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High rate anaerobic thermophilic technologies for distillery wastewater treatment

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Abstract In this paper, performance of two high rate technologies, upflow anaerobic fixed-film reactor and fluidized bed laboratory-scale, treating distillery wastewater (wine vinasses) at anaerobic thermophilic conditions have been compared. The results obtained show that the stationary packed bed, with a corrugated plastic support, operated under stable conditions at organic loading rates (OLR_0) around 20 kgCOD/m^3/d, gives maximal total CODr of 76% at OLR0 of 6.29 kgCOD/m^3/d; the fluidized bed reactor, operated on open pore sintered-glass media, gives total CODr of 96% at OLR_0 of 5.88 kgCOD/m^3/d. The anaerobic fluidized bed technology is more effective than the upflow anaerobic fixed-film technology due, fundamentally, to this technology favouring the transport of microbial cells from the bulk to the surface and enhancing the contact between the microorganism-substrate phases. In this sense, the stationary packed bed technology is adequate for the treatment of easily biodegradable wastewater, or for the cases where elevated percentages of COD_r removal are not required, while the fluidized bed technology is especially suitable for treatment of hazardous wastes with recalcitrant compositions.
Keywords Anaerobic digestion; fixed film reactor; fluidized bed reactor; thermophilic; support media; wine vinasses

Introduction

The anaerobic treatment of industrial wastewater has a number of potential benefits, including low energy consumption, low excess sludge production, enclosure of odours and aerosols. High rate anaerobic digesters which retain biomass also have a high treatment capacity and, hence, low site area requirements. The major process configurations developed for high-rate digesters over the last twenty years have been reviewed by Hickey *et al*. (1991), and are the upflow anaerobic sludge blanket (UASB), upflow and downflow stationary packed beds, and fluidized and expanded beds.

The loading capacity and the subsequent biogas production rate and wastewater depollution yield are dictated by the amount of active biomass able to grow on the biodegradable fraction. Hence, the biomass retention capacity of a reactor, the specific sludge activity, the substrate biodegradability and concentration, and the daily availability of the wastewater will define the performance of the process. Furthermore, a suitable fluid flow pattern provides adequate distribution of the feed to the microorganisms, enhances the contact between biomass-substrate and, therefore, reduces the limitations by substrate transfer (Tyagi, 1990).

The main objective of this paper is to compare the performance of two high rate technologies at laboratory scale, anaerobic filter and anaerobic fluidized bed, treating distillery wastewater (wine vinasses) at thermophilic conditions.

Methods

The experimental protocol was designed to examine the effect of organic loading rate on the efficiency of UAFF (up-flow anaerobic fixed-film reactor) and AFB (anaerobic fluidized bed reactor) in anaerobic thermophilic treatment of vinasses of wine. This study was conducted at laboratory scale.

Feed solutions

Distillery wastewater proceeding of an ethanol producing wine-distillery plant placed in Tomelloso (Ciudad Real, Spain) was used. In general, vinasses show an adequate relationship between the different macro- and micro-nutrients with a favourable COD/N/P ratio suitable for microbiological treatment. The vinasses were transported and maintained at 4°C before their utilisation. This feed was diluted with tap water to attain the required feed chemical oxygen demand (COD) concentration to be used in this experiment (around 15 $kgCOD/m^3$) and was supplemented with sodium hydroxide to maintain a neutral pH.

Vinasses biodegradation bath experiments (Pérez, 1995) indicated that this was a complex medium formed by two substrates of different nature and biodegradability: S_1, easily biodegradable substrate fraction (85% of the total), and S_2, recalcitrant substrate fraction.

Experimental system

The reactors used in this research are anaerobic fluidized bed (AFB) and upflow anaerobic fixed-film (UAFF), as can be seen in Figure 1.

Fluidized bed reactor

A transparent Plexiglas column with a cross-section of 5.11 cm^2 and length of 170 cm was used. Its bottom was moulded into a conical shape to promote uniform fluidization of media and bioparticles (that is, biofilm coated media). Recycle flow was drawn using a centrifugal pump to provide upflow velocities for media and bioparticle fluidization. Such upflow velocities also ensured that completely mixed conditions were maintained in the liquid phase (Pérez, 1995; Pérez *et al.*, 1999; 2001). Recycle flow was drawn 7 cm below the free liquid surface in the enlarged section to avoid entrapment of gas accumulated in the headspace above and pumped into the bottom assembly. This stream was collected in a settler in order to separate the solid fraction from the liquid stream. The pumping rate was adjusted periodically to account for the varying biomass in order to keep a constant fluidized bed level (25% expansion). Feed was pumped directly from the refrigerator into the recycle lines. The reactor effluent passed throughout a sealed contact chamber connected to an inverted siphon

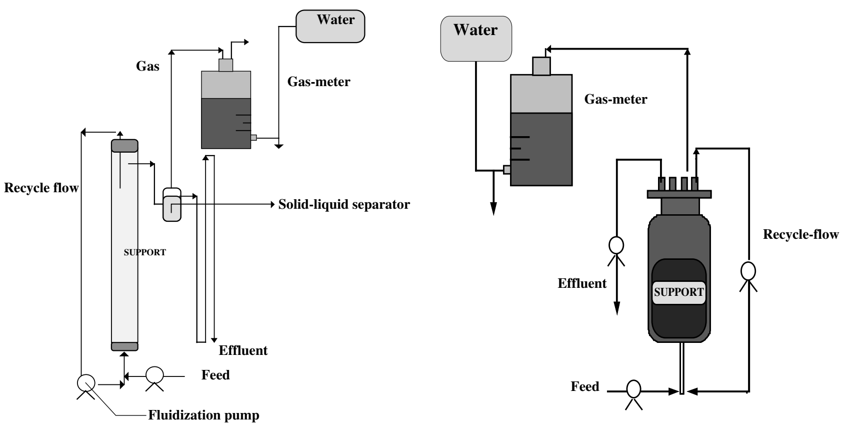

Figure 1 Schematic diagram of the experimental reactor: anaerobic fluidized bed reactor (AFB) and upflow anaerobic fixed-film reactor (UAFF)

to separate the gas form the liquid in the effluent. Reactor temperature was maintained at 55°C with external heating water jackets.

Anaerobic fixed-film reactor. This reactor consists of a vertical cylindrical tank (25 cm length and 10 cm internal diameter). The active liquid volume was 2 L, and the empty volume was 2.4 L. The reactor was filled with 600 randomly distributed media support entities (16 mm length). Reactor temperature was maintained at 55°C and the biogas generated was collected in a gas-meter. Feed was supplied by a peristaltic pump connected to a programmable timer. Effluent recirculation was used to mix and homogenise the liquid in the system.

Gas produced in both reactors was collected in a gas-meter filled with acidified saturated salt solution. A gas sampling valve was installed at the top of the collector to allow direct gas sampling with a syringe. The volume of gas produced in the reactor was directly measured in terms of the volume salt solution displaced from the gas collector.

Characteristics of support media

Corrugated plastic tubes and open-pore sintered glass pearls (SIRAN) were used as the media for cell immobilisation and retention in this research. The main characteristics of both corrugated plastic tubes and SIRAN pearls (Breitenbucher *et al.*, 1990) are shown in Table 1.

Before operating each system, the initial quantities of immobilised biomass (as Volatile Solids, VS) were evaluated. Representative units of both supports were extracted from the reactors and the content of V and Total Solids (TS) were determined. These analyses indicated a global biomass concentration of 66.3 $kgVS_{att}/m^3$ plastic support (Pérez *et al.*, 1996; 2001) and 89.26 $kgVS_{att}/m^3$SIRAN. The last determination was performed in accordance with the protocol described by Shieh *et al.* (1985).

Analytical methods

All analytical determinations were performed according to *Standard Methods* (Clesceri and Greenberg, 1989). For liquid samples, the parameters analysed in both the effluent and the influent were pH, soluble Chemical Oxygen Demand (COD) and both Total and Volatile Suspended Solids (TSS, VSS). For gaseous samples the parameters analysed were the volume of biogas produced at STP and its composition.

Soluble COD was determined by the dichromate reflux methods: the sample was first filtered as in the TSS analysis and the filtrate was used for the COD analysis. TSS and VSS were determined by the glass fibre filter method as described in *Standard Methods*.

Gas production was measured continuously by water displacement. Gas composition (methane and carbon dioxide) was determined using a modified gas chromatography previously described by Nebot *et al.* (1995).

Experimental design

The experimental protocol was designed to examine the effect of organic loading rate (OLR_0) on the efficiency of COD_r in: a) fluidized bed reactor (on SIRAN media), and b)

Table 1 Main characteristics of supports used in this research

Main characteristics	Corrugated plastics	SIRAN
Bulk density (g/L)	1161.4	1832.0
Aparent density (g/L)	73.0	499.3
Porosity (%)	93	55–60
Surface area (m^2/m^3)	450	87,000
Height (cm)	1.6	–
Diameter (cm)	1.6	0.15

upflow anaerobic fixed-film reactor (operating on corrugated plastic); all experiments performed at steady state conditions.

Hydraulic retention time (HRT) remained constant during each stage until reaching the steady-state. The attainment of the steady state was verified after an initial period (over 3 times the HRT) by checking whether the constant effluent characteristic values were the mean of the last measurements (Chemical Oxygen Demand removal (CODr), effluent suspended solids (SST), effluent VSS, pH, gas production and composition of gas (% methane).

Results and discussion

Performance and operation parameters for the duration of the experiments are shown in Table 2. All the results shown are the average values of the last three data.

Performance of fluidized bed (25% expansion on SIRAN)

The assays in this investigation were performed with the expanded bed height in the reactor controlled at 50 cm, yielding a working reactor volume of 0.25 L. Under all the tested conditions, the liquid phase in the reactor was completely mixed. HRT, defined in terms of the expanded bed volume occupied by the bioparticles, was gradually decreased between 2.50 and 0.46 days. The volumetric COD loadings were between 5.9 kgCOD/m^3/d and 32.3 kgCOD/m^3/d.

Removal efficiencies were quite high. At HRT of 0.59 days, a COD_r of 92.5% was obtained at organic loads of 25.3 kgCOD/m^3/d, and at HRT of 0.46 days, COD_r decreased to 81.5% at organic load of 32.3 kgCOD/m^3/d. A posterior applied organic overload produced the instability of the process. Methane gas production averaged 3.3, 7.8 and 9.0 m^3/m^3/d at HRT of 1.34, 0.59 and 0.46 days respectively. The methane yield, as litres of methane produced per grams of COD_r remained constant at 0.33 m^3CH_4/kgCOD_r.

Table 2 Performance and operation parameters of: a) anaerobic fluidized bed reactor; b) anaerobic fixed-film reactor

a) Fluidized bed (25% expansion)

HRT*, d	OLR_0	%COD_r	pH	TSS	VSS	CH_4	CO_2
2.50	5.88	96.57	8.97	0.29	0.14	1.08	–
1.70	8.58	96.40	8.96	0.31	0.14	1.61	–
1.34	11.16	95.50	8.57	0.41	0.21	3.27	2.03
0.85	17.62	95.03	8.52	0.34	0.22	5.30	3.81
0.59	25.26	92.47	8.36	0.33	0.26	7.82	3.16
0.55	27.02	90.47	8.50	0.54	0.46	8.12	7.03
0.52	28.85	89.75	8.27	0.62	0.55	8.22	7.75
0.46	32.31	81.48	7.65	0.70	0.61	9.00	9.04

b) Upflow anaerobic fixed-film reactor

HRT**, d	OLR_0	%COD_r	pH	TSS	VSS	CH_4	CO_2
2.50	6.29	75.55	8.24	1.38	1.09	1.45	0.78
1.28	12.32	61.42	7.52	1.58	1.26	1.96	1.08
1.01	15.66	55.78	7.33	1.65	1.34	2.85	2.75
0.87	18.46	52.14	7.47	1.72	1.55	3.28	3.57
0.83	19.04	49.33	7.44	1.72	1.52	3.13	3.04
0.82	19.56	47.89	7.31	1.69	1.45	3.55	3.70

* Refereed bed volume of 0.208 L

** Refereed bed volume of 2 L

(HRT, as day; Organic load rate, OLR_0, as kgCOD_0/m^3/d; % COD removal; pH; TSS and VSS expressed as kg/m^3; CH_4 and CO_2 as m^3/m^3/d)

Initially, a detachment of immobilised biomass was produced. Subsequently, the effluent SSV concentration increases only slightly for large increases in the hydraulic loading rate (period between 1.34 to 0.46 days). The fluidized bed reactor contained an average between 0.21 and 0.61 $kgVSS/m^3$ at HRT 1.34 and 0.46 days respectively. This is one of the major advantages of the AFB system over a suspended microbial system in that even at very high loading (hydraulic and organic) conditions, washout is not a problem. The pH maintained a range of between 8.57 and 7.65 during all the stable processes. When the HRT decreased to 0.37 days, the pH dropped dramatically down to 5.5. The generation of biogas ceased and the effluent's suspended volatile solids increased from 0.58 $kgVSS/m^3$ to 0.75 $kgVSS/m^3$ in the last stage.

Upflow anaerobic fixed-film reactor on corrugated plastic tubes

Volumetric organic loading was gradually increased between 6.29 and 19.56 $kgCOD/m^3/d$. The hydraulic retention time, defined in terms of the volume occupied by bioparticles (2 L), was between 2.50 and 0.82 days. Soluble COD_r removals were observed to vary from 75.5 to 47.89%. This reactor with non-porous packing showed instability above an OLR_r higher than 20 $kgCOD/m^3/d$. Methane gas production averaged 1.45 and 3.55 $m^3/m^3/d$ at HRT of 2.50 and 0.82 days respectively. The methane yield remained constant at 0.31 $m^3CH_4/kgCOD_r$. The stationary-packed reactor contained an average between 1.09 and 1.52 $kgVSS/m^3$, at HRT 2.50 and 0.83 days respectively. The pH maintained a range of between 8.24 and 7.31 during all the stable processes.

In conclusion, the degradation efficiency is the most important parameter to be considered. In all the OLR_0 ranges studied, the soluble COD_r efficiency of the fluidized technology was greater than the stationary packed bed reactors. Figure 2 shows OLR_r as influenced by organic load rate applied (OLR_0). Maximum organic load rate feed was 32 $kgCOD/m^3/d$ for the fluidized bed reactor and 20 $kgCOD/m^3/d$ for the anaerobic fixed-film reactor. Under these conditions, the organic removal efficiency was 81.5% and 50% respectively.

The organic removal efficiency of the reactors compared to the organic load rate is shown in Figure 3. The maximum organic removal efficiency was 97% and 75% respectively. So, the fluidized bed reactor can degrade the substrate S_2 (recalcitrant substrate) in all stage operations. Other wine distillery waste studies have been reported using other anaerobic processes such as the anaerobic fixed-film reactor (Perez, 1995; Pérez *et al.*, 2001) and the

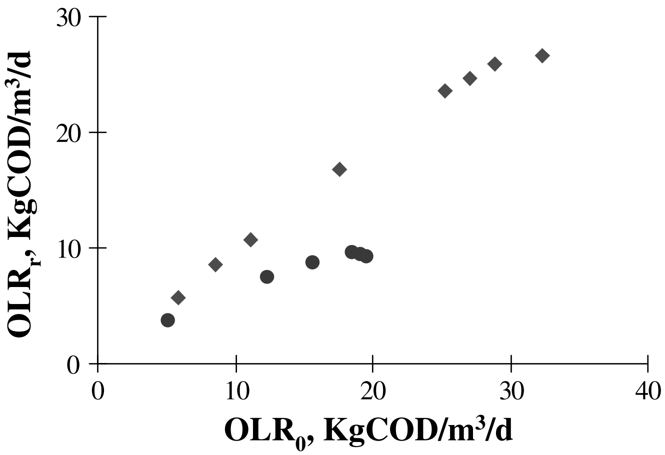

Figure 2 Organic load rate removal (OLR_r) as influenced by organic load rate applied (OLR_0) (◆ fluidized bed on SIRAN; ● upflow anaerobic fixed-film reactor on corrugated plastic tubes)

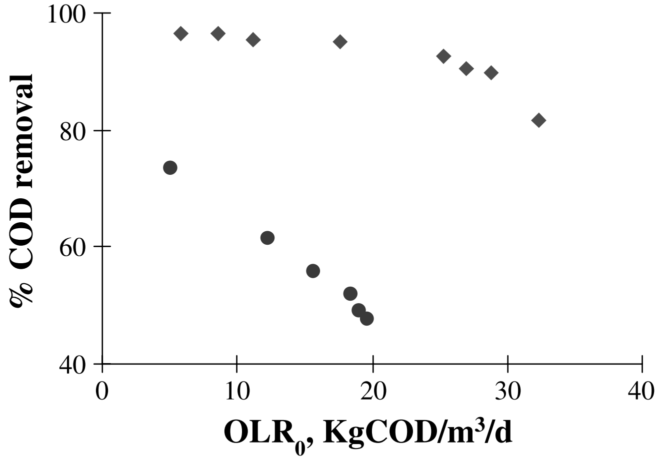

Figure 3 Effect of organic load rate feed on the organic removal efficiency (%COD removal) (◆ fluidized bed on SIRAN; ● upflow anaerobic fixed-film reactor on corrugated plastic tubes)

UASB reactor (Craveiro *et al.*, 1986) with 80% and 83% COD_r at organic loads of 12 and 13.2 kg/m^3/d and HRT of 1.4 and 2.4 days respectively. Thus, fluidized technology enhances the high rate organic operation with high efficiency COD_r.

Figure 4 shows the changes in VSS concentration in the reactors at different OLR_0, with a higher VSS concentration profile in the effluent from the stationary packed reactors than the fluidized process. In all the cases, the biomass concentration increased with the applied OLR_0. Thus, the technology used determines the extent of the detachment. The porous glass medium favours a rapid biomass accumulation and is not susceptible to shear stresses, and the fluidized technology enhances a suitable fluid flow pattern that provides adequate distribution of the feed to the microorganisms, favours the contact between biomass-substrate and, therefore, reduces the limitations by substrate transfer.

Figure 5 shows the methane yield and the methane volumetric rate in the off-gas system having greater stability in the fluidized system at higher OLR_0. In the range between OLR_0 6

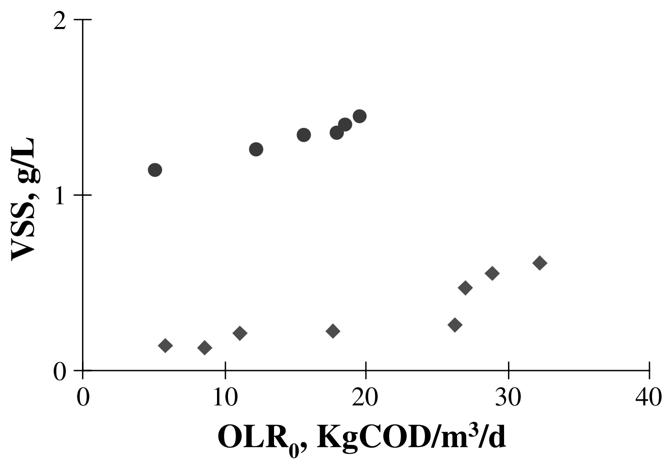

Figure 4 Effect of OLR_0 applied on the VSS (◆ fluidized bed on SIRAN; ● upflow anaerobic fixed-film reactor on corrugated plastic tubes)

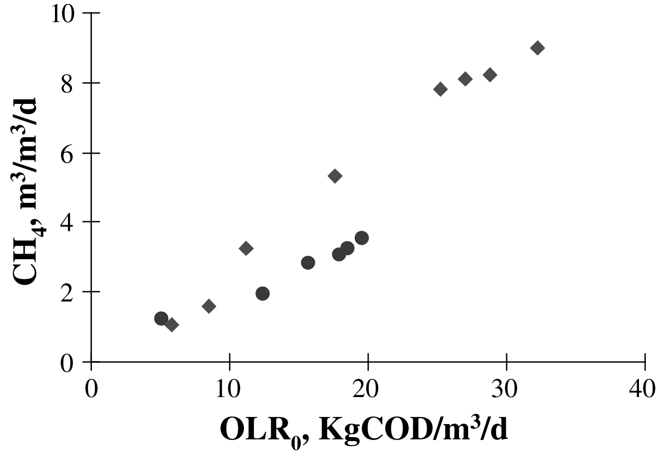

Figure 5 Volumetric methane rate as a function of the OLR_0 applied. (◆ fluidized bed on SIRAN; ● upflow anaerobic fixed-film reactor on corrugated plastic tubes)

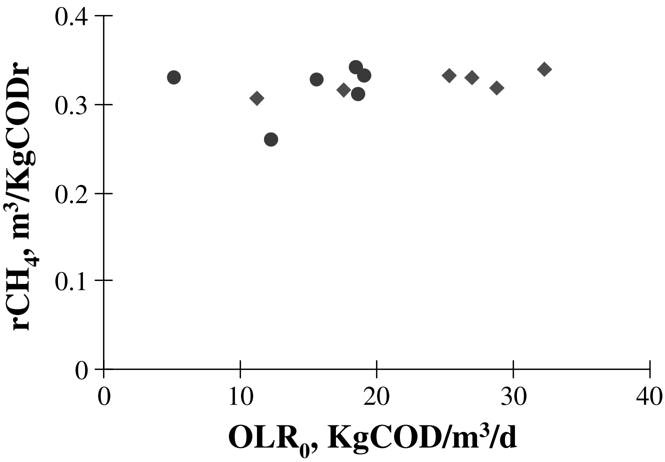

Figure 6 Methane yield as a function of the OLR_0 applied (◆ fluidized bed on SIRAN; ● upflow anaerobic fixed-film reactor on corrugated plastic tubes)

and 20 kgCOD/m^3/d, the fluidized bed produced a much higher methane rate than those stationary systems. The methane yield is similar in both reactors, as can be seen in Figure 6.

All the results obtained confirm that the fluidized technology may give a better performance than an upflow anaerobic fixed-film reactor. Breitenbucher *et al.* (1990) confirmed the ability of the porous media (SIRAN) to retain a large quantity of biomass and demonstrated that using porous sintered glass media increases reactor performance compared to other media. However, corrugated plastic is an adequate carrier for stationary filters to avoid the main problems of these systems: the obstruction of the filter when excessive growth is reached.

Conclusions

The results of this research show that the stationary packed bed, with a corrugated plastic support, operated under stable conditions at OLR_0 around 20 kgCOD/m^3/d, gives maximal total COD_r of 76% at OLR_0 of 6.29 kgCOD/m^3/d; the fluidized bed reactor, operated on open pore sintered-glass media, gives total COD_r of 96% at OLR_0 of 5.88 kgCOD/m^3/d.

As a result, the anaerobic fluidized bed technology is more effective than the upflow anaerobic fixed-film technology due, fundamentally, to this technology favouring the transport of microbial cells from the bulk to the surface and enhancing the contact between the microorganism-substrate phases. At the end of the investigation, attachment studies were carried out and the results showed a heavy biomass attachment in porous media, whereas mainly unattached biomass was retained in the voids of the non-porous media. However, their application considerably increases the costs of operation and maintenance and, also, demands a strict control process.

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