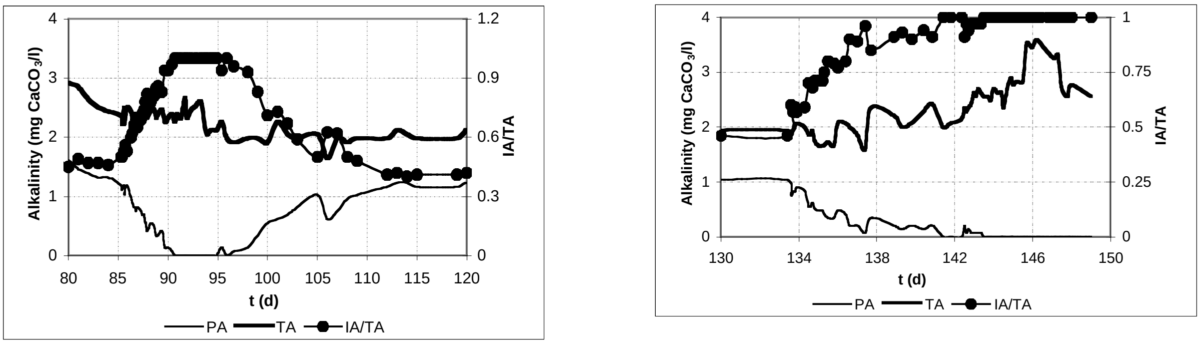

Figure 16. Partial alkalinity (PA), total alkalinity (TA) and alkalinity ratio (IA/TA) during the organic overloads.

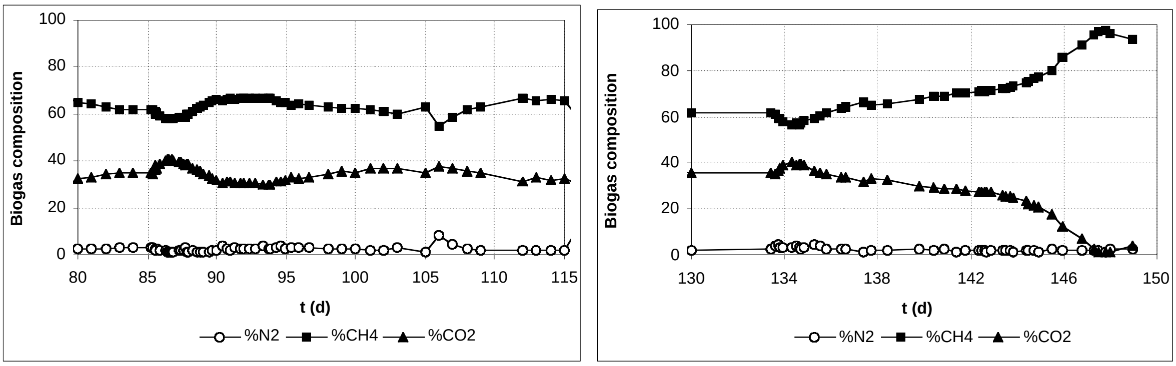

Figure 17. Biogas composition during the organic overloads

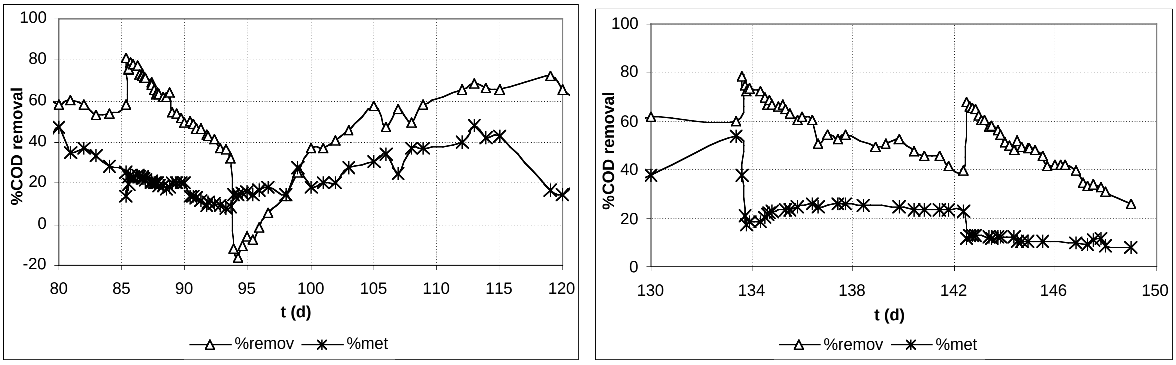

Figure 18. Percentage of COD removal and percentage of methanization

Conclusions

The experience acquired in both UASB and CSTR reveal that a slow start-up is required for avoid the destabilization of the process, since a sudden increase in the load applied may cause the acidification of the system and the lose of the steady state necessary to perform the overload experiments.

References

Fernández, N., Fdz-Polanco, F., Montalvo S. J., Toledano D. "Use of activated carbon and natural zeolite as support materials, in and anaerobic fluidized bed reactor, for vinasse treatment". *Wat. Sci. Technol* (2001), 44(4, Anaerobic Digestion), 1-6.

Ruiz G., Rodriguez J., Baeza J., Roca E. and Lema JM. (2001). Advanced monitoring and supervision of an anaerobic pilot plant. In: Proceedings of the Anaerobic Digestion World Congress (AD9), A.F. van Velsen and W. Verstraete (eds), vol 2, Technologisch Instituut vzw, Antwerpen, pp. 207-210.