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# Semi-continuous anaerobic digestion of a food industry wastewater in an anaerobic filter

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#### Abstract

The experimental results of semi-continuous tests of anaerobic digestion of confectionery industry wastewater, carried out at different residence times and organic loads in an upflow anaerobic filter, are presented and discussed. Giving COD removals higher than 80% under the whole range of conditions tested, the anaerobic filter demonstrated not only a great ability of biomass to adapt itself to a new carbon source but also an excellent capability to deal with organic load fluctuations and to utilise dilute feeds. Sampling at different filter heights demonstrated that the biogas development ensured mixing within the filter and that most of the organic substances were utilised at the bottom of the reactor, especially when very dilute wastewater was fed. The results of this work could be taken as a starting basis for scaling-up the process to the industrial scale. © 1999 Elsevier Science Ltd. All rights reserved.

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# 1. Introduction

Anaerobic processes can favourably compete with the traditional aerobic processes for the treatment of food industry wastewater, provided that the wastewater from the industrial activity is sufficiently concentrated, available at relatively high temperature (Sixt and Sahm, 1987), and characterised by high biodegradability (Mata-Alvarez et al., 1992). In those cases where the effluents are quite dilute, with CODs from 2 up to 4 times higher than those usually present in domestic wastewater, the aerobic processes can be too expensive and then the anaerobic digestion can constitute an interesting alternative.

The present case study deals with the effluent coming from a Portuguese confectionery factory, with average polluting power ranging from 0.63 to 2.62 g-COD/l. The existing completely-mixed digester is not able to biodegrade it effectively because the factory effluent is quite dilute and the residence time too short (2.0 d). As a consequence of this, the specific growth rate of biomass is lower than the dilution rate, thus preventing the development of a stable and abundant methanogenic population. For this reason, the reactor needs to be modified so that the anaerobic biomass can adequately be retained independently of the wastewater flowrate.

Among the possible alternatives, one of the most attractive would be the adoption of the fluidised-bed reactor (Hickey and Owens, 1981). Excellent results were obtained both at bench- and pilot-scales for the anaerobic digestion of dilute (Switzenbaum and Jewell, 1978) and concentrated (Converti et al., 1993) domestic sewage, of effluents from washing operations of industrial wine production (Converti et al., 1990), and of whey (Boening and Larsen, 1982). Methane yields always exceeding 85% of the maximum theoretical value were achieved in all those cases.

Since, however, industrial applications of this technology appear to be too complicated, a much simpler solution is the anaerobic filter. In this reactor configuration, anaerobic bacteria grow abundantly attached to an inert support and form aggregates in the shape of flocs. These are retained and concentrated in the reactor, thus allowing a biomass retention time longer than the hydraulic residence time. The convective motion originated by the biogas production ensures the agitation of the digesting mass. This type of reactor has already been utilised with success for the anaerobic digestion of different effluents in the presence of different supports

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(Bonastre and Paris, 1989; Marques et al., 1986–1988; Oliveira Marques et al., 1989; Solisio and Del Borghi, 1987; Solisio et al., 1987).

In order to obtain the figures necessary to modify the existing well-mixed reactor into an anaerobic filter, the experimental results of anaerobic digestion in a benchscale version of this equipment are presented and discussed. To this purpose, this food industry effluent has been digested at different loads and residence times.

## 2. Methods

## 2.1. Feed preparation

Several batches of effluents from the industrial installation were collected before entering the existing treatment plant and transferred into storage vessels with volume of 15 l. In order to have a constant composition of the feed and to stop its spontaneous degradation, the batches collected during different 8 day-periods were mixed, stored in a room refrigerated at 4°C, and then used as feed for the bench-scale equipment.

The average compositions of these feeds (Table 1), which were analysed in quadruplicate before starting each trial at a given residence time, show that they were relatively dilute, scarcely stabilised, mainly soluble, quite biodegradable, and acidic, with the only exception being the last sample. Total solids and COD varied within the ranges 1.10–2.30 g/l and 0.53–2.62 g/l, respectively, with the lowest concentrations being detected in the last feeds due to some reductions of activity in the factory.

## 2.2. Experimental set-up and digestion conditions

The upflow laboratory-scale column used for the anaerobic digestion tests was described in detail in a previous paper (Di Berardino et al., 1997). The inert support was PVC tubes, which were cut to give 25–30 mm-long pieces. In this way, the size of the filling material to be used in the industrial-scale plant (about 6 cm

in diameter) was simulated. The reactor, whose working volume was regulated at about 10 l, was surrounded by a water jacket to regulate the temperature at the operating value ( $35^{\circ}$ C). It was also provided with 4 holes for sampling at different heights from the bottom (25, 50, 75, and 100 cm).

The column, filled with two separate layers, each of 0.2 m height, was fed semi-continuously by means of a peristaltic pump. The feed was left at room temperature for about 16 h and then neutralised with a solution of 15% NaOH before use. Biogas production was followed by means of a wet gas meter provided with a totalizer.

## 2.3. Inoculum and start-up

To accelerate the start-up operation preceding the pseudo-steady-state conditions, a stable population of bacterial flocs from a bench-scale anaerobic filter was used as inoculum. This anaerobic population had developed within the bed during a period of two years when the filter had previously been employed for the treatment of milk-industry wastewater (Oliveira Marques et al., 1989).

#### 2.4. Analytical procedures

The course of digestion was followed by daily determining in both the feed and the effluent samples the contents of total solids (TS), volatile solids (VS), total suspended solids (TSS), volatile suspended solids (VSS), chemical oxygen demand (COD), and volatile fatty acids (VFA), according to Standard Methods (APHA, 1985).

The methane content of the biogas at room temperature and pressure was daily determined by gas chromatography, as explained in more detail in a previous work (Di Berardino et al., 1997).

Following the experience of previous studies on anaerobic digestion, waiting for at least five days of relatively stable values of specific biogas production at a given average residence time was the criterion used to get an indication of steady-state achievement.

Table 1

Average compositions of different effluents collected at various times from the real-scale system

	No. of sample							
	1	2	3	4	5	6	7	
рН	6.2	5.4	5.3	4.9	4.0		7.0	
TS (g/l)	2.30	1.80	1.90		2.05	1.10	1.20	
VS (g/l)	1.44	1.13	1.19		1.13	0.43	0.59	
TSS (g/l)		0.65	0.38			0.19	0.26	
VSS (g/l)		0.56	0.35			0.17	0.19	
COD <sub>tot</sub> (g/l)	2.52	2.50	2.32	2.62	2.09	1.53	0.53	
COD <sub>sol</sub> (g/l)		0.87	1.54					

TS = Total solids; VS = Volatile solids; TSS = Total suspended solids; VSS = Volatile suspended solids;  $COD_{tot} = Total$  COD concentration;  $COD_{sol} = Soluble$  COD concentration. Each sample, which was analysed in quadruplicate, consisted of a mixture of effluents from the industrial plant collected during different 8 day-periods.

## 3. Results and discussion

## 3.1. Experimental schedule

The whole experimental programme was subdivided into two separate phases. When the reactor was initially fed after a two-month stop, it rapidly began to work regularly. After only 5 days, it released a well purified effluent and produced biogas in proportion to the organic load. At the start of this first phase, which lasted about 35 days, the filter was semi-continuously fed at a relatively high average hydraulic residence time (5 days), corresponding to an average flow-rate of 2.0 l/d. Programmed variations of the pump capacity allowed the flow rate to vary between 0.85 and 3.85 l/d, thus simulating the organic load oscillations observed in the realscale system (Fig. 1, curve (b)). During the second phase of the experimental work, in order to study a wide range of different conditions, the hydraulic residence time was



Fig. 1. Variations of (a) the production of biogas and (b) the organic load during the whole experimental programme.

Table 2

Trial conditions and steady-state results of the digestion of wastewater in a bench-scale anaerobic filter

Trial conditions								
No. of sample	1	2	3a	3b	4	5	6	7
COD of the feed (g-COD/l)	2.52	2.50	2.32	2.32	2.62	2.09	1.53	0.53
Residence time (h)	133	83	51	53	51	44	35	31
Organic load (g-COD/l.d)	0.45	0.72	1.09	1.05	1.23	1.14	1.05	0.41
Trial duration (d)	14	24	12	6	18 a	22	22 <sup>b</sup>	10
Experimental results								
Effluent COD (g-COD/l)	0.19	0.20	0.28	0.23	0.44	0.21	0.18	0.097
COD removal (%)	92.5	92.1	87.8	90.0	83.2	89.9	88.4	81.7
Biogas production (1/d)	0.99	1.34	3.56	4.00	3.85	3.98	2.66	1.84
Specific biogas production (l/g-	0.24	0.20	0.37	0.42	0.38	0.39	0.29	0.55
COD <sub>removed</sub> )								
Methane content of biogas (%)	85.9	87.3	86.1	86.1	84.3	86.5	89.9	

Experimental results refer to mean data of samples daily collected along the entire duration of each trial at a given average residence time.

<sup>a</sup> Not including 10 final days of feeding stoppage.

<sup>b</sup> Not including 6 days of feeding stoppage between days 114 and 119.

gradually lowered to 30 h, corresponding to a flow rate of about 8.0 l/d.

In two different periods of the experimental schedule, starting from days 74 and 113, respectively, the reactor feeding was stopped for 10 and 6 days, in order to simulate undesired occasional production stoppages of the real-scale plant.

## 3.2. Process efficiency and effluent characteristics

Table 2 summarises the conditions tested and the main results obtained under pseudo-steady-state conditions during the digestion of this food industry wastewater in the bench-scale anaerobic filter. Since the samples were taken daily, the number of data used for each mean value is coincident with the number of days each trial lasted. From these mean values and those shown in Fig. 2, it is evident that the efficiency of organic matter removal was very high at all the tested values of the organic load. In fact, not even when very dilute feeds were used did the efficiency fall below 80%, while a maximum value higher than 92% was achieved.

The COD concentration of the treated effluent never exceeded 0.30 g/l, with the only exception being the test carried out at the highest organic load. Besides the typical performance worsening at high organic load, an instantaneous discharge of organic matter during this sampling may have taken place. As shown in Fig. 3, the pH never decreased below 7.2, with a peak of 8.4, which is indicative of a complete biodegradation of the organic acids, as it was confirmed by periodic analyses of VFA.

#### 3.3. Biogas production and characterisation

The methane content of the biogas and the specific biogas production are plotted versus the organic load in Figs. 2 and 4, respectively.



Fig. 2. Influence of the organic load on ( $\blacksquare$ ) the COD removal yield and ( $\Delta$ ) the methane content of biogas.



Fig. 3. pH behaviour during the whole experimental programme.



Fig. 4. Specific biogas production ( $\bullet$ ) and COD concentration in the effluent ( $\bigcirc$ ) versus the organic load.

Biogas production fluctuated according to the organic load (Fig. 1), thus showing conditions of great stability for the system. The very low biogas production observed when the reactor feeding was stopped could have been the result of the final degradation of the organic substances previously introduced into the reactor. The lowest values of the specific biogas production were obtained at the highest residence times (83 and 133 h) corresponding to organic loads of 0.72 and 0.45 g-COD/l.d, respectively. This result could have been due to one or more of the following reasons: (a) accumulation of organic matter within the filter because of imperfect mixing; (b) consumption of organic matter for maintenance instead of cell growth; (c) lack of process stability. At hydraulic residence times lower than 51 h, on the contrary, this process parameter achieved values (about 0.40 l/g-COD<sub>removed</sub>) close to the maximum theoretical methane production (0.35 l-CH<sub>4</sub>/g-COD<sub>removed</sub>), thus suggesting a complete conversion of biodegradable substances into biogas.

Methane content of the biogas was very high (84.3–89.9%) under all conditions tested.

## 3.4. Activity distribution along the anaerobic filter

The microbial activity along the bed was studied by means of three series of TSS, VSS, and soluble COD determinations on samples withdrawn at 4 different filter heights. Each series was characterised by a different soluble COD concentration in the feed (Table 3).

From these values on the whole it is evident that, at the hydraulic residence times tested in this part of the study (30–51 h), most of the organic matter degradation took place in the first part of the filter bed (Fig. 5). In particular, when the most concentrated feed was used, 85.5% of the soluble organic substances was reduced within the first 25 cm from the bottom. A satisfactory degree of reduction of the organic matter was also observed at the remaining three lateral sampling

Table 3

The concentrations of soluble COD and of suspended solids at different filter heights

Sample port	Dimensionless filter height	TSS (g/l)	VSS (g/l)	COD <sub>sol</sub> (g/l)			
Soluble COD of the feed: 1.59 g/l							
4	1.00	0.125	0.105	0.125			
3	0.75	0.136	0.122	0.129			
2	0.50	0.278	0.240	0.181			
1	0.25	0.150	0.140	0.232			
Soluble COD of the feed: 1.33 g/l							
4	1.00	0.075	0.075	0.098			
3	0.75	0.065	0.065	0.088			
2	0.50	0.132	0.128	0.122			
1	0.25	0.115	0.110	0.146			
Soluble COD of the feed: 0.53 g/l							
4	1.00	0.015	0.015	0.057			
3	0.75	0.015	0.015	0.052			
2	0.50	0.060	0.050	0.057			
1	0.25	2.145	1.615	0.057			

TSS = Total suspended solids; VSS = Volatile suspended solids;  $COD_{sol} = Soluble COD$  concentration.



Fig. 5. Distribution of soluble COD concentration along the filter height. Soluble COD of the feed (g-COD<sub>sol</sub>/l): ( $\bullet$ ) 1.59; ( $\Delta$ ) 1.33; ( $\Box$ ) 0.53.

ports with the two most concentrated feeds. On the other hand, when using the less concentrated effluent, the soluble COD kept at very low values (0.052–0.057 g-COD/l) with negligible dependence on the filter height, which means that the organic matter was nearly totally degraded before reaching the first sampling port.

The same type of organic matter distribution inside the reactor, which strongly depended on the feed concentration, has been observed for quite different biological processes, such as the treatment of gaseous streams in a biofilter (Zilli et al., 1996). In all cases, it was possible to propose zero-order or first-order degradation kinetics (Zilli et al., 1992, 1996). Mass velocity distribution studies were also carried out, which suggested that the diffusion of substrate from the bulk to the biofilm could be the limiting step in most fixed-bed processes (Converti et al., 1996, 1997).

The profiles of TSS and VSS evidenced in Table 3 demonstrate, for the two most concentrated feeds, the existence of some agitation inside the filter, probably created by the remarkable biogas production which re-suspended the solid matter near the second port. A fraction of these solids rose due to biogas development, whereas another fraction went down because of the degasification provoked by the impact with the support. Anaerobic bacteria, as suggested by nearly coincident values of VSS and TSS, mainly constituted this material. In the case of the most diluted feed, due to its low organic content, the slow production of biogas was only able to expand the sludge bed existing at the bottom. This expansion, which reached the level of the first sampling port, was responsible for the high suspended solid concentration detected in this region.

Biogas production fluctuations (results not shown) were quite small compared with those detected for the

other parameters. Redox potential and pH assessments revealed favourable average values for the process, namely -268 mV and 6.94, while volatile acids kept at values always lower than the detection limit of the instrumentation, thus suggesting their complete consumption at the bottom of the reactor.

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