Improvement of the properties of granular sludge in UASB reactors by flow pulsation

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Abstract The objective of this work is to improve the characteristics of granular sludge by modification of the hydraulics of the bed through flow pulsation. Three UASB reactors, two operated with pulsing flow (P1 and P2) and a third without pulsation (NP), were started-up. Both recycling and feeding flow were pulsed in the reactor P1, while in reactor P2, only the feeding was pulsed. A high increase in the removal capacity and stability were achieved by applying pulsation in reactors P1 and P2 when compared to the non-pulsed one. Besides, pulsation promotes the formation of particles of smaller size and higher porosity, thus increasing the specific surface of the bed and consequently, the specific activity. In fact, while reactors P1 and P2 had a 95% COD removal when working at high organic loading rates (12 kg COD/m³·d), reactor NP only reached 6 kg COD/m³·d with 85% of removal capacity.

Keywords Flow pulsation; granular sludge; UASB

Introduction
The development of granular sludge with good bulk properties is essential for the stable operation and the good performance of high-rate UASB reactors. The composition of the wastewater has an important influence on granule properties. Indeed, the treatment of certain types of substrates could result in the development of a poor sludge bed (Batstone and Keller, 2001). However, the selection of the wastewater to be treated is not an option. A possible alternative for the improvement of the properties of the granular sludge is the modification of the operating conditions, including environmental parameters and hydraulics. Pulsation has been employed in many chemical engineering units to improve mass transfer rate, being firstly used in separation processes in order to enhance the contact between phases. Recently, it has been developed and adapted for its application in several fields, including biochemical reactors (Sanromán et al., 1994; Roca et al., 1996; Mielgo et al., 2002).

The main objective of this study is the evaluation of the influence of flow pulsation on the characteristics of a granular sludge in UASB reactors. An additional important point is to assess the interest of applying that technique for improving the efficiency of UASB reactors, by increasing: i) mass transfer rate (by reducing the granules size); ii) specific methanogenic activity (SMA) of the biomass; and iii) the useful volume of the reactor.

Materials and methods
Three lab-scale UASB reactors of 0.8 L of useful volume were operated for a period of four months. Two reactors, P1 and P2, were operated with pulsing flow and the third one, NP, was operated without pulsation. The pulsing flow was introduced to reactors P1 and P2 by an elastic membrane pulsator (EMP) (Roca et al., 1994), which consists in an elastic tube connected to an electrovalve controlled by a timer. A pump accumulates liquid in the tube until the electrovalve is opened. In the reactor P1 both recycling and feeding flow were pulsed, while in reactor P2, which was operated without recycling, only the feeding flow was pulsed. Previous studies (Franco et al., 2002) showed that the optimal shutting time \( t_s \) to be applied ranged between 200 s and 1800 s after each pulsation. Table 1 shows the
shutting times ($t_s$), the frequencies ($f$) and the pulsed volume ($V_p$)/reactor volume ($V_r$) ratio.

A solution of monohydrated dextrose was employed as substrate, with a Chemical Oxygen Demand (COD) concentration of 5 g/L, and NH$_4$Cl and K$_2$HPO$_4$ were added as macronutrients. No micronutrients were employed. The inoculum of the reactors was a granular sludge from an UASB-UAF reactor treating dextrose, with a SMA of 0.27 kg CH$_4$-COD/kg VSS·d. The initial concentration of biomass in the reactors was 15 g VSS/l.

The parameters measured daily to control the performance of the reactors were COD, Volatile Fatty Acids (VFA), alkalinity and biogas composition. COD was determined by a semi-micro method (Soto et al., 1989). VFA was determined by gas chromatography (Hewlett-Packard 5890A), equipped with a flame ionisation detector (FID) and alkalinity was measured by titration with sulphuric acid (Ripley et al., 1986). Biogas composition was analyzed via TCD gas chromatography (HP 5890 SerieII), with helium as carrier gas. The parameters measured for biomass characterization were total suspended solids (TSS) and volatile suspended solids (VSS), specific methanogenic activity (SMA), specific acidogenic activity (SAA), specific density (SD) and size distribution. The specific methanogenic and acidogenic activities were determined according to the methods described by Soto et al. (1993b). The specific density of granules was obtained by the method described for Beun et al. (2002). Size distribution of the granules from the sludge bed was determined by the method indicated by Jeison and Chamy (1998). The structure of the biomass was observed by scanning electron microscopy (SEM) using a Digital SEM LEO 435 VP controlled by computer, with achievable magnifications between 15× and 290,000×.

### Results and discussion

#### Reactor operation

The three reactors were started-up simultaneously and the same operating conditions were maintained for the three reactors until an organic loading rate (OLR) of 4.5 kg COD/m$^3$·d. However, due to the low SMA of the seed sludge, the non-pulsed reactor suffered a destabilization in that point, and the OLR had to be decreased. The OLR applied to reactors P1 and P2 was increased until 12.5 and 14 kg COD/m$^3$·d, respectively (Figure 1), while reactor NP achieved only an OLR of 6 kg COD/m$^3$·d.

In Figure 2 the evolution of the intermediate alkalinity/total alkalinity (IA/TA) is represented. The IA is the difference between the total and the partial alkalinity (PA), and it is equivalent to the VFA content in the reactor. It was reported that the limit value of this parameter for the stability of anaerobic reactors is 0.3 (Switzenbaum et al., 1990; Soto et al., 1993a). For reactor P1, this parameter was most of the time below 0.3, except on day 105, when the OLR of the influent was increased from 10 to 12.5 kg COD/m$^3$·d, coming back to normal values. The alkalinity ratio of reactor P2 was around 0.2 during the whole experiment, indicative of the stability of the operation. In reactor NP two important destabilizations occurred (days 30 and 60 approximately), when the alkalinity ratio increased to 0.4 and to 0.45, respectively. As a consequence of the first destabilization, it was necessary to decrease the OLR to 2 kg COD/m$^3$·d. After a period of 18 days, the OLR was progressively increased, attaining a maximum value of 6 kg COD/m$^3$·d.

In Figure 3 the average COD removal for the three reactors at different OLR is represented. Reactor P1 operated very efficiently (COD removal between 98 and 93%) until an

<table>
<thead>
<tr>
<th>Shuttering time ($t_s$)</th>
<th>Frequency ($f$)</th>
<th>Pulsed volume ($V_p$)/reactor volume ($V_r$) ratio</th>
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<tr>
<td>200 s</td>
<td>4.9·10$^{-3}$</td>
<td>1/27.5</td>
</tr>
<tr>
<td>1800–900 s</td>
<td>(1.4–11.0)·10$^{-4}$</td>
<td>1/230–1/60</td>
</tr>
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</table>
OLR of 8 kg COD/m³-d. At higher OLR, the efficiency decreased slightly until 85%, on average. However, reactor P2 reached an efficiency of around 95% in the whole experiment, even at the highest OLR. On the contrary, the NP reactor attained a much lower COD removal than pulsed reactors.

In order to explain the different behaviour of the three reactors, the hydraulics, the properties of the sludge (size, specific density and structure), and the SMA and SAA were evaluated throughout or at the end of the experiments.

**Hydraulics**

The hydraulic behaviour of the three reactors was determined by Residence Time Distribution (RTD) experiments. Dextran blue and LiCl were simultaneously employed as tracers, in order to obtain the useful working volume, the volume occupied by biomass and the void volume of the sludge bed in the three reactors. The experiments were performed in the period between days 80 and 90, when the operating Hydraulic Residence Time (HRT) was 0.7 days for the pulsed reactors and 1.14 days for reactor NP.

In Figure 4 the results corresponding to the tracers are jointly plotted (∆ or •), as well as the ideal behaviour of a single CSTR or a series of two tanks (solid line). From the curves of Dextran Blue, the hydraulic model of each reactor was determined. The curve of LiCl is not useful since it penetrates into biomass. NP and P1 reactors present a behaviour quite similar to a CSTR, as expected in reactors with recycling. However, the hydraulics of reactor P2 was close to two tanks in series, since no recycling was applied. A delay can be seen in the LiCl curve when compared with Blue Dextran curve for P1 and P2 reactors, due to the higher porosity of the sludge in the pulsed reactors.

The average HRT of each reactor was determined by using the curves of the two tracers. The comparison between these values allows the void volume in each bed to be calculated.
Reactors P1 and P2 had a liquid volume 7.5% and 6.25% higher than reactor NP, respectively, which is indicative of the high porosity of granules in P1 and P2 (Table 2).

**Biomass**

In Figure 5 the size distribution of the granules from reactors NP, P1 and P2 is presented. It can be seen that pulsed reactors have a higher fraction of small granules. Particularly in the reactor P2, 40% of granules had an average diameter lower than 0.6 mm, which led to a noticeable increase of the specific surface of the sludge bed, resulting in a better performance of the reactor.

At the end of the experiment the concentration of TSS and VSS, and the SD of the sludge of each reactor was determined. Concentration of solids is quite similar in the three reactors (Table 3), although the SD of granules of P1 and P2 reactors are higher. This should be not expected due to the higher porosity of the biomass in pulsed reactors. An increased bacterial aggregation in the granules of P1 and P2 would explain these results. However, a further study should be developed in order to confirm this hypothesis.

In Figure 6, SMA of the granular sludge on days 36, 84 and 123 are shown. The sludge of reactor P2 increased very much with respect to the initial value of 0.27 kg CH₄-COD/kg VSS-d. Sludge from reactor P1 also increased that value, although at the end of the experiment it decreases down to the initial value. However, SMA of the sludge from NP decreased to 0.11 kg CH₄-COD/kg VSS-d, indicative of the worse performance of this reactor during start-up. The SAA of the sludge from the three reactors was determined at the end

**Table 2** Liquid volume \( (L_v) \), void volume \( (V_v) \) and biomass volume \( (B_v) \) for reactors NP (Non pulsed), P1 (influent and recycling pulsation), and P2 (influent pulsation)

<table>
<thead>
<tr>
<th></th>
<th>( L_v ) 0</th>
<th>( V_v ) 0</th>
<th>( B_v ) 0</th>
</tr>
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<tbody>
<tr>
<td>NP</td>
<td>0.53</td>
<td>0.22</td>
<td>0.27</td>
</tr>
<tr>
<td>P1</td>
<td>0.59</td>
<td>0.25</td>
<td>0.22</td>
</tr>
<tr>
<td>P2</td>
<td>0.58</td>
<td>0.27</td>
<td>0.21</td>
</tr>
</tbody>
</table>
It is interesting to highlight the important difference between the values from the pulsed reactors (7.4–7.6 kg COD/kg VSS·d) in contrast to the non-pulsed one (15.7 kg COD/kg VSS·d).

In Figure 7, glucose degradation profiles of biomass from the three reactors are shown. The higher SAA of the sludge from the NP reactor favoured the quick degradation of dextrose into VFA, although its smaller SMA impedes the complete metabolisation of the VFA produced. On the contrary, the pulsed reactors presented more balanced values between SAA and SMA, which favours the proper performance of the system, this being another factor which explains the better behaviour of the pulsed reactors.

Finally, the structure of the biomass was observed by SEM. In Figure 8 the difference between the structure of the external layer of the granules from the pulsed reactors and the non-pulsed one can be appreciated. Bacteria from the external layer of granules in the NP reactor present a compact structure, almost without channels, while granules from P1 and P2 show cavities, characteristic of their high porosity.

### Conclusions

The positive effect of flow pulsation on the behaviour and efficiency of UASB reactors working with granular sludge could be explained in terms of biomass characteristics and hydraulics.

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**Figure 5** Size distribution of granules from reactors NP (Non pulsed), P1 (influent and recycling pulsation), and P2 (influent pulsation)

**Figure 6** Specific methanogenic activity (SMA) of sludges from reactors NP (Non pulsed), P1 (influent and recycling pulsation), and P2 (influent pulsation)

**Figure 7** Glucose degradation profiles of sludges from reactors NP (Non pulsed), P1 (influent and recycling pulsation), and P2 (influent pulsation)

**Table 3** Concentration of suspended solids and specific density (SD) of sludges from reactors NP (Non pulsed), P1 (influent and recycling pulsation), and P2 (influent pulsation)

<table>
<thead>
<tr>
<th></th>
<th>NP</th>
<th>P1</th>
<th>P2</th>
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<tbody>
<tr>
<td>TSS (g/l)</td>
<td>26.88</td>
<td>25.0</td>
<td>24.88</td>
</tr>
<tr>
<td>VSS (g/l)</td>
<td>26.38</td>
<td>22.25</td>
<td>22.88</td>
</tr>
<tr>
<td>SD (gVSS/l)</td>
<td>69.2</td>
<td>84.6</td>
<td>82.0</td>
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</table>
Influent pulsation improves very much the Specific Methanogenic Activity of the granular sludge. Besides a good balance between the different microbial trophic groups implied in the anaerobic degradation is achieved. Pulsation allows the production of granules with a higher porosity, which is an interesting factor for facilitating the diffusion of nutrients and substrate into the granules. Finally, pulsation favours the formation of smaller granules, which increases the specific surface of the sludge bed and the specific density of the granules.

From the point of view of hydraulics, pulsation allows a more efficient degassing of the sludge bed and avoids the formation of preferential channels. All of these effects on the biomass structure and on the hydraulics improves significantly the performance of the pulsed reactors, leading to a better removal capacity and to a quicker start-up.

References

Figure 8 SEM photos of the external layer of granules from reactors NP (Non pulsed), P1 (influent and recycling pulsation), and P2 (influent pulsation)