

## Sludge deterioration in a full scale UASB reactor after a pH drop working under low loading conditions

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### ABSTRACT

A full scale UASB reactor treating the effluent of a malting plant was operated during nearly two years. During 37 weeks of operation the reactor worked with a COD removal efficiency of 80% and a biogas production of nearly 300 m<sup>3</sup>/d with a methane content of 77%. After the start up and during these months of operation the volumetric organic load was 4 kgCOD/m<sup>3</sup>.d and the specific organic load was between 0.2–0.4 kgCOD/kgVSS.d. The sludge SMA in this period was around 0.25 kgCOD/kg VSS.d. On week 37 as a result of a problem at the industrial process the pH in the reactor dropped to a value of 4.8. After pH recovering, the reactor worked with fluctuating COD values in the exit and showed a downward trend in the COD removal efficiency. On week 81 the presence of filaments in the granules was detected. High proportion of *Chloroflexi* filaments were detected by FISH in the sludge. Changes in the microbial population caused by the low pH probably destabilize the reactor performance. The presence of filamentous granules in the sludge and its further growing could be encouraged by the pH drop and the low specific organic load applied to the reactor. The low specific organic load was a consequence of the high VSS content in the UASB reactor, due to the lack of purges. The length of the filaments attached to the granules grew throughout time. In order to eliminate the sludge with poor settlement properties a recycle was applied to the reactor. As a consequence, low amount of granular sludge stayed in the reactor. At the end, COD concentration in the influent reached higher values than in normal operation; at the same time a complete sludge wash out occurred. On the other hand, using the same sludge (after the recycle implementation) in a bench scale reactor the good properties of the sludge were completely recovered.

**Key words** | *Chloroflexi*, filaments, full scale UASB, loading rate, pH drop

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### INTRODUCTION

There are a considerable number of full scale UASB reactors working all over the world (Frankin 2001; van Lier *et al.* 2001). Nevertheless, there are not many scientific reports about full scale reactors performance during long operational periods. Microbial characteristics of the sludge normally change throughout time and depend on the environmental conditions in the reactor (Fukuzaki *et al.* 1995). The start up strategy and the steady state conditions imposed influence the microbial composition of the sludge and as a consequence determine the reactor performance

(Saiki *et al.* 2003; Zhou *et al.* 2007). To detect slow changes in the microbial community long term studies of the reactor performance must be done.

The microbial composition of the sludge could determine the failure of a reactor. For instance, several works reported the presence of filamentous sludge even in granular bed reactors (Abreu *et al.* 2007). It is well known that filamentous sludge has settlement problems. Besides, microbial distribution within granules can determine sludge properties like settlement characteristics (Saiki *et al.* 2002).

However, the most important thing is to relate the conditions imposed to the reactor with the consequences observed in the sludge. In this way should be possible to know how to manage the reactor conditions in order to obtain an adequate sludge.

The volumetric organic load is a common parameter used to follow the performance in scientific reports. Nevertheless, the specific organic load (*i.e.* organic load per VSS content) presents more accurate information than the volumetric load. For instance, for a reactor fed with a certain volumetric load the specific organic load depends on the solids content in the reactor. For this reason the specific loading rate is a more proper parameter to be reported. Besides, the specific loading rate varies even at constant fed conditions because of sludge growing and the solids lost (wash out and purges).

A full scale UASB reactor was operated during 111 weeks treating the effluent of a malting plant. In previous works results from the full scale and a pilot experiences were reported (Martínez *et al.* 2001; Borzacconi *et al.* 2006). In this work, the performance of the full scale reactor was analyzed before and after a functional problem. The scope of this work was to correlate the operational conditions with the sludge characteristics and the reactor performance.

## MATERIALS AND METHODS

A full scale (250 m<sup>3</sup>) UASB reactor (Borzacconi *et al.* 2006) was inoculated with sludge taken from an anaerobic lagoon used for the treatment of slaughterhouse effluents. The amount of sludge used was 3900 kg of TSS with an average content of 77% VSS (3,000 kg) and with a SMA of 0.1 gCOD/gVSS.d. Small granules of an average size of 0.7 mm were present in the inocula. The average flow rate to be treated coming out of the steeping process of a malting plant was 350 m<sup>3</sup>/d. The average concentration of the stream was around 2,400 mg COD/L. Working temperature was clearly influenced by the industrial process temperature and was around 20°C.

For monitoring purposes COD, VFA and alkalinity were measured at the inlet and outlet of the reactor. COD was determined by the reflux method (APHA 1995) and VFA and alkalinity by a simplified method proposed by

DiLallo & Albertson (1961). The pH, temperature and flow rates (liquid and gas) were registered online using the industrial plant SCADA system. The biogas production was established throughout time and gas chromatography was performed to determine its composition. Samples of the sludge bed were periodically collected from different depths and solid contents and size of the aggregates were determined. The granules were immobilized in agar in order to measure the size distribution. Tests of specific methanogenic activity (SMA), settlement velocity and sludge volumetric index (SVI) were also performed. Fluorescent in situ hybridization (FISH) was performed in samples taken from the sludge using probes directed to the *Chloroflexi* phylum GNSB 941 and CFX 1,223 (Björnsson *et al.* 2002)

## RESULTS

The UASB reactor was inoculated with granular sludge and this condition remained during all the operational period, even when filaments appeared in the granules on week 81. As shown in Figure 1, the volumetric organic load was around 4 kgCOD/m<sup>3</sup>.d in all the operational period while the values obtained for the specific organic load depends of the sludge amount in the reactor. As after the start up purges were performed at different times, the specific loading rate presented variations according to the microorganisms growing rate and the purges regime.

The evolution of TSS and VSS content in the reactor are presented in Figure 2. The increase and decrease of TSS and VSS content were due to the microorganisms growing and purges respectively. On week 100 flow recirculation was performed in order to eliminate a fraction of the sludge which presented poor settlement characteristics due to the filaments presence. As a consequence, a significant portion of the sludge was washed out and the solids content in the reactor diminished.

In Figure 3 inlet and outlet COD variation during time are presented. The start up was performed in 9 weeks achieving COD removal efficiencies higher than 80%. After the start up the biogas production was nearly 300 m<sup>3</sup>/d with a methane content of 77%. The sludge presented a SMA around 0.25 gCOD/gVSS.d. On week 37 due to a problem in the industrial process the pH in the influent dropped to

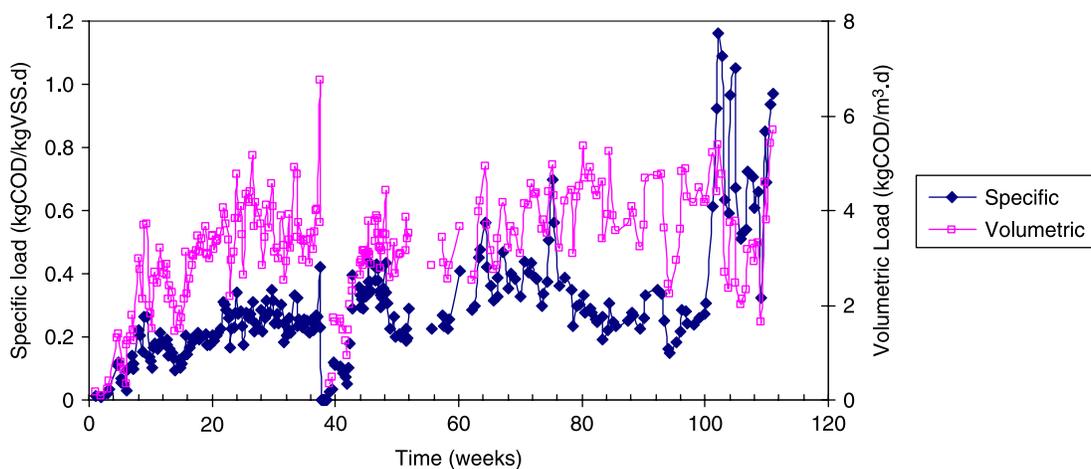


Figure 1 | Specific and volumetric organic load.

4.8. The reactor was fed with this acidified influent for almost three days. After that, the feeding was stopped and  $\text{Na}_2\text{CO}_3$  was added. After recovering of the pH to values close to neutrality, the feeding was restored increasing the flow rate gently. Until this event happened, outlet COD values were generally below 500 mg/L. From that week forward, the quality of the effluent decrease with important variations in the COD removal efficiency. In Figure 4 specific loading rate and COD removal efficiencies are presented in the operational period.

It can be observed from Figure 4 that COD removal efficiency was about 80% before the acidification episode on week 37. After that, values of COD removal showed a downward trend.

After week 81 the appearance of the granules change, filaments were found to sticking out of the surface of the

granules (Figure 5). The filamentous appearance of the granules increased through time. This sludge had fluffy characteristics and worse settlement properties than those previous to week 81. High proportion of thin filaments hybridizing with the *Chloroflexi* probe was detected by FISH in the sample taken after this moment (Figure 5), suggesting an overgrowth of this filaments in the granules. Besides, after the detection of these filamentous granules, COD removal was significantly lower.

In Figure 6 the cumulated theoretical methane production per COD removed and the determined experimentally are presented. The theoretical biogas production was calculated assuming a yield of  $Y = 0.10 \text{ gVSS/gCOD}_{\text{rem}}$ . This value was determined in the pilot experience (Martínez *et al.* 2001) and confirmed in the full scale experiment using data from the first 35 weeks (Borzacconi *et al.* 2006).

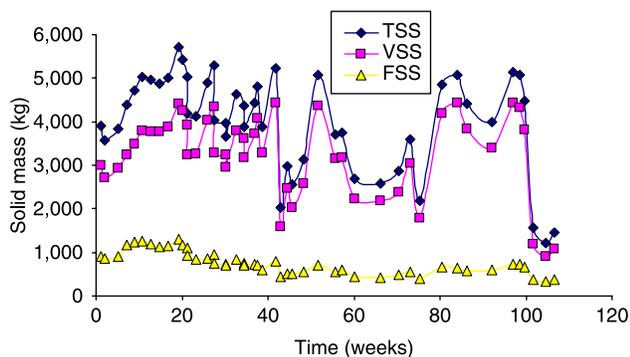


Figure 2 | Total solid content in the reactor.

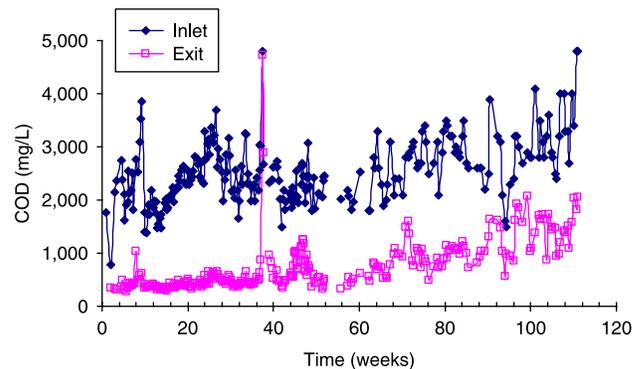
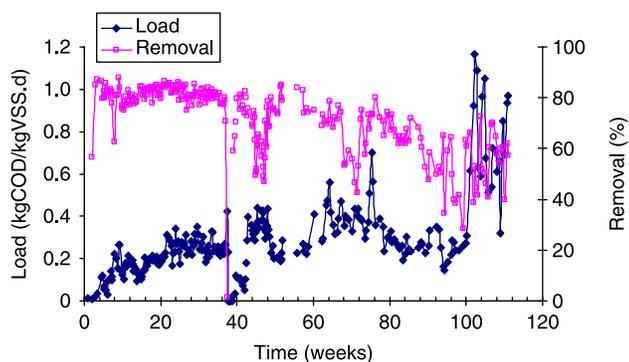


Figure 3 | Inlet and outlet COD throughout time.



**Figure 4** | COD removal and organic load per VSS.

After week 80 (filamentous granules appearance) a clear trend to divergence was observed.

Intending to wash out filamentous granules and to retain those with less filamentous characteristics, a recycle was imposed. After a few days of the recycling operation the sludge with worse settlement properties was washed out and 900 kg VSS maintained in the reactor. Because of this, the specific organic load increased after week 102. Additionally, in this period the COD values at the inlet were very high and the specific organic load reached values higher to 1 kgCOD/kgVSS.d.

In the last period after the filamentous granules wash out the specific load rate increased and the COD removal was partially recovered. Nevertheless, the COD removal had a fluctuating behavior probably due to the specific loading rate variations.

On week 110 because of changes in the industrial process, the inlet COD reached values of 4,000 mg/L.

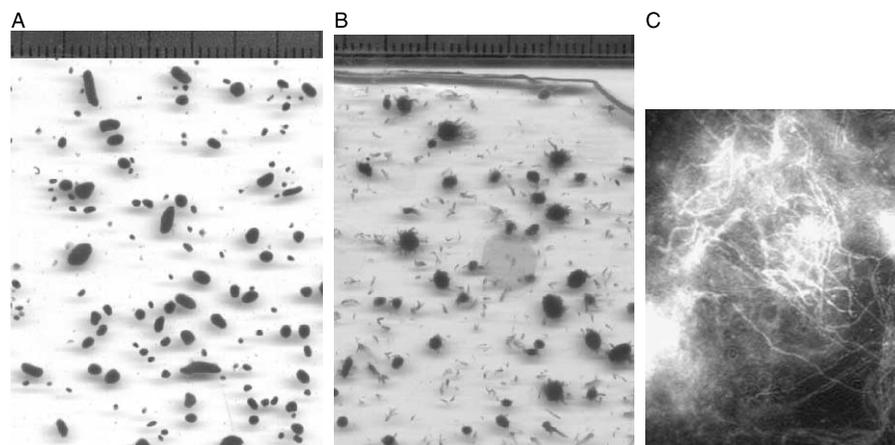
As these higher values of inlet COD were the result of changes in the industrial process it was possible that changes in the COD nature also happened. After these changes a complete wash out of the sludge in the reactor was detected.

Using the sludge obtained after the recycling operation a bench scale reactor was operated with high specific loading rate under controlled conditions. After 3 weeks the granular sludge was completely recovered achieving COD removal of 80% with a specific loading rate of 1 kgCOD/kgVSS.d (data not shown).

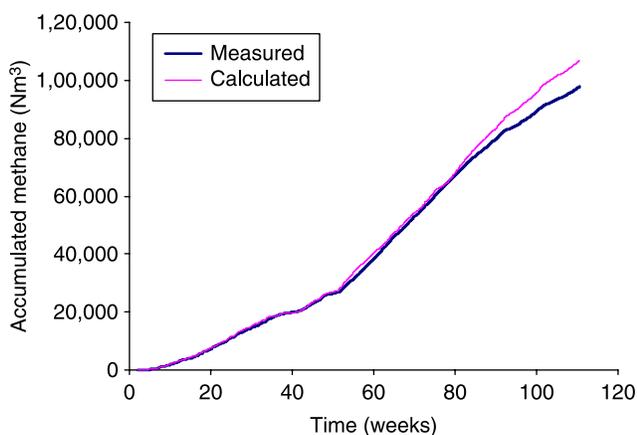
## DISCUSSION

During the start up and until week 37 the specific organic load presented low values, around 0.2–0.4 kgCOD/kgVSS.d. In spite of these low values in the specific organic load, no impact was detected on the sludge characteristics or in the SMA. In former studies in a pilot reactor treating malting plant effluent, the specific organic loads achieved were between 0.6 and 1.0 kg COD/kg VSS d (Martinez *et al.* 2001).

After the pH drop in the influent on week 37 the biogas production instantaneously stopped. After the normal pH values were recovered the loading rate was progressively increased. The COD removal achieved was lower than before and presented a random behavior. This fact could indicate that even the inhibition could be apparently reverted the microorganisms population was affected. As the methanogenic population is generally more sensitive



**Figure 5** | Photographs of granules immobilized in agar. A: week 71; B: week 92, C: FISH microphotography showing the filaments hybridizing with the *Chloroflexi* probe.



**Figure 6** | Accumulated methane production values, experimentally measured and calculated (for the calculation a  $Y = 0.10 \text{ gVSS/gCOD}_{\text{rem}}$  was used).

to changes, these microorganisms could be more affected. On week 81, filamentous microorganisms were found in the sludge. Granules with similar appearance cause settling problems in anaerobic UASB reactors as was reported by Sekiguchi and coworkers (Sekiguchi *et al.* 2001; Yamada *et al.* 2005). Filaments affiliated to the *Chloroflexi* sub-phylum I was identified in the filaments projections of the granules. According to this report, these bacteria can degrade carbohydrates and yeast extract (similar to cellular compounds). After the pH drop, lyses of a fraction of anaerobes could occur and consequently the growth of the *Chloroflexi* bacteria could be favored. Due to the fact that purges were made not often enough the cellular residence time of the sludge was high. Filaments from the *Chloroflexi* phylum have been reported to present a very slow growth (Sekiguchi *et al.* 2001) so, the high cellular residence time could promote its development. As the filamentous granules increased throughout time, settlement properties became worse. Saiki *et al.* (2002) measured in granules the depth of a surface layer of bacteria and an inner one of the methanogens. They found that the depth of the layers was related with the tendency to float. High values of these layers depth difficult the mass transfer phenomena. As a consequence lyses of bacteria inside the granule may occur and gas formed is not readily released. In this work, after the filamentous granules appeared several void granules were detected (data not shown). This may be due to the growing of the bacteria layer with the consequent transfer mass problems. Then, the flotation of the sludge could be

favored by the presence of the filamentous granules and also by formation of low density granules with the biogas trapping inside.

Specific loading rates were low, particularly after week 80; this fact could stimulate lyses and as a consequence favor the growth of *Chloroflexi* filaments from this cellular detritus.

Regarding the biogas production per COD removed, the difference between the theoretical and experimental values detected after week 80 could be explained by a change in the microorganisms population with a concomitant change in the  $Y$  value. Probably after the operational problem, bacteria with a higher  $Y$  value were selected. Because of that, the methane production per COD removal decreased.

In spite of the partial recover showed after the pH adjust (week 37) the full scale reactor behavior was not stable. Nevertheless, working in bench scale using sludge from the reactor (week 107) the performance of the reactor was completely recovered using high specific loading rates. Besides, filamentous granules predominance was not detected in the sludge working in these conditions. It must be noticed that the bench scale reactor was operated with controlled conditions avoiding loading rates fluctuations and high values of COD.

After the new start up using an adequate purge regimen the full scale reactor will be operated with a higher specific organic load and consequently a higher SMA in order to avoid the problems presented in this work.

## CONCLUSIONS

Operational conditions in a reactor affect the microorganisms population development. In this case, the low specific load applied had no consequences until a pH drop due to a problem in the industrial process occurred. From pH drop forward, the low value of the specific organic load applied during several month of operation and the high cellular retention time values seem to favor microorganisms which grow from lyses products. Therefore, this microorganisms could be encouraged as a consequence of the pH drop. At the beginning granules had a normal shape and after the pH drop filamentous granules were developed, increasing filaments length through out time. High proportion of thin

filaments was detected by FISH in the sludge after the pH drop. Then, settling problems and low performance in the reactor could be related with the *Chloroflexi* presence. The changes in the microbial population produced destabilization in the reactor. Also the change detected in the biogas production per COD removed was probably due to the yield change because of the variation of the sludge population. Applying high specific loading rates in a bench reactor was possible to recover the same sludge that was not possible to recover in the full scale reactor conditions.

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