Anaerobic hydrolysis of a municipal wastewater in a pilot-scale digester

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Abstract Raw domestic wastewater from the city of Santiago de Compostela (Northwest Spain) was fed into a pilot-scale hydrolytic up flow sludge bed (HUSB) digester with an active volume of 25.5 m^3. The total influent chemical oxygen demand (COD) ranged from 360 to 470 mg/l, the influent SS varied from 190 to 370 mg/l, and the temperature was between 17° and 20°C. The organic load rate (OLR) applied increased step by step from 1.2 to 3.9 kgCOD/m^3.d, while the hydraulic retention time (HRT) decreased from 7.1 h to 2.9 h. A high suspended solids (SS) removal of about 82–85% from the influent was reached, most of which (81 to 88%) was eliminated by hydrolysis, while the rest remained in the purge stream. The total COD removal ranged from 46 to 59%. On the other hand, a high acidification of the COD remaining in the effluent was obtained, so the percent COD in the form of volatile fatty acids (VFA_{COD}) with respect to total effluent COD was about 43% for the highest HRT applied, and about 27% for the lowest HRT. The soluble to total COD ratio (CODs/CODt) increased from 25–32% for the influent to 71–86% for the effluent. The results obtained confirm the viability and interest of direct anaerobic hydrolytic pre-treatment of domestic wastewater.

Keywords Anaerobic digestion; hydrolysis and acidification; urban wastewater

Introduction

Compliance with the criteria as stipulated in Directive 91/271/CE of the Council of Europe involves the construction of new treatment facilities in some countries, with the need for new economic and efficient treatment methods. During the last decade different anaerobic technologies have been applied to the treatment of low concentrated effluents, such as domestic wastewater and some industrial effluents, providing good treatment efficiencies at low or very low hydraulic retention times. The principal application of anaerobic digestion in urban wastewater treatment consists of the utilisation of single-step methanogenic digesters for organic load removal (Lettinga *et al*., 1993; Ruiz *et al*., 1998; Foresti, 2001).

Another option is the separation of phases, in which wastewater undergoes a pre-hydrolysis–acidification step before anaerobic digestion. Two-step systems permit the optimisation of each of the individual process steps that are involved in anaerobic digestion. However, the first step should be considered as a wastewater pre-treatment, as the hydrolysed effluent could be followed by an aerobic treatment, such as activated sludge, lagoon or wetland systems, instead of the final methanogenic step.

Some advantages of the direct hydrolytic pre-treatment of domestic wastewaters are the following (Wang, 1994; Gonçalves *et al*., 1994; Ligero, 2001a,b): it serves to remove an elevated percentage of SS, substituting the primary settler at a similar HRT; it stabilises the sludge, totally or partially; and increases the biodegradability of the remaining COD, which favours the subsequent biological elimination of nutrients (N, P).

Different variables, such as wastewater characteristics, type of digester, up flow velocity (v), mixing mechanism, HRT and solids retention time (SRT) influence this process. The conversion reached during the hydrolytic pre-treatment of an actual urban wastewater, and the influence of HRT and SRT were investigated in a previous work carried out on a lab scale (Ligero, 2001a,b). In this article we report the operation and efficiency of a pilot plant digester.

Materials and methods

An existing metallic cylinder 2.5 m in diameter with a total height of 7.1 m was adapted and used as digester. The total and active volumes were 34.9 and 25.5 m^3, respectively. Four lateral ports allowed us to take samples of the digester content at different heights: 0.1 m (P1), 1.25 m (P2), 2.5 m (P3) and 4.5 m (P4). There was a solid/liquid/gas separator in the upper zone similar to that used in UASB digesters. The influent flow was determined by using an electromagnetic meter, type MS-1000 of Iberfluid. Biogas production flow rate was measured by a gas flow meter, type E-7000 of Iberfluid.

The digester was located in the Municipal wastewater treatment facility of Santiago de Compostela, and was fed with the raw domestic wastewater, after sand and grease removal. Sludge seed was not used, the start-up being accomplished by operating the digester at a higher HRT until a sludge bed was developed, as reported previously (Álvarez *et al*., 2001). At the beginning of this study, the digester was working with an HRT of 11 h, and had an overall sludge concentration of 10.7 g VSS/l with a methanogenic activity of 0.025 g COD_{CH4}/gVSS.d (Alvarez *et al*., 2001).

Pulse experiments were carried out in order to determine the hydraulic residence time distribution. For this purpose, one litre of a solution containing 182 g of LiCl was instantaneously introduced into the wastewater stream entering the digester. Next, during a time period longer than three or four times the HRT, successive samples of the effluent were taken and their lithium concentration was determined.

Influent and effluent samples were collected every three hours by using an automatic sampler and then combined in order to obtain a daily composite sample. Generally, this procedure was followed five days a week. Analytical methods were previously described (Ligero *et al*., 2001a).

Results

Wastewater characteristics and digester operation conditions

Figure 1 shows the main characteristics of the digester operation and performance. The digester operation was divided into four periods, in regard to the HRT applied, ranging from 7.1 to 2.9 h. Table 1 presents the operational conditions, while Table 2 gives the influent, effluent and conversion parameters of the digester, as average values for each period.

As the digester operation elapsed from May to July, the temperature increased from 16 to 18°C during period I, and was kept at about 20°C for periods II to IV. Influent total COD and SS also increased throughout the operation time. Total COD ranged from 300 mg/l at the beginning of the operation up to 500 mg/l at the end, while SS varied from 160 to more than 300 mg/l. The COD present in this wastewater was mostly in the form of particulate matter, so total and volatile suspended solids (SS and VSS) contributed 68–75% of the total

Table 1 HUSB operational conditions

Period	Days	Duration (d)	T^a (°C)	HRT (h)	OLR (kgCOD/m^3.d)	v (m/h)	X_R (gVSS/l)	SRT (d)
I	1 – 32	32	17,5	7,1	1.22	0.7	10,2	50.5
II	33 – 56	24	19,5	5,2	1.95	1.0	6,3	24.7
III	57 – 73	17	19,9	3,4	3.11	1.5	7,0	10.5
IV	74 – 85	12	20,5	2,9	3.88	1.8	6,9	10.4

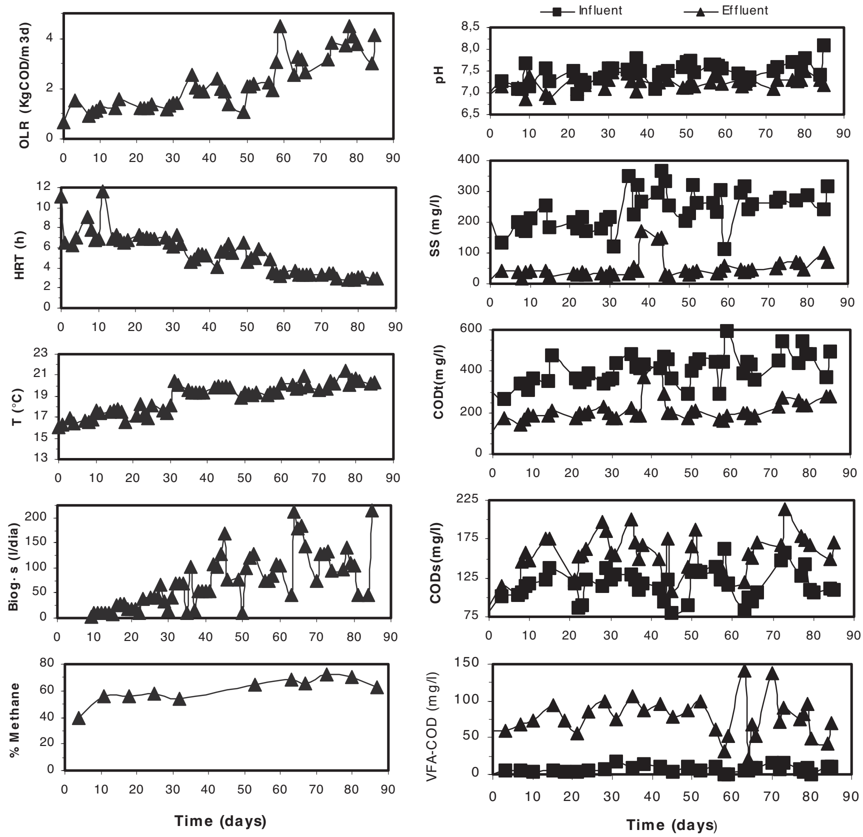

Figure 1 Main characteristics of the digester operation and performance

COD. On the other hand, the influent soluble COD remained mostly constant, accounting for a decreasing proportion of the influent COD. Only a minor portion, less than 2%, of the influent COD appeared in the form of VFA.

The OLR was increased step by step from 1.2 kgCOD/m^3.d for period I to 3.9 kgCOD/m^3.d for period IV, following the HRT decrease imposed on the system and the increase of influent COD. Furthermore, Table 1 shows the overall sludge concentration in the digester, expressed as VSS, and the resulting SRT. The overall amount of sludge in the digester was calculated from the VSS concentration profiles, which are shown below. SRT was estimated as the ratio between total sludge mass in the digester, M_x (as VSS), and sludge production rate, F_{SP}, as previously indicated (Ligero *et al.*, 2001b).

As the height of the sludge bed in the digester increased over time, periodic sludge purges were carried out by withdrawing the digester content above port P4. This was done each week or when the effluent SS concentration became higher than on previous days. An exception to this occurred during period II (days 38 to 43) when the purge was delayed for several days, then increasing noticeably the effluent SS and VSS concentration. Total COD, SS and VSS data from these days were not used to obtain average values in Table 2.

Although a biogas with a methane content of 55 to 70% was recovered, the methane generation rate was very low. However, we should take into account that the major portion of generated methane left the digester dissolved into the effluent.

Effluent characteristics and conversion

Effluent SS and VSS were very low, in accordance with the high suspended solids removal

Table 2 HUSB influent and effluent characteristics and digester efficiency

Period	I	II	III	IV
Influent				
pH	7.33	7.53	7.48	7.7
SS	188	284	257	373
VSS	169	243	207	283
COD_t	361	423	440	469
COD_s	115	117	121	118
%CODs/CODt	31.9	27.7	27.5	25.2
VFA_{COD}	6	9	8	6
$\%VFA_{COD}/CODt$	1.7	2.1	1.8	1.3
Effluent				
pH	7.15	7.23	7.27	7.28
SS	33	43	47	61
VSS	31	36	44	59
COD_t	182	198	181	254
COD_s	155	155	156	180
%CODs/CODt	85.2	78.3	86.2	70.9
VFA_{COD}	76	87	74	68
$\%VFA_{COD}/CODt$	41.8	43.9	40.9	26.8
$\%VFA_{COD}/CODs$	49.0	56.1	47.4	37.8
Removal				
%SS	82.4	84.9	81.7	83.6
%VSS	81.7	85.2	78.7	79.1
%CODt	49.6	53.2	58.9	45.8
%CODs	–34.8	–32.5	–28.9	–52.5
Acidification				
$VFA_{COD}/CODadded$	0.211	0.206	0.168	0.145

Concentrations in mg/l. Acidification in mg VFA_{COD}/mg CODt (effluent/influent)

capacity developed by the digester, which was always above 80% for SS and VSS. Part of the retained solids were hydrolysed, thus contributing to an increase in the effluent soluble COD, while another part was removed through the purge stream. So, the CODs/CODt ratio increased from 25–32% for the influent to 71–86% for the effluent (see Table 2).

The determination of which part of the retained suspended solids were actually hydrolysed or solubilised requires the calculation of sludge balances applied to the digester, as shown below. The data obtained for VSS hydrolysis (Table 3) indicate that suspended COD solubilisation was about 80% of the suspended COD retained, and suggest that the digester maintained most of its capacity to hydrolyse suspended COD even at the lowest HRT applied.

On the other hand, soluble COD was partially converted into volatile fatty acids. Thus, the VFA concentration in the effluent ranged from 68 to 87 mg COD/l, leading to an acidification of 15–21% of the original COD, while the percent of effluent COD as VFA was in the range of 41–44% for periods I to III and 27% for period IV. Thus, the overall

Table 3 VSS balance in the digester

	VSS (kg)			Remaining VSS (% of influent)		Hydrolysed VSS	
Period	Influent	Effluent	Sludge	Effluent	Sludge	% of influent	% of removed
I	452	83	46	18,4	10,2	71,5	87,5
II	658	97	77	14,7	11,7	73,6	86,3
III	596	127	187	21,3	31,4	47,3	60,1
IV	657	137	97,4	20,9	14,8	64,3	81,3
Overall	2363	444	407	18.8	17.2	64.0	78.9

Table 4 COD balance in the digester

Period	COD (kg)				Recovered COD (% of influent)			
	Influent	Effluent	Sludge	CH_4	Effluent	Sludge	CH_4	Total
I	964	486,3	85,3	229	50,4	8,8	23,8	83,1
II	1144	536,0	211	172	46,9	18,4	15,0	80,3
III	1269	521,3	347	168	41,1	27,3	13,2	81,7
IV	1088	589,6	146,2	143	54,2	13,4	13,1	80,8
Overall	4465	2133	790	712	47.8	17.7	16.0	81.5

acidification capacity of the digester decreased for the lower HRT. The acidification of soluble COD also decreased for period IV, as indicated by the percent of effluent CODs that appeared in the form of VFA.

Generally, 100% of the effluent VFA corresponded to acetic acid, since propionic or butiric acids were not found. Finally, the higher VFA concentration in the effluent of the digester caused a drop in the effluent pH, which generally was in the range of 7.1 to 7.3, always below the influent pH.

Sludge profiles and flux distribution

Both the overall sludge concentration and distribution are important factors which influence the digester operation, as the sludge bed should act as a filter which retains the influent suspended solids. Furthermore, the determination of sludge profiles in the digester was required to calculate the overall amount of sludge and the SRT. Sludge profiles, such as VSS, are shown in Figure 2. The SS profiles were very similar to VSS profiles, as the percent of VSS to SS was nearly constant, giving a value of 63.8 ± 2.7% as average for all ports and periods, although the %VSS increased slightly with the digester height and operation time. The sludge concentration was high at the bottom of the digester and decreased sharply through the first or first two metres, remaining nearly constant from this height upwards. On the other hand, the sludge concentration decreased in all ports from period I to II, increasing again slightly in periods III and IV.

These results point to a very regular pattern of solid sedimentation over the entire period of operation. The sludge distribution was not noticeably influenced by the up flow velocity of the liquid in the digester, which increased as the HRT decreased, thus ranging from 0.7 to 1.8 m/h for periods I to IV, respectively (see Table 1).

This behaviour is consistent with the flux distribution pattern that occurred in the digester, as indicated by the results of hydraulic residence time distribution (RTD) experiments (Figure 3). These experiments show that the digester did not have significant stagnant regions or other important flux deviations such as channelling or by-pass and internal

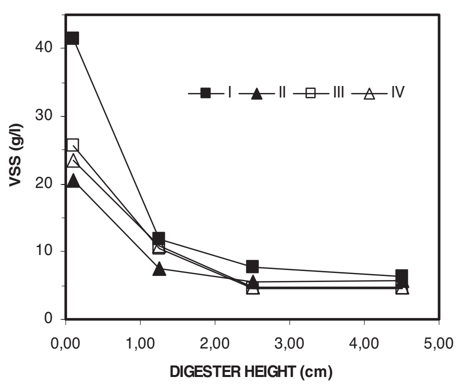

Figure 2 VSS sludge profiles in the digester

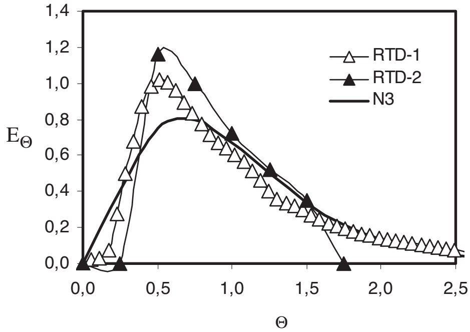

Figure 3 Results of the hydraulic residence time distribution (RTD) experiments (experiment RTD-1 was performed during the digester start-up, while experiment RTD-2 corresponded to period III)

recycling of fluid. In Figure 3, the experimental RTD is compared with the theoretical curve for N (number of tanks) = 3 given by the mixed tanks model. The application of this mathematical model to the experimental curves results in $N = 3.5$ for RTD-1 and $N = 4.6$ for RTD-2. However, experimental RTD curves point to the existence of an initial plug flow fraction which increased slightly with the drop in HRT, and the axial dispersion was lower at a lower HRT.

VSS and COD balances and excess sludge production

In order to determine the excess sludge production and the percent of influent VSS hydrolysed, a sludge balance was performed for each operational period, the results of which are shown in Table 3. In this way, sludge generation was in the range of 10–15% with respect to the fed VSS, except for period III when it reached 31%. The latter may be due to changes that occurred in the characteristics of the influent wastewater caused by occasional industrial discharges. These data indicated an elevated hydrolysis and biodegradation of solids retained in the reactor, which was calculated to be higher than 80%, except for period III (60%). Excess sludge production increased slightly when the HRT and SRT decreased, as a consequence of a decrease in hydrolysis efficiency. The purged sludge contained about 23% grease in relation to VSS. Further stabilisation in anaerobic batch digesters (data not shown) led to the removal of 40 to 50% of VSS contained in the sludge.

Similar calculations for COD balance may be performed, although in this case the use of a factor that converted sludge VSS into COD was needed, as the COD of sludge streams was not generally determined. The results of the COD balance for each operational period are shown in Table 4, assuming the available experimental values of VSS to COD, which ranged from 1.5 to 2.7 g COD/g VSS, and determining the overall amount of methane that left the digester. For the latter calculation, both the methane contained in the biogas and the methane dissolved in the effluent were taken into consideration. About 80 to 83% of influent COD was recovered in the different exit streams (effluent, purge and biogas), which means that 17 to 20% of COD was lost during the process, probably due to the effect of different biological conversion mechanisms. Overall methane production accounted for 24 to 13% of influent COD and clearly decreased with the drop in HRT and SRT.

Let us consider as a calculation base 100 kg VSS which enter the digester and the VSS balance results for the entire operational period (Table 3). As is shown in Figure 4a, only 18.8 kg VSS are going to remain in the effluent and 17.2 kg VSS in the purge current, 64 kg VSS being eliminated through hydrolysis. Only minor variations in this picture may be found when the balance is referred to a SS base.

Taking into account the results of COD balances shown in Table 4 and the influent and effluent characteristics given in Table 2, a balance for different COD fractions may be

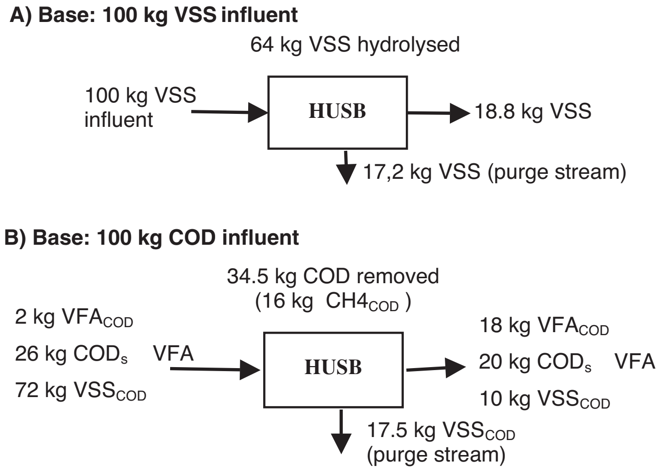

Figure 4 VSS (a) and COD (b) balances for the entire operational period of the digester

performed as shown in Figure 4b. The 100 kg COD_t that enter the digester were distributed in the following manner: 2 kg VFA_{COD}, 26 kg COD_s different from VFA, and 72 kg VSS_{COD}. For these 100 kg of COD_t that enter the digester, approximately 17.5 kg VSS_{COD} are generated in the purge current, while 34.5 kg COD are eliminated during the process as a consequence of methane generation and other mechanisms. About 48 kg COD leave the system in the effluent, distributed in the following manner: 18 kg VFA_{COD}, 20 kg COD_s different from VFA, and 10 kg VSS_{COD}.

Discussion and conclusions

Direct hydrolysis of municipal wastewater was previously reported (Gonçalves *et al.*, 1994; Ligero *et al.*, 2001a,b; Wang, 1994). Gonçalves *et al.* (1994), working with two small pilot plants with a volume of 113 and 79 l at a temperature of 20°C, obtained elevated SS elimination levels (above 69%) at up flow velocities of below 1.5 m/h, and optimum acidification results at an HRT of 2.8 h. COD_t elimination was below 25%, and COD_s increased. In a full-scale plant of 170 m^3, Wang (1994) achieved SS elimination levels of 83% and COD_t elimination levels of 43% at an HRT of 2.5 h. At an up flow velocity of 0.94 m/h and an optimum HRT of 2.2 h, Ligero *et al.* (2001a,b) reported SS elimination levels of 63% and COD_t elimination levels of 38%.

In the three studies referred to above, the retention capacity of SS increased with the volume of the plant. This may perhaps be due to a higher sludge bed height which would increase its filtration capacity. This is in accordance with the results of this research, as the sludge bed height was always above 4.5 m. Furthermore, a mixing mechanism was not necessary since the contact between the influent wastewater and the sludge was adequate, as indicated by RTD experiments.

The digester maintained a very good SS and VSS retention capacity throughout the entire period of operation regardless of the HRT. However, at the shortest HRT applied of 2.9 h, the COD acidification efficiency decreased by 30%, from 0.21 to 0.15 mg VFA_{COD}/mg COD_t added, while the VSS hydrolysis efficiency decreased by only 7% and the soluble COD production rose slightly. These facts indicate that, under these operating conditions, the hydrolysis of soluble COD – either influent or generated from VSS through liquefaction – may be the limiting step in the process.

Surplus sludge production reached about 17% of influent VSS, although about half of this sludge may be removed by further digestion. In a previous study, performed in a small lab digester working with a different influent wastewater (Ligero *et al.*, 2001b), the surplus sludge production was very low, about 2.2% of influent SS. By means of a comparison of the operational characteristics of both digesters, the difference in surplus sludge generation may be attributed in part to the differences in the retention capacity of suspended solids and not to a different hydrolysis behaviour. In fact, the fraction in which the influent suspended solids were hydrolysed is nearly the same in both studies: 60% of SS for the lab digester and 64% for the pilot plant. These considerations suggest that the suspended COD fraction remaining in the digester effluent had a lower anaerobic biodegradability than the overall influent suspended COD.

The results obtained confirm the viability and interest of direct hydrolytic pre-treatment of domestic wastewater, as reflected by an elevated SS retention and removal, and by the increase in biodegradability of the remaining COD. Thus, anaerobic hydrolysis of raw domestic wastewater may be considered as a good pre-treatment method for anaerobic secondary treatment in a two-stage digestion process, as well as for aerobic treatment, substituting the primary decanter in both cases.

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