

ENVIRONMENTAL MICROBIOLOGY

Effect of the Feed Frequency on the Performance of Anaerobic Filters

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Key Words: anaerobic digestion, anaerobic filter, feed frequency, feeding systems, distilleries wastewaters Anaerobic filter technology is suitable for the treatment of biodegradable organic wastes: for example, wastewaters from food industries. These wastewaters are not produced in a continuous mode since they are of seasonal nature and hence their production varies considerably during the year. In this work, a study of the performance of anaerobic filters with changing feeding systems was undertaken. A comparison was made between continuous feeding and different semicontinuous modes of feeding (in which the overall volume to be added into the reactors was divided into several doses and each of them were added at a constant interval of time). Filter performance was characterized by determining the different operational variables: depurative efficiency, methane production, biogas composition and volatile acids. From the obtained results, we conclude that the optimum feed frequency range is 24 doses/day or more. The continuously fed system has both greater stability and degradation efficiency.

Introduction

In the last decade, research about the anaerobic digestion of high strength organic wastes has increased and, consequently, a great number of new reactor designs have been developed [1–5]. These type reactors have a special common characteristic in that they include a device to retain the microorganisms responsible for the process in the system. Thus, the reactors are capable of reaching both a high solid retention time and a high degradative efficiency, at a high volumetric flow rate (i.e. greater than required to produce the wash-out of the microorganisms in that system without biomass retention).

Anaerobic filter technology has been shown to be especially suited for the treatment of organic wastes at

an industrial scale [1]. These reactors consist of a stationary media support in which the microorganisms are retained. High specific surface area and high porosity are main characteristics of the support material.

The characterization of the anaerobic filter performance versus organic load added is very important. Several studies [6,7] have been carried out to study the influence of the organic load shocks in the anaerobic filters. The shock can be produced by both the applied volumetric flow rate or the increase in the added organic load [8] — these are called hydraulic shock or organic load shock, respectively. Likewise, a combination of both phenomena is possible.

The importance of the study described here is that, in the industrial operation, variations in the flow rate and the influent organic matter concentration are very 114 E. Nebot et al.

common and for this reason the study of the feed fluctuation influence on the biological treatment is of great interest.

Very few papers studying the effect of feeding frequency on the performance of anaerobic filters have been published. However, the feed for the filter can be added in either a continuous or semicontinuous mode. In the latter case, the overall volume of liquid to be added to the reactor is divided into a number of doses per day.

Although continuous operation mode may increase the stability of the process, the semicontinuous mode presents an advantage in that the operating costs are less. Several authors have shown that the methane production yield is greater in the latter case [9].

The scope of the present work was to study the performance of anaerobic filters when the feeding protocol was modified from a continuous to semicontinuous mode (for different daily frequencies).

Materials and Methods

Anaerobic filters on a laboratory scale were used for this experimental work. Reactors used in this study had been started up previously and fed with waste from wine distilleries (wine vinasses) lacking suspended solids and presenting high organic concentration [10,11]. The feed was supplied only once per day

The reactors were operated at 55°C (optimum of the thermophilic range). On an industrial scale, most of the factories operate in the mesophilic range (35°C); however, several authors have demonstrated that the thermophilic process is more efficient than mesophilic for efficiency depuration [12]. The thermophilic process is especially interesting when the waste is discharged at high temperature, as is the case with vinasses.

Wine vinasses

The main characteristic parameters of the wine vinas-

Table 1. Feed composition (wine vinasses)

| pH | 3.5 |
|--|-------|
| COD (g/L) | 15.0 |
| Dissolved solids (g/L) | 9.05 |
| Volatile dissolved solids (g/L) | 7.20 |
| Suspended solids (g/L) | 0.5 |
| Nitrogen (g N/L) | 0.242 |
| Phosphate (g P ₂ O ₅ /L) | 0.05 |
| Polyphenols (g Ac. Galic/L) | 0.26 |
| | |

COD = chemical oxygen demand.

ses are shown in Table 1. As can be seen, the acidic character implies that they must be neutralized prior their introduction into the reactors. Neutralization was achieved by adding a concentrated NaOH solution. The C/N/P ratio is slightly less than the optimum reported in the literature [13]; however, nutrient addition was not necessary, as can be seen in the subsequent experiments.

Reactors

Figure 1 schematically shows the reactors and the support material. All the experiments were conducted in duplicate cylindrical glass reactors (12 cm internal diameter and 25 cm height), with working volumes of 2.2 L. All the results presented are the average values of the two reactors. The temperature was maintained at 55°C and the biogas generated was collected in gasometers. Feed was supplied by a peristaltic pump; when the operation was semicontinuous, the pump was connected to a programmable timer. Effluent recirculation was used to mix and homogenize the liquid in the system.

The media support used, Flocor R, consisted of corrugated plastic tubes (16 mm diameter and 16 mm length) randomly distributed into the reactor. Its main characteristics were: specific surface: 450 m²/m³ (which was much higher than most of those described in the literature [14]), porosity: 93.71%, density: 1.161 kg/L.

Analytical techniques

All analytical determinations were performed according to standard methods [15]. For liquid samples, the parameters analysed in both the influent and the effluent of the reactors were pH, chemical oxygendemand (COD) and volatile acids. For gaseous samples, the parameters analysed were the volume of biogas produced at STP and its composition. Determination of biogas composition was carried out using gas chromatography separation accomplished using a stainless steel column packed with Carbosieve SII (diameter of 1 and 2 m length) and a thermal conductivity detector (TCD). The injected sample volume was 1 cm³ and operational conditions were as follows: furnace temperature: 7 min at 55°C; ramped at 27°C/min until 150°C and 5 min at 150°C; detector temperature: 225°C; injector temperature: 100°C. The carrier was helium and the flow rate used was 30 mL/ min. A standard gas of (by Carburos Metálicos, S.A.) composition was 4.65% H₂; 5.33% N₂; 69.92% CH₄ and 20.10% CO₂ was used for the calibration of the system.

Experimental plan

The present work was divided into two stages. In stage 1, the effect of the feed frequency on the performance of anaerobic filters was studied, with constant applied organic loading rate (OLR) of 4 gCOD/L.day. In stage 2, a comparison between continuous and semicontinuous feeding methodologies was performed for an OLR of 2.5–11 g COD/L.day.

Stage 1: Influence of the feed frequency on anaerobic filter performance

In the experiments performed at this stage, the hydraulic retention time (HRT) was maintained between 2.5 and 3 days and the OLR was 3.5 and 4.4 g COD/L.day. The feed was supplied using a peristaltic pump connected to a timer. Feed frequencies of 1, 3, 6, 12, 24 and 48 doses per day were tried and compared to continuous feeding. The imposed feeding conditions were maintained sufficiently long (2–3 weeks) to reach steady state and to make sure that the changes observed in the values of the system variables are fundamentally due to the changes in the feed frequency and not to the acclimation of the system. Table 2 shows these conditions for each experiment. As can be seen, both the vinasse concentration and the

daily feed volume varied within a small range so that the feed frequency is the only intervening variable.

The HRT was evaluated by the effluent volume (measured daily) and the useful reactor volume (2.150 L). The OLR was calculated as the quotient between the vinasse concentration, S_{0} , and the HRT.

Once the steady state was reached, the following parameters were measured to characterize the state of the system:

Biogas: daily production, % CH_4 , % CO_2 , % N_2 and % O_2 .

Liquid effluent: pH, volatile fatty acids (C_2 – C_5), COD.

These variables were then used to define other variables (such as the purifying efficiency and the COD fraction that is transformed into methane) that facilitate the treatment and interpretation of the results.

Stage 2: A comparative study of continuous and semicontinuous systems

To enable extrapolation of the conclusions derived from the feed frequency studies of stage 1, two series of experiments with reactors working continuously and semicontinuously (1 dose daily), respectively, were carried out. In contrast to stage 1, for this case we worked with four different OLRs (between 2.5 and

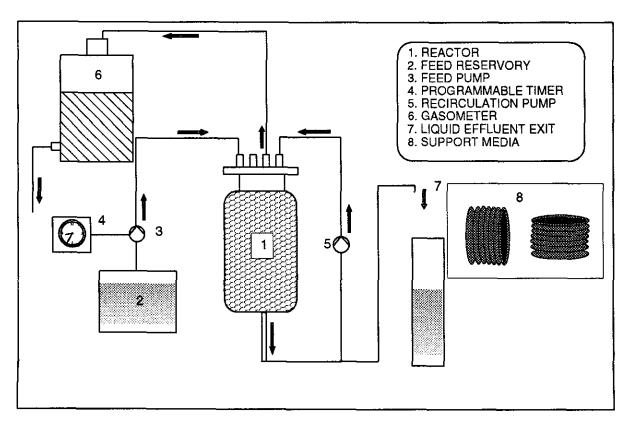


Figure 1. Reactors and support material entities.

Table 2. Feeding conditions for feed frequency study (stage 1)

| Number of doses (doses/day) | HRT (days) | S ₀ (gCOD/L) | OLR (gCOD/L.day) |
|--------------------------------|------------|-------------------------|---------------------|
| 1 | 2.44 | 9.806 | 4.013 |
| 3 | 2.53 | 9.806 | 3.88 |
| 6 | 2.72 | 9.806 | 3.60 |
| 12 | 2.59 | 10.303 | 3.98 |
| 24 | 2.59 | 11.3 | 4.36 |
| 48 | 2.82 | 11.151 | 4.01 |
| Continuous | 3.19 | 11.164 | 3.56 |

Abbreviations: HRT, hydraulic retention time; COD, chemical oxygene demand; OLR, organic loading rate. S_0 = vinasse concentration.

11.0 g COD/L.day) and only two feed frequencies: semicontinuous (one dose daily) and continuous feeding.

To be able to compare both series of experiments the OLR, S_0 and HRT values were maintained to be as similar as possible. Table 3 describes and classifies these experiments as C and S for continuous and semicontinuous feeding, respectively. Experiments with the same number are compared since these have analogous OLRs (the OLRs for the continuous system were slightly higher, approximately 10%, than the semicontinuous system).

Results and Discussion

Stage 1: Influence of the feed frequency on anaerobic filter performance

The following variables are used in the discussion of the results: purifying efficiency, methane production, biogas composition and volatile acid content.

Purifying efficiency. Figure 2 shows purifying efficiency expressed as a percentage of COD removed (plotted with error bars: averages and standard deviations values) versus the number of daily feed doses. One can observe a continuous increase in the efficiency with an increase in the feed frequency, reaching a maximum of 94.8% for continuous feeding. For the first two experiments (1 and 3 doses) the

degradation efficiency was about 70%, for the next two (6 and 12) the degradation efficiencies approached 80%, for the experiments with 24 and 48 doses a purification rate of 90% was reached. For the continuous mode the degradation was around 95%.

Methane yield. It is necessary to establish or define a parameter that links the quantity of methane to the processed substrate. Methane yield can be calculated and expressed as litres of methane produced per gram of COD removed. Thus one can calculate the fraction of substrate destroyed that is converted into methane—this reflects the efficiency of the process. The efficiency is estimated taking into account that 1 g of COD is equivalent to 0.35 L of methane at STP conditions.

In Figure 3, one cannot observe significant differences for the methane yield versus feed frequency, which stayed in the range 0.25–0.30 L CH₄/g COD removed. These parameter fluctuations may be due to the error in such a calculated value from the highly variable values of gas production, gas composition and COD values. Therefore, it is verified that upon increasing the feeding frequency the purifying efficiency increases, but the methane production yield stays approximately constant.

Biogas composition. In the biogas there are four detectable gases: CH_4 , CO_2 , N_2 and O_2 . The presence of N_2 and O_2 (always lower than 10% of total biogas) may be due to air being introduced during sampling. Thus the chosen control parameter is the ratio of CH_4/CO_2 , which may give some idea of the main metabolic routes at a given moment during fermentation. However, biogas composition is also determined by other system variables, i.e. pH and temperature of the medium, which modify the solubility equilibrium for the different biogas components. Thus, CO_2 solubility is enhanced by increasing the pH of the medium but is diminished by increasing the temperature.

If both the pH and the temperature of the medium can be maintained at constant values, an increase of the CH_4/CO_2 ratio with the increase in the feed frequency can be expected. It can be observed in Figure 4 that the experimental data adopt this tendency when the medium pH is nearly constant, i.e. for experiments at 1, 3, 6 and 12 doses/day, and for

Table 3. Feeding conditions for the comparative study of the continuous and semicontinuous systems (stage 2)

| Code | Organic loading rate (gCOD/L.day) | Feed concentration (gCOD/L) | Code | Organic loading rate (gCOD/L.day) | Feed concentration (gCOD/L) |
|------|---|-----------------------------------|-------------|---|-----------------------------------|
| C1 | 2.722 | 10.161 | \$ 1 | 2.511 | 16.723 |
| C2 | 4.395 | 10.161 | S2 | 3.718 | 16.723 |
| C3 | 8.034 | 10.161 | S3 | 7.834 | 15.667 |
| C4 | 11.077 | 17.641 | S4 | 10.059 | 15.089 |

experiments at 24 and 48 doses/day with continuous feeding. Differences between both ranges can be explained by the sharp increase of the medium pH detected for the experiment at 24 doses/day or greater. As the pH of the medium is closely related to

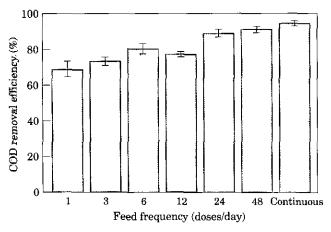


Figure 2. Purifying efficiency, expressed as percentage of chemical oxygen demand (COD) removed, versus the number of daily feed doses.

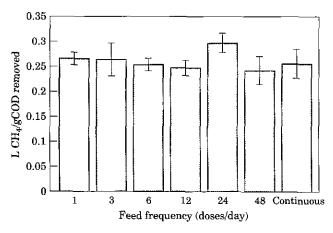


Figure 3. Methane yield (litres of methane per gram COD removed) versus the number of daily feed doses.

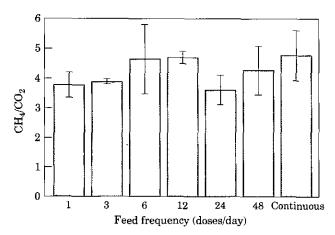


Figure 4. Relation of CH₄/CO₂ to the number of daily doses.

volatile acidity, the establishment of a predictive model for biogas composition is very complex.

Volatile acids. Volatile acid concentration in the effluent is one of the most important variables for determining the reactor stability for depurative anaerobic processes. Figure 5 shows the total volatile acidity (acetic, propionic and butyric acids), expressed as mg/L, versus the number of feed doses added per day.

In this experiment, data were not available for 1 dose/day but it can be clearly observed that volatile acidity decreases sharply when the feed frequency increases in the range 3–24 doses/day. From 24 doses per day, volatile acidity was stabilized at values of 200 mg/L or lower.

Acetic acid was the most important component for the experiments carried out at 3 and 6 doses/day. However, the relative importance of acetic acid became lower when the feed frequency increased and, from 24 doses/day, its concentration was similar to both the propionic and the butyric acids level.

Propionic acid concentration was very high for the lower feed frequencies (500 mg/L) and its concentration decreased when the feed frequencies increased. Likewise, changes in the propionic acid concentration were more slight than for acetic acid, which demonstrates its higher resistance to degradation and transformation into methane.

Butyric acid concentration remained nearly constant (about 50 mg/L) for all the experiments conducted.

As can be see in Figure 6, pH increases when the feed frequency increases, a consequence of lower volatile acids concentrations. A sharp pH increase (about 0.5 units) can be observed from 12 to 24 doses/day, reaching a value of 8.5. At mesophilic conditions, several authors have shown that the optimum pH for anaerobic processes is around 7.2, but in thermophilic conditions (55°C) using neutralized vinasses, the optimum pH reaches the 8–8.5 range. This is explained by the fact that optimum pH and operation temperature are related.

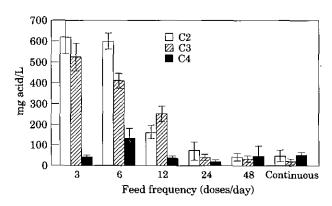


Figure 5. Total volatile acidity (acetic, propionic and butyric acids), expressed as mg/L, versus the number of daily feed doses.

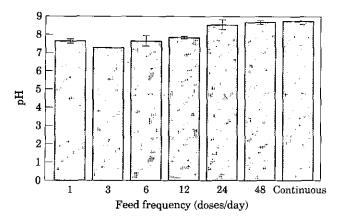


Figure 6. Variation of pH with the feed frequency.

Table 4. Comparison of purifying efficiency for continuous and semicontinuous feeding

| Continuous | | Semicontinuous | | Continuous | |
|------------|--------------------------|----------------|--------------------------|-------------------------------|--|
| Code | Purifying efficiency (%) | Code | Purifying efficiency (%) | over semicontinuous (%) | |
| C1 | 94.75 | S1 | 90.10 | 4.9 | |
| C2 | 93.04 | S2 | 88.94 | 4.4 | |
| C3 | 89.67 | S3 | 89.75 | 0.0 | |
| C4 | 89.37 | S4 | 85.55 | 4.3 | |

Comparison of pH values for both the continuous and the semicontinuous experiments shows that, in the latter, the pH is in the range 7.7–8.0, and in the former, the major pH value is 8.5.

Stage 2: A comparative study of continuous and semicontinuous systems

To apply the stage 1 results to a wider range of OLRs the results from several continuous and semicontinuous experiments (1 daily dose) were compared. The following variables are used in the discussion of the results in this stage: purifying efficiency, methane production and biogas composition.

Purifying efficiency. Table 4 shows that the COD removal efficiency values for the continuously fed units were around 4.5% greater than those obtained for semicontinuous feeding. This difference is much less than that observed in experiments where the feed frequency was modified. This can be attributed to the different biodegradability of feeds used in the various series of experiments. One may conclude that using continuous feeds or at least a feed frequency level about 24 doses/day increases the net purifying efficiency of the system reaching values close to 90%. Figure 7 shows that the efficiency values are always higher within the OLR of 2.5 and 11 g COD/L.day when using continuous feed.

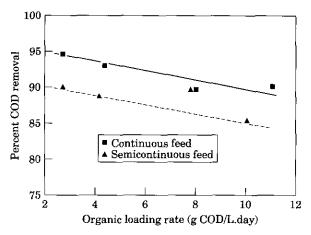


Figure 7. Purifying efficiency for continuous and semicontinuous experiments within an organic load rate range of 2.5–11 g COD/L.day.

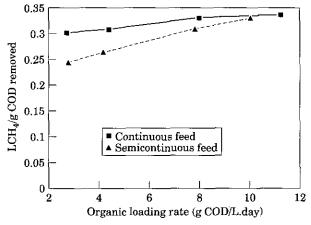


Figure 8. Methane yield versus the organic load rate applied for continuous and semicontinuous experiments.

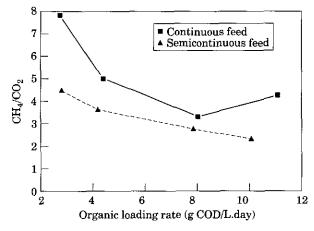


Figure 9. Relation of CH_4/CO_2 for continuous and semicontinuous experiments within the organic load rate range of 2.5–11 g COD/L.day.

Methane yield. Figure 8 shows methane yield versus the OLR for the experiments C1–C4 and S1–S4. One can observe that, on increasing the OLR, the difference in methane yield is reduced, from almost 20% for an

| Control variable | Maximum variable value (frequency) | Minimum variable value (frequency) | Optimum variable value range | Optimum feed frequency range |
|---|---|---|------------------------------------|------------------------------------|
| Purifying efficiency (%) | 94.8 (continuous) | 68.9 (one dose) | > 90 | 24 — continuous |
| Methane yield | | _ ′ | - | Unrelated to feed frequency |
| otal acidity | 1800 (one dose) | 125 | < 200 | 24 — continuous |
| mg/L) CH ₄ /CO ₂ | (one dose) 4.75 (continuous) | (continuous) 3.90 (one dose) | > 4 | 6—continuous |

Table 5. Conclusions of the feed frequency effect on anaerobic filters

OLR of 3 g COD/L.day to 5% for OLRs greater than 8 g COD/L.day. Although continuous feeding yields a higher methane production, this becomes less as the OLR is increased.

Biogas composition. In this experiment it was observed that the CH₄/CO₂ ratio decreased when organic load added increased, for both cases (continuous and semicontinuous). Moreover, the obtained value was always greater for continuous than for semicontinuous operation mode but, as can be seen in Figure 9, the difference between both cases diminishes when the organic load added increases.

Conclusions

Variables selected in the discussion of results for evaluating the performance of the system under study—depurative efficiency, methane production yield and volatile acidity—have shown to be very useful. These variables allow one to determine the effect of the feed frequency on the anaerobic filters' performance.

As can be seen in Table 5, the optimum operation range is around 24 doses/day or greater. Likewise, operation under 3 doses/day makes the system unstable.

Depurative efficiency is maximum with reactors working continuously. Methane production yield is independent of the feed frequency, for a fixed OLR. CH₄/CO₂ ratio increases with the number of doses feeding because it makes the system more stable and the methane percentage in biogas increases. However, this variable is closely related to the medium pH, and hence it is not possible to determine the state of the system from its value only. Volatile acid concentration shows that lower feed frequencies make the system unstable since the volatile acidity levels are very high.

These results are valid for short-term system behaviour for the performance of microbial systems devel-

oped under constant loading. Microbial communities have a remarkable ability to adapt to change, but this adaptation would be rather slow in systems with long SRTs (solid retention times) and slow microorganism growth rates (i.e. methanogens). Therefore, in the long term, these results may not be the same.

On the other hand, comparative study of the continuous and semicontinuous feeding methodologies shows that the continuous, rather than semicontinuous, feeding is better in all ranges studied (2.5–11 g COD/L.day). In this way, the depurative efficiency is higher for continuous feeding. However, with respect to the methane production yield, an increase in the organic load added to the system makes the values obtained for both processes very similar.

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