Treatment of distillery vinasse in a high rate anaerobic reactor using low density polyethylene supports

J.V. Thanikal*,, M. Torrijos*, F. Habouzit* and R. Moletta***

*INRA, UR50, Laboratoire de Biotechnologie de l'Environnement, Avenue des Etangs, Narbonne, F-11100, France (E-mail: *torrijos@ensam.inra.fr*; *habouzit@ensam.inra.fr*; *Rene.Moletta@univ-savoie.fr*)

**Kumaraguru College of Technology, Coimbatore 641 006, India (E-mail: *jthanikal@gmail.com*)

Abstract An anaerobic fixed bed reactor, filled with small floating supports of polyethylene material (Bioflow 30) as inert media, was operated for 6 months to treat vinasse (wine residue after distillation). Bioflow 30 has a density of 0.93 and a specific area of 320 m^2/m^3. The experimental results showed that the efficiency of the reactor in removal of soluble COD was very good with a maximum organic loading rate of more than 30 g of COD/L.d and a COD removal efficiency of more than 80%. Bioflow 30 showed a high capability of biomass retention with 4 – 6 g of dried solids per support. Thus, at the end of the experiment, the fixed biomass represented 57 g of solids/L of reactor. The visual observation of the supports and the specific activity (0.54 g COD/g solids.d) of the fixed solids, which remained close to the values obtained with suspended biomass, showed that entrapment was playing an important role in the retention of the biomass inside the reactor. It was then possible to operate the reactor with a very high loading rate as the result of the increase of the solids in the reactor and the maintaining of the specific activity. Bioflow 30 is then an excellent support for use in a high rate anaerobic fixed bed.

Keywords Anaerobic digestion; fixed bed; inert media; wastewater treatment; wine vinasse

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Introduction

An anaerobic fixed bed is composed of a vertical bed, which contains an inert media, which acts as a stationary support for microbial attachment. Two kinds of supports can be used in this type of reactor: well ordered supports and loose supports (Malina and Pohland, 1992). It is possible to maintain a much higher quantity of biomass in the fixed bed reactor than in suspended-growth processes, then allowing the use of high volumetric loading rates, short liquid retention time and good performance stability (Henze and Harremoes, 1983; Malina and Pohland, 1992). Many different materials have been tested for biomass retention in anaerobic fixed bed systems and the performance of these materials appears to be directly related to the ease with which bacteria can become entrapped or attached to the supports. Anaerobic fixed beds often have a loosely attached biofilm on the support, responsible for a significant part of the bacterial activity (Henze and Harremoes, 1983) and it appears that most of the biological activity is often due to the entrapped biomass in the interstitial volume between the supports rather than to the attached biofilm (Young and Dahab, 1983; Hickey *et al.*, 1991). Results from the studies of Young and Dahab (1983) have shown that the ability of the media to entrap the biomass or to prevent its washout from the reactor is more important than the specific surface area of the media.

One of the problems associated with anaerobic fixed beds is the risk of clogging and channeling due to the accumulation of solids in the media, particularly in the case of small loose support. This problem can be quite acute when the reactors are operated at high loading rates or with an effluent containing solids. To reduce the risk of clogging, several solutions are possible among which are the use of a well ordered media with a high percentage of void and a substantial distance between the inner surfaces, or the use

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of a small loose media which can be easily unclogged by fluidization with liquid or by gas re-circulation, to remove the excess solids accumulated in the reactor.

In the present work, studies have been done to investigate the potentially of the use of small loose floating supports as media in an anaerobic fixed bed. The polyethylene support used has a density of 0.93. To prevent any clogging of the reactor, the floating media was intermittently fluidized by an up-flow current of liquid within the reactor created by a submerged pump, kept at the bottom of the reactor. The aim of this work was to study the performance of an anaerobic fixed bed containing this support (minimum hydraulic retention time, maximum loading rate, purification efficiency, etc.) and to measure the quantity of biomass retained on the support at the end of the experiment and its activity.

Materials and methods

Experimental device

The reactor of 23 L working volume used in the study was fabricated out of PVC material and consisted of a tubular section of 190 mm internal diameter and 1,150 mm total height with a conical bottom.

The system was equipped with a water jacket to keep the temperature of the reactor at 35 °C. The reactor was equipped with a substrate feed inlet at the bottom of the reactor and an overflow arrangement was provided such that the effective height of the liquid inside the reactor was maintained at 810 mm. A sampling port was fixed at the bottom of the reactor. A submerged pump (flow rate 480 L/h) was fixed inside the reactor at the bottom to facilitate fluidization of the supports.

The support

The reactor was filled with a polyethylene support, Bioflow 30, manufactured by Rauschert®, for 60% of the volume of the reactor. This trapezoidal support is 29 mm high and measures 35 mm at the bottom and 30 mm at the top. It has a density of 0.93 and a specific surface area of 320 m^2/m^3.

Reactor operating conditions

The reactor was fed with a distillery vinasse (wine residue after distillation) with a total COD varied between 10 and 24 g/L and soluble COD between 10 and 19 g/L. The pH of the feed (4–5.5) was adjusted between 7 and 7.5.

The reactor was inoculated with anaerobic sludge collected from an anaerobic reactor treating distillery vinasse and concentrated to 45 g/L by settling. The volume of sludge added represented 10% of the volume of reactor.

The substrate was fed into the reactor through the inlet at the bottom of the reactor, using a peristaltic pump. The inlet substrate was fed at equal intervals of time, sequentially, as per the designed daily volume. The operation of the pump for fluidization was programmed 15 mm every 3 hours.

Measurements and analysis

Soluble COD, volatile fatty acids, and suspended solids were determined daily through off-line analysis. COD was measured by a colorimetric method (Jirka and Carter, 1975). Volatile fatty acids were measured using a gas chromatograph with a flame ionization detector (GC 8000 Fisons instrument with automatic sampler AS 800). Total and Volatile solids inside the reactor and at the outlet of the reactor were measured by *Standard Methods* (1992). The attached biomass on a support was measured by weight, the oven dried support (100 °C, 24 hours).

Results and discussion

Operation of the reactor

At the beginning of the experiment, the reactor was operated with a high hydraulic retention time (HRT) and a low organic loading rate (OLR). Then, the hydraulic retention time applied to the reactor was regularly decreased and the organic loading rate increased by increasing the volume of vinasse treated. The reactor was operated for 180 days and the total operation period can be divided into three stages as shown in Figure 1: (i) at the first stage (days 0 to 81), the increase in the OLR was slow, the HRT was always more than 3.6 days and the OLR was always less than 5.6 g/L.d; (ii) during the second stage (days 82 to 101), the HRT had to be maintained at a constant high value (7.7 days) due to insufficient availability of vinasse. The OLR was low, i.e. between 1.6 and 2.6 g/L.d; (iii) during the third stage (days 102 to 180), the HRT was rapidly decreased from 7.7 days to a minimum of 0.7 day and the OLR increased from a value of 1.6 to 36 g/L.d.

Reactor performance

During the first 81 days, when the reactor was fed with a low and slowly increasing OLR, soluble COD of the treated effluent remained low with values less than 3.1 g/L (Figure 2). VFA concentration represented always less than 1.6 g COD/L. The slight increase in soluble COD at the end of period 1 (day 60 to 81) is linked to the use of new vinasse, for which the non-biodegradable fraction (1.5 g/L) was higher than that of the previous one (0.55 g/L). During this period, COD removal efficiency was always more than 85%.

During the second stage, OLR remained low and at the end of this period, soluble COD was very low with 0.85 g/L and VFA concentration was nil.

At the third stage, during the rapid increase of the OLR from 1.3 to 36 g COD/L.d in 78 days, the global COD removal efficiency was always good with an average value of 85% and soluble COD at outlet was always less than 5.5 g/L.

For OLR value up to 12.5 g COD/L.d (days 102 to 153), the average values were 1.4 g/L for soluble COD and 0.3 g/L for VFA concentration. Purification efficiency was very good with 89% COD removal on average.

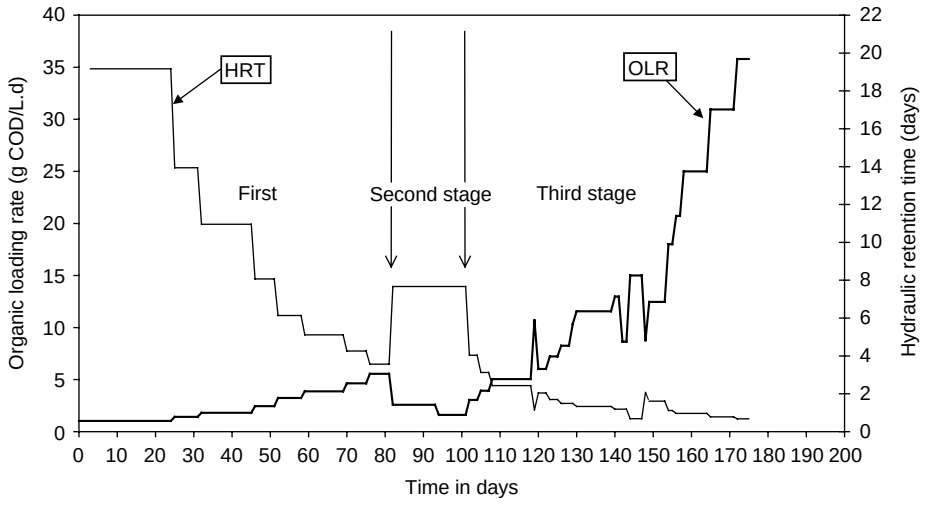

Figure 1 Evolution of hydraulic retention time and organic loading rate with time

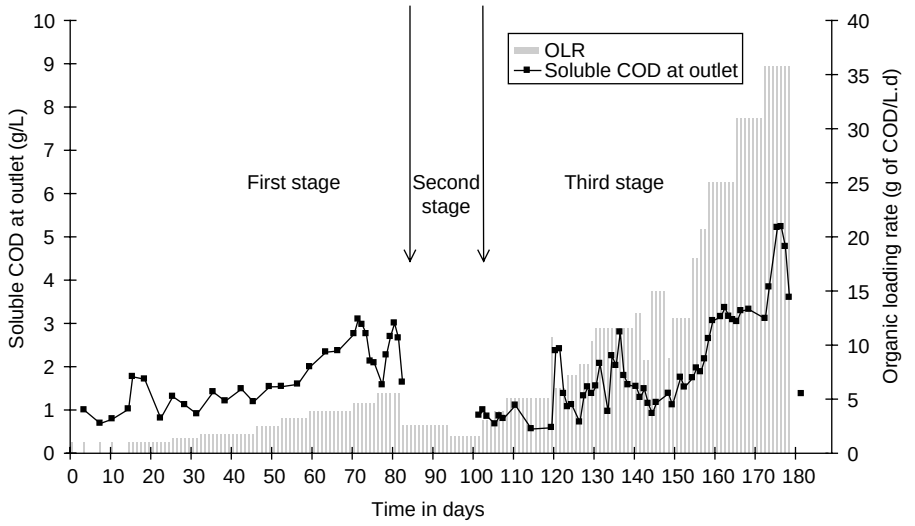

Figure 2 Evolution of the soluble COD at outlet and the organic loading rate with time

For higher OLR and up to 31 g COD/L.d, purification efficiency decreased slightly but was still more than 80% with an average value of 83%. Soluble COD was always less than 3.5 g/L and VFA concentration was 1.15 g COD/L on average.

The most important data obtained during this experiment can be summarized in Figures 3 and 4 which represent the evolution of the purification efficiency with the OLR (Figure 3) or with the HRT (Figure 4). These figures clearly show that, when treating a concentrated effluent such as a distillery vinasse, an anaerobic fixed bed with Bioflow 30 can be operated at high organic loading rates of more than 30 g COD/L.d and low HRT of less than one day with a purification efficiency of more than 80%. It is important to emphasize that the maximum loading rate obtained in this study (>30 g COD/L.d) is quite high for a fixed bed reactor treating distillery vinasse, which shows that Bioflow 30 is an excellent support which can be used in anaerobic digestion. Indeed, an anaerobic

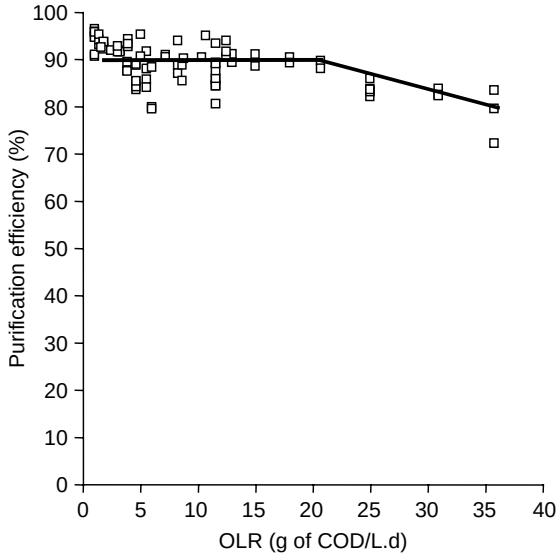

Figure 3 Evolution of the COD removal efficiency with organic loading rate

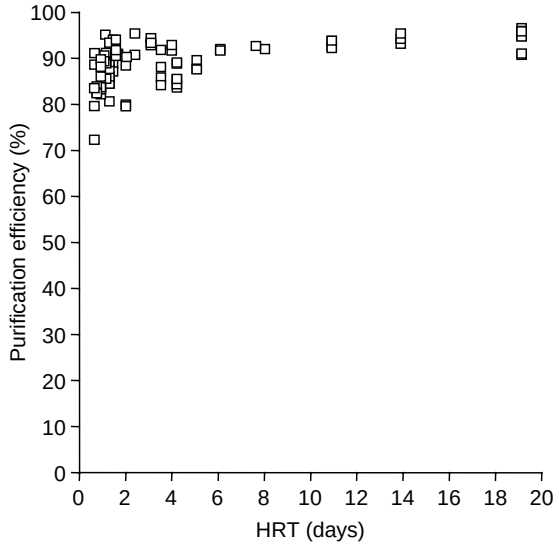

Figure 4 Evolution of the COD removal efficiency with hydraulic retention time

fixed bed containing cloisonyle, which is a well-ordered medium made up of PVC tubes of 102.5 mm in diameter divided up into 14 canals with a specific area of 180 m^2/m^3, and treating a distillery vinasse could only reach OLR around 14 g COD/L.d (Ouichanpagdee *et al.*, 2004). Furthermore, Malina and Pohland (1992) reported that full-scale fixed bed processes have been generally designed for organic loading rates of up to 16 g COD/L.d.

Biomass evolution

The suspended solid (SS) concentration in the reactor was regularly measured to follow the evolution of the biomass in suspension in the reactor (Figure 5).

During the first stage of the experiment (first 81 days), the suspended solid concentration remained high with values between 3.5 and 5 g/L. Suspended solids started to decrease towards the end of the first stage indicating the wash-out of the free biomass.

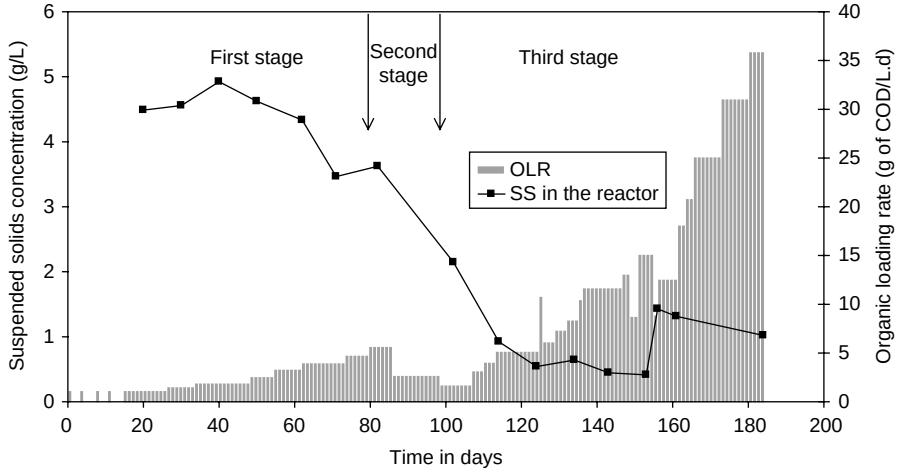

Figure 5 Evolution of the suspended solids concentration at outlet of the reactor and of organic loading rate with time

During the third part of the experiment, SS concentration stabilized at low concentrations with values between 0.4 and 1.5 g/L.

After two months of operation, floating supports were taken from the top of the reactor, in the first 10 cm below the liquid surface. The first sampling of support on day 66 showed that the quantity of solids on the supports was around 2.5 g of solids/support. Between days 66 and 156, fixed biomass increased by 30% with 3.2 g of solids/support on day 156. However, it appeared clear that the quantity of floating supports was decreasing with time and supports were sinking to the bottom of the reactor. On day 156, samples were taken close to the surface and as deep as possible inside the reactor (60–70 cm down the surface). The quantity of solids was 3.2 g on the floating supports and 4.1 g on the supports from deep inside the reactor which represents a difference close to 30%. The distribution of the supports in the reactor did not seem to be homogeneous and the quantity of attached biomass was not constant from one support to another. It was therefore not possible to accurately estimate the quantity of fixed biomass just by weighting a few supports. However, the quantity of solids on the floating supports after 66 days of operation was quite high suggesting a good aptitude of the biomass to attach to the support.

At the end of the experiment, after 180 days of operation, the total quantity of fixed biomass was quantified by weighting all the supports. Supports were taken out of the reactor from top to bottom, in batches of 40 supports for the first five samples and of 50 and 67 supports, respectively, for the two last samplings (Figure 6).

The average attached biomass on the supports was not constant and varied between 3.2 and 5 g of solids/support (Figure 6). For the deeper supports in the reactor, attached biomass was the lowest. The decrease in the attached biomass on the supports close to the bottom of the reactor could be attributed to the detachment of biofilm due to the high velocity of liquid generated near the vicinity of the pump.

The total quantity of attached biomass in the reactor was 1,300 g. The concentration of attached biomass was then 57 g/L and the biomass in suspension concentration was only 1 g/L.

When emptying the reactor, it became clear that the supports at the bottom of the reactor were adhered together and could not be fluidized anymore due to the small

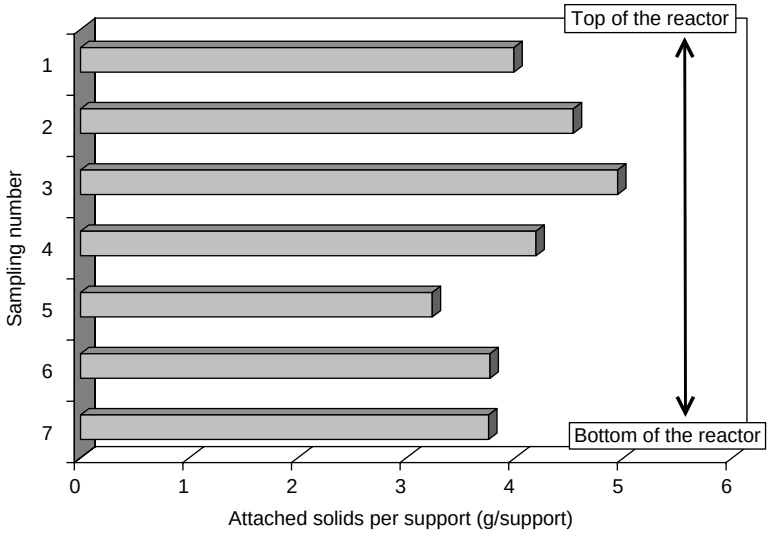

Figure 6 Evolution of the attached solids per support according to the height of the reactor

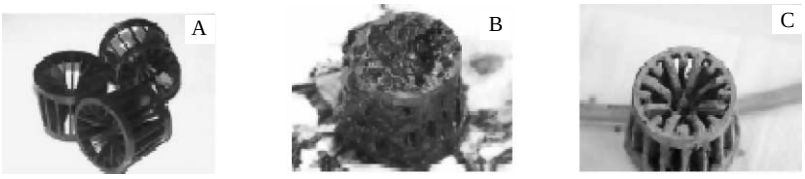

Figure 7 Photographs of biological solids attached to the anaerobic fixed bed media: (A) non-colonized support; (B) colonized support; (C) support after heat drying

diameter of the reactor and to the low flow rate of the mixing pump. In these conditions, the bottom of the reactor behaved like an anaerobic filter with a stationary support.

Visual observation of the media showed a biofilm formation on the surface of the support but there was also biomass entrapped inside the support, filling most of the voids (Figure 7). Similar results were reported by Young and Dahab (1983) for an anaerobic fixed bed filled with cylindrical Pall rings 90 mm tall by 90 mm diameter but with much lower loading rates.

Biomass activity

From the OLR applied at the end of the experiment and the measurement of the total quantity of biomass attached to the supports, it is possible to estimate the specific activity of the fixed biomass. This activity was 0.54 g COD/g of dried solids.d. This value is similar to the specific activity measured by Ruiz (2002) for biomass in suspension treating sugar cane vinasses (0.52 g COD/g of dried solids.d) or molasses vinasses (0.48 g COD/g of dried solids.d). With cloisonyle, Ouichanpagdee *et al.* (2004) found a much lower activity (0.18 g COD/ g dried solids.d) due to the accumulation of mineral solids in the biofilm attached at the surface of the PVC support. Lastly, the specific activity measured in this work is significantly higher than the specific activity reported by Switzenbaum (1983) for an anaerobic fixed bed (0.4 g COD/g.d) but lower than the specific activity of an expanded bed (0.8 g COD/g.d) and in the lower range of the specific activity of granular sludge (Henze and Harremoes, 1983).

The results obtained show that the activity of the biomass kept on the support remains good and have a value quite close to that of a suspended biomass. This suggests that the entrapped biomass may play an important role in the global behavior of the reactor and that the support serves to create a biofilm on its surface but, also to entrap the biomass in its void space, thus preventing it from being washed out of the reactor.

Conclusions

The operation of a fixed bed reactor containing Bioflow 30, a polyethylene support with a density lower than 1 and a specific area of 320 m^2/m^3, demonstrated that Bioflow 30 is a promising support for an application in anaerobic digestion. Indeed, after six months of operation a loading rate of more than 30 g COD/L.d could be applied, while maintaining a COD removal efficiency of more than 80%.

The study of the attached biomass showed that it was possible to fix a high quantity of solids on the support. Indeed, the quantity of biomass in the reactor was increased around 5–6 times compared to a reactor with suspended biomass. The activity of the fixed solids on the supports remained good with a value close to that of suspended solids. It was then possible to operate the reactor with a very high loading rate (more than 30 g COD/L.d) as the result of the increase of the solids in the reactor and the maintaining of the specific activity. The visual observation of the supports and the specific activity of the attached

solids suggest that due to its configuration, the support entraps a lot of solids which play an important role in the overall performance of the reactor.

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