# Pretreatment of urban wastewaters in a hydrolytic upflow digester

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# Abstract

Domestic wastewater was fed continuously to a laboratory-scale upflow digester operated at short hydraulic retention times (HRT <4 h). The digester acts as a system for sedimentation and hydrolysis of suspended solids (SS) and for acidification of solubilised substances. Optimum results were obtained at an HRT of 2.3 h. Over 60% SS are retained in the digester and hydrolysed. Average influent SS content is 230 mg/ $\ell$ , whereas effluent SS is 90 mg/ $\ell$ . Effluent SS concentrations shows very stable behaviour, varying little with influent concentration, as with HRT. Retention and hydrolysis of SS causes an increase in volatile fatty acid (VFA) concentration, from about 20 mg/ $\ell$  in the influent to above 100 mg/ $\ell$  in the effluent, also contributing toward soluble fraction acidification. Thus, VFA reached 25% of effluent chemical oxygen demand (COD), while influent VFA<sub>COD</sub> was below 4%. During the process, a reduction in domestic wastewater COD of between 30 and 40% was observed.

# Nomenclature

COD	Chemical oxygen demand (,: total, .: soluble).
HRT	Hydraulic retention time
OLR	Organic load rate
SS	Suspended solids
VSS	Volatile suspended solids
SRT	Solids retention time
VFA	Volatile fatty acids
VFA <sub>COD</sub>	Volatile fatty acids expressed as COD
HUSB	Hydrolytic upflow sludge bed (anaerobic digester)

# Introduction

Anaerobic and aerobic treatments constitute two major processes for biological purification of wastewaters and biodegradable organic wastes. Anaerobic digestion is very favourable in terms of energy due to the fact that aeration is not necessary and that biogas is produced which could be used at the same plant as an energy source, allowing in many cases for the autonomy or self-sufficiency of the treatment plant. Another major advantage is that sludge is generated in much lower amounts than those obtained with aerobic processes. As a result, energy and sludge management costs are reduced, making anaerobic digestion the most frequently used biological system for treatment of waste effluents with mediumand high-organic loads (Lettinga et al, 1993).

Moreover, during the 1980s and especially during the 1990s some research groups paid attention to the development of anaerobic digestion for application in the treatment of low concentration effluents (Jewell, 1987; Sanz and Fdz.-Polanco, 1990; Lettinga et al., 1993; Vieira et al., 1994; Kato, 1994). The principal application of anaerobic digestion in urban wastewater treatment consists of the utilisation of single-step methanogenic digesters for organic load removal (Ruiz et al, 1998). In the case of urban waste effluents, some authors have reported 30 to 60% reductions in operating costs as a result of introducing one or more anaerobic steps in treatment systems (Schelinkhout, 1993; Alaerts et al., 1993; Wang, 1994). However, further research on the process and better technological development are necessary to consolidate this technology.

Another option is the separation of phases, in which wastewater undergoes a pre- hydrolysis–acidification step before anaerobic digestion. Some advantages of the direct hydrolytic pretreatment of domestic wastewaters are the following (Wang, 1994; Gonçalves et al, 1994):

- removes an elevated percentage of SS, substituting the primary settler at a similar HRT
- stabilises the sludge, totally or partially
- increases the biodegradability of the remaining COD
- favours the subsequent biological elimination of nutrients (N, P)
- avoids or reduces bulking in the activated sludge process.

To achieve the separation of phases, raw wastewater is fed into an upflow sludge bed reactor, in which the HRT is sufficiently reduced, thereby avoiding the conversion of certain organic fractions into methane. The SS settle in the digester where they remain for a longer period of time than the liquid (SRT, higher than HRT), forming a sludge bed, where some soluble substances are also adsorbed. In this way, both the particulate and the soluble organic matter that are retained can undergo solubilisation and fermentation.

The influence of different variables, such as wastewater characteristics, type of digester, mixing mechanism, HRT and SRT on this process is not yet well understood. The objective of this study was to determine the conversion during the hydrolytic pretreatment of actual urban wastewater, in a system operating at an elevated SRT and at different HRTs.

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TABLE 1   Operation periods of the hydrolytic digester and proposed objectives									
Phase	Period	Days	Duration (d)	HRT (h)	Observations				
Phase I		0			Start up				
Start-up	Ia	1-15	15	4.5	Recirculation optimisation and				
-	Ib	16-34	19	3.7	sludge bed stabilisation				
	Ic	35-49	15						
Phase II									
Operation at	IIa	50-85	36	4.4	Steady state				
4.4 h HRT	IIb	86-125	40	4.5	Steady state				
	IIc	126-208	83	-	Pause (no feed supplied)				
	IId	209-225	17	4.5	Restart up				
Phase III	IIIa	226-308	80	3.5	HRT change and adaptation				
Operation at	IIIb	309-343	35	3.4	Adaptation				
3.4 h HRT	IIIc	344-443	100	3.4	Steady state				
	IIId	444-495	52	3.5	Steady state				
	IIIe	496-512	17	3.6	Steady state				
Phase IV Operation at 2.2 h HRT	IV	513-574	62	2.2	Steady state				

# Materials and methods

# **Analytical methods**

Determination of SS, VSS, COD<sub>t</sub> (total), COD<sub>s</sub> (soluble), fats, phosphates and sulphates was carried out according to *Standard Methods* (1985). Total Kjeldahl nitrogen (TKN) was determined by sample digestion with sulphuric acid and selenium reagent, then, using a Kjeldahl apparatus for distillation and titration of samples with hydrochloric acid. Ammonia was determined by

using an ion-selective electrode. Biogas composition was analysed chromatographically (HP 5890 serie II), using a thermal conductivity detector (*Standard Methods*, 1985).

# Wastewater

Urban wastewater was collected directly from a main sewer of the city of A Coruña (Galiza, Spain). Wastewater samples were passed through a 1mm sieve, in order to remove gross solids. The feedstock was kept at 4°C, and renewed twice weekly. Wastewater charac-

teristics varied depending on the time of year, and especially on the amount of rainfall, since samples came from a non-separative sewer system. For this reason, sample collection was avoided on days of heavy rainfall.

# Description of the laboratory digester

A 2*l*-active volume reactor, shown in Fig. 1, was constructed of plexiglass, having a height of 44 cm and an internal diameter of 8 cm. The main body was cylindrical with a conical bottom. Feed supply was carried out by means of a tube leading the influent to the cone centre at the bottom of the reactor, circulating upward through the sludge blanket. The reactor was equipped with an internal recirculation system, for the homogenisation of the sludge blanket and to favour wastewater-sludge contact.

Digester height, and therefore, volume, was changed during the study, in order to vary HRT while maintaining a constant influent flow. The recirculation flow was connected in two different positions, as indicated in Fig. 1:

- above the sludge blanket level (a), recirculating only supernatant
- below the sludge blanket level (b), recirculating sludge.

# Inoculum

The reactor was inoculated with anaerobic sludge originated from a methanogenic digester used to treat effluents from a canned-foods industry at industrial scale. An inoculum volume of 800 ml was used, with

a concentration of 13.6 g SS/ $\ell$  and 11.1 g VSS/ $\ell$ . The methanogenic activity of the inoculum was 0.18 g COD<sub>CH4</sub>/g VSS·d at 20°C.

#### **Operational strategy**

Reactor operation was divided into four periods or phases (Phases *I* to *IV*) which were subdivided into shorter periods (Table 1). The phases differed fundamentally in the HRT applied: Phase *I*, 4.0 h (start-up); Phase *II*, 4.4 h; Phase *III*, 3.4 h and Phase *IV*, 2.2 h. A constant feed flow rate was maintained throughout the operation, thus the different HRT values were obtained by varying digester volume and active height. The active reactor volume in each phase was 2.00  $\ell$  (Phases *I* and *II*), 1.75 L (Phase *III*) and 1.15 L (Phases *I* and *II*), 38.5 cm (Phase *III*) and 25.3 cm (Phase *IV*).

Table 1 presents the operational characteristics in each period, as well as the proposed objectives. In Phase *I*, during periods *Ia-Ic*, the system was started up by selecting an adequate recirculation configuration, in order to achieve correct mixing of digester content, and to develop a sludge with appropriate characteristics for the system. Periods *IIa* and *IIb* can be considered to be steady state, and were used to determine the operational characteristics of the digester at an HRT of 4.4 h. Later, the HRT was gradually reduced,



TIME (days)

Fiaure 2

Influent and effluent characteristics for the hydrolytic digester

operating at each period long enough until achieving steady state operation.

# Results

Figure 2 shows the results of the analyses performed on the reactor influent and effluent, while Fig. 3 presents the corresponding conversions reached, in terms of total and soluble COD removal, SS and VSS, and generation of volatile fatty acids. These same parameters are presented in Table 2 as averages for each period.

# Start-up

The start-up process took place from Days 1 to 49 of operation. During this time different recirculation positions and flow were tested in order to obtain a homogeneous and stable sludge bed.

The upflow velocity produced by the influent flow was 0.9 m/h, constant throughout the different phases. In periods *Ia* and *Ib*, recirculation was maintained connected in position *a*) (Fig. 1) recirculating only supernatant. Operation was initiated with continuous recirculation, producing a total upflow velocity of 2.4 m/h inside the reactor during period *Ia*.

Digester operation was initiated with elevated levels of SS and



Figure 3 COD and SS removal for the hydrolytic digester

TIME	(days)
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TABLE 2   Operational characteristics and conversion during the different periods										
Period HRT	<i>lla</i> 4.4	11b 4.5	<i>lld</i> 4.1	<i>IIIa</i> 3.5	111b 3.4	<i>IIIc</i> 3.4	IIId 3.5	<i>IIIe</i> 3.6	<i>IV</i> 2.2	
Influent										
PH	8.4	8.5	8.11	8.3	8.6	8.3	8.4	8.6	8.3	
COD <sub>t</sub>	648	625	666	525	654	710	599	633	630	
COD <sub>s</sub>	304	288	308	258	322	344	275	257	302	
SS	222	221	244	186	208	245	232	268	242	
VSS	180	199	186	153	173	201	197	232	195	
VFA <sub>cod</sub>	11	21	15	29	6	16	24	23	26	
Alkalinity	0.2	0.3	-	0.3	0.3	0.2	0.4	0.3	0.2	
Effluent										
PH	7.5	7.5	7.4	7.6	7.5	7.4	7.4	7.4	7.2	
COD	375	337	351	325	383	485	425	390	401	
COD	226	199	222	192	238	291	244	227	252	
SS	95	88	70	77	82	93	100	99	88	
VSS	87	83	65	70	71	82	90	93	77	
VFA	31	25	34	48	59	91	103	99	107	
HAc (% VFA <sub>COD</sub> )	97.4	97.8	97.5	93.1	96.0	88.6	86.5	85.6	87.4	
Alkalinity	0.2	0.2	-	0.2	0.3	-	0.3	0.3	0.4	
Conversion										
%COD <sub>t</sub>	42	46	47	37	41	35	33	40	38	
%COD	26	30	28	25	26	18	14	13	16	
%SS	57	60	68	55	60	65	61	64	63	
%VSS	52	56	63	52	59	62	59	61	62	
%VFA COD (i/i)	1.8	3.2	2.4	4.2	1.0	3.1	3.6	3.5	4.4	
%VFA <sub>COD</sub> /COD (e/i)	4.6	4.1	5.1	9.3	9.2	13.2	17.3	15.6	18.4	

Concentrations in mg/ $\ell$ , except pH. Alkalinity is expressed as mg CaCO<sub>3</sub>/ $\ell$ . HRT in h. Conversion is given as percentage removed ((influent-effluent)/influent), except in the case of VFA, where (i/i) indicates influent/influent and (e/i) indicates effluent/influent. VSS removal, 68 and 67%, respectively. Furthermore, COD<sub>t</sub> and COD<sub>s</sub> removal (53 and 44%) were higher than expected. This indicated that during the first days of operation the reactor clearly acted as a settling system and in addition, methanogenic bacteria introduced by the inoculum removed soluble COD. However, this removal was reduced progressively in the first 15 d of operation at high upflow velocities. During period *Ia*, the sludge bed became compact and the formation of preferential pathways was observed, which explains the sudden fall in efficiency upon restricting wastewater-biomass contact.

In view of this situation, the upflow velocity was reduced. From period *Ia* onwards, the recirculation was performed intermittently for 15 min per 75-min cycle. Thus, the resulting upflow velocity was 0.9 m/h when the recirculation was disconnected, and 2.4 m/h during the short recirculation periods. A slight recovery was observed in the efficiency of SS retention (from Day 15 onwards, Period *Ib*), but was insufficient for water-sludge contact improvement: the sludge continued to become compact, and the preferential pathways remained. This behaviour suggests that effluent liquid (sludge bed supernatant) recirculation was not sufficient for obtaining a homogeneous sludge bed.

We opted to modify the recirculation position and to recirculate the sludge directly to achieve homogenisation. On Day 34, recirculation was changed from position a) to position b), as shown in Fig. 1, and maintained until the end of the study. Intermittent recirculation (15-min periods for each 75-min cycle) in this new position, resulted in adequate mixing and homogenisation of the sludge bed.

# Steady state operation at 4.4 h HRT

During the operation periods after Day 50, the digester showed adequate behaviour in terms of homogenisation of the sludge bed, and the different conversion parameters were progressively becoming stabilised. Thus, Periods *IIa* and *IIb* could be considered at steady state as far as the different properties of the system are concerned, but a slight variation in the total amount of sludge in the digester during these periods was observed. This implies a variation in SRT.

The results obtained in Periods *IIa* and *IIb* are representative of digester operation at an HRT of 4.4 h. These results (Table 2) indicate good SS retention, and reflect that the system acted as a hydrolysis-acidification reactor, in which significant removal of easily biodegradable soluble organic matter still exists (probably due to the conjunction of the different biochemical processes taking place, among them conversion to methane). Thus, VFA concentration was still reduced, reaching less than 5% of influent COD.

#### Change from 4.4 to 3.4 h HRT

Phase *III* was initiated on Day 226 with operation at an HRT of 3.4 h. Periods *IIIa* and *IIIb* correspond to the transition to the new operational conditions. The percentage of soluble COD removal was slightly reduced during *IIIa* and *IIIb* as compared to previous periods, decreasing more abruptly in periods *IIIc*, *IIId* and *IIIe*, corresponding to steady state operation at an HRT of 3.4 h. Therefore, HRT influence on net accumulation of effluent VFA is apparent immediately. Effluent VFA<sub>COD</sub> percentage, over total influent COD, changed from 5 to 9% from Period *IId* to *IIIa*. During Period *IIIb*, the situation was very similar. Moreover, acid accumulation increased even more in the periods to follow (*IIIc-IIIe*).

#### Steady state operation at 3.4 h HRT

As indicated, Periods *IIIc*, *IIId* and *IIIe* could be considered at an HRT of 3.4 h as operating at steady state. Compared to operation at an HRT of 4.4 h, clearly an improvement in the hydrolytic-acidogenic behaviour of the reactor was observed, acidification increased until a range of 13 to 17% for the VFA<sub>COD</sub>/COD<sub>1</sub> effluent/ influent ratio (Table 2) was reached, whereas soluble COD removal was below 20%, and suspended solids elimination remained above 60%.

During these periods, the acetic acid contribution to VFA, on a COD basis, fell from values above 90% to within 86 to 88% (Table 2).

#### Operation at an HRT of 2.2 h

On Day 512, HRT was reduced from 3.4 to 2.2 h to study the digester behaviour at an even shorter HRT (Phase *IV*). The system responded positively, maintaining an elevated SS removal percentage (63%) and reaching VFA concentrations of about 115 mg COD/ $\ell$ . This suggests that close to 20% of total influent COD is found as VFA in the effluent.

#### Discussion

Previous studies that could be used as a reference to compare the results of the work presented here are scarce. Gonçalves et al. (1994) obtained elevated SS elimination levels (above 69%) at upflow velocities of below 1.5 m/h, and optimum acidification results at an HRT of 2.8 h and at a temperature of 20°C, with a fermentation rate of 0.17 mg HAc/mg influent COD<sub>t</sub>. About 60% of the VFA produced is a result of soluble fraction fermentation, the remaining 40% corresponding to solubilisation of insoluble organic material (VSS). In the above-mentioned study, acetic acid always reached levels of above 80% of COD corresponding to VFA, whereas COD elimination was below 25%, and COD increased.

In other work, Wang (1994) achieved SS elimination levels of 83% and COD<sub>t</sub> elimination levels of 43% at an HRT of 2.5 h. Similarly, Wang evaluated the influence of influent COD levels on efficiency (%COD eliminated) in a hydrolytic upflow sludge blanket (HUSB) anaerobic reactor, observing that the system absorbed increases in influent COD very well, with little variation in effluent COD.

In the present study the upflow velocity applied was 0.94 m/h, being within the optimum value indicated by Gonçalves et al. (1994), whereas the optimum HRT was the lowest studied, 2.2 h. Under these conditions, SS elimination was 63%, COD, removal 38%, at a time when soluble COD was reduced by 17%. The retention capacity of SS was lower than in the two studies referred to, perhaps due to a lower sludge bed height which would limit its filtration capacity. Generation rate of acids increased as HRT decreased, until reaching 0.18 mg VFA<sub>COD</sub>/mg COD, fed to the digester, very similar to the rate obtained by Gonçalves et al. (1994). The percentage of VFA as acetic acid was always above 85%.

The low acid generation rate at elevated HRT is probably due to the elimination of acids formed by different processes, such as methanogenic respiration, sulphate-reduction or nitrificationdenitrification. Because methane generated in small quantities can leave the system dissolved in the liquid, it was not included in the quantification of biogas generation.

In the studies of Wang (1994), as well as in the present study, the hydrolytic digester was inoculated with digested primary sludge. In both cases, canals and preferential pathways were observed in the sludge bed during start-up. Wang resolved this problem by introducing mechanical mixing, while in this study, water to sludge contact was improved by recirculating the sludge instead of recirculating the supernatant. Upon visual observation, the appearances of the inoculated sludge and of the sludge developed in the hydrolytic digester were clearly different. Perhaps, the elevated mineralisation of the inoculum employed, and its elevated density, aided the initial compactness of the sludge blanket. In the present study, these problems did not reappear during the remainder of the experiment. This problem could have been resolved from the beginning by recirculating the actual sludge, or, perhaps, by starting-up without inoculum, and allowing it to develop from influent solids retained in the digester.

The results obtained confirm the importance of direct hydrolytic pretreatment of domestic wastewaters, this being reflected by elevated SS retention and removal, and by increased biodegradability of remaining COD. Thus, anaerobic hydrolysis of domestic wastewater is of great interest for the secondary treatment using the anaerobic pathway (two-stage digestion), as well as for the aerobic pathway with its multiple variations. Furthermore, a greater proportion of easily biodegradable COD favours phosphorus elimination, 0.1 mg P/mgVFA<sub>COD</sub> (Henze et al., 1995), such that a biological phosphorus removal post-treatment could potentially eliminate all the phosphorus contained in wastewaters.

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