TWO-PHASE ANAEROBIC DIGESTION OF MUNICIPAL ORGANIC SOLID WASTES

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ABSTRACT

Two stages anaerobic digestion of the organic fraction of municipal solid wastes (OFMSW) is proposed using a batch anaerobic trickling bed (BATB) reactor in the first hydrolysis and acidogenesis stage. At total solids loading of 135 g/L and reaction times around 30 days, total solids (ηTS) and chemical oxygen demand (ηCOD) removal efficiencies above 46% were obtained independently of pH (between 4.1 and 6.4). Dependent on pH were methane production, four times more at pH 6.4 than at 4.8 and four times more volatile fatty acids (VFA) production at 6.4 than at 4.1 and twice than at 4.8. Leachates generated in the BATB reactor were diluted with municipal wastewater and fed to an upflow anaerobic sludge blanket (UASB) reactor at volumetric organic loading rates from 11 to 28 g/L.d where 90% COD removal efficiencies (ηCOD) and 11.4 g COD/L.d were obtained. Two stages anaerobic digestion results in high rates of solids removal and methane production (0.63 kWhr/kg TS fed).

Indexing terms/Keywords
Organic solid wastes, trickling bed, leachate, hydrolysis, methanogenesis

Academic Discipline And Sub-Disciplines
Biothecnology

SUBJECT CLASSIFICATION
Digestión anaerobia

TYPE (METHOD/APPROACH)
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INTRODUCTION

The total generation of municipal solid wastes in Mexico reached 40 million tons in 2010, where the organic fraction (OFMSW) was 53%. Only 56% of these wastes ended up in landfills, 11% were deposited in rustic landfills and the remaining 33% in uncontrolled places or clandestine landfills [1]. Landfilling in Mexico is not a sustainable alternative to dispose of the OFMSW because due to the extended treatment area they need can only be located in the outer reaches of the cities where transportation of wastes is expensive and use of delayed and uncontrolled methane production is inefficient.

Alternatively, two stage anaerobic digestion of OFMSW in compact and controlled reactors is a sustainable treatment, because they can be installed in small plants within the cities reducing transportation costs and using in the surroundings methane, to produce enough energy to carry out the entire management of the municipal solid wastes [2].

In the first acidogenic stage the high concentration of fermentable matter in the OFMSW will produce rapidly high concentrations of volatile fatty acids (VFA) at pH <6. In a second methanogenic stage these VFA can be transformed to methane in an upflow anaerobic sludge bed (UASB) reactor.

Two stages AD of OFMSW using batch stirred tank reactors for the hydrolytic-acidogenic phases have been studied. Vieitez and Ghosh, showed that acidogenesis is inhibited at 13 g VFA.L\(^{-1}\) [3], although Dinamarca et al., obtained more than 25g VFA.L\(^{-1}\) in pH uncontrolled reactors [4].

Using a leaching bed reactor, AD two stages was studied by Chungh et al., Šan & Onay, Zhang et al., Chen et al., Demirer & Chen, Xu et al. They all recycled part of the leachate (the first two using bulking agents to aid percolation through the solids bed) and found that when buffered, the VFA production was increased [5-10]. There are reports that pH affects the hydrolysis first-order constant, [8, 11].

Hence, this step must be hastened. On the other hand, when hydrolysis is accelerated, the VFA may accumulate to a level that is inhibitory to methanogenic and acidogenic microorganisms, [12, 13]. Moreover, build up of VFA level depresses the pH level of the liquid, a condition which decreases the hydrolysis rate of proteins and lipids that are present in the OFMSW [14]. Elango et al. reported that high TS and COD removal are enhanced by neutral pH [15].

The leachates are then treated in UASB reactors, at loading rates from 6 to 32 g COD.L\(^{-1}\).d\(^{-1}\) and concentrations from 0.2 to 60 g.L\(^{-1}\), to obtain COD removal efficiencies (ηCOD) higher than 90% buffering the leachates to pH 7 and supplementing phosphorus [16, 17].

Tran et al. [18], evaluated the AD of OFMSW a two-phase system composed of a fed-batch hydrolysis-acidogenesis reactor (phase I) and a methanogenic Upflow Anaerobic Sludge Blanket (UASB) reactor (phase II). Feeding the first reactor 2 kg of OFMSW with 0.5 L of water, the leachate generated is fed to a UASB, adjusting the pH when necessary and with the addition of nutrients. They worked with organic load rate (OLR) of 1.48 kgCOD.m\(^{-3}\).d\(^{-1}\) and 2.87 kgCOD.m\(^{-3}\).d\(^{-1}\). COD removals obtained 47.5% of the total COD present in the feed OFMSW and the highest methane generation rate was 2.6 Ld\(^{-1}\) or 6.6 g CH\(_4\).COD d\(^{-1}\).

All the reported two stage processes are based on the use of water, to obtain slurries to produce VFA which need to be neutralized and supplemented with nutrients in order to be methanized. For these reasons this study aimed to evaluate the anaerobic digestion in two stages of OFMSW (15% TS): hydrolysis and acidogenesis in a batch anaerobic trickling bed (BATB) reactor to maintain the normal humidity of the OFMSW by trickling of a small amount MWW over the solids bed; and acetogenesis and methanogenesis of the leachate, diluted as needed with MWW, in a UASB reactor (figure 1), in order to use the waste generated in cities.

MATERIALS AND METHODS

OFMSW samples

OFMSW were collected from households and stored at 5°C from one to four days before being fed to the reactors. It was composed of a mixture of fruit residues (melon, papaya, watermelon, pineapple, mango, cucumber, orange, lemon, grapefruit, banana, apple, peach, avocado), vegetables (tomato, spinach, chard, hibiscus flowers, zucchini, peppers, carrots, purslane, parsley, cilantro, onion), processed foods (egg, egg shell, rice, sausage, ham, cheese, tortilla, bread, pasta) and garden wastes. The samples were ground to a particle size of 1-5 mm prior to feeding the hydrolytic reactor batch.

Batch Anaerobic trickling bed (BATB) reactor

Three 4 L propylene cylindrical containers (D = 16 cm, H = 17 cm) were loaded with batches of 3.5 kg of ground OFMSW (Table 1) forming beds of 14 cm high with particle sizes between 1 and 5 mm. Municipal wastewater with COD concentrations around 0.3 g.L\(^{-1}\) was fed to the beds at a rate of 25 mL.L\(^{-1}\).d\(^{-1}\) (Fig 1), maintaining the normal humidity of the OFMSW by trickling a small amount of municipal wastewater (MWW) over the solids bed.

One of the reactors (R\(_{0w}\)) was operated only with MWW. In the other two reactors (R\(_{0L7}\) and R\(_{0L}\)), 10% of the leachate (2.5 mL.L\(^{-1}\).d\(^{-1}\)) was recycled, together with 22.5 mL.L\(^{-1}\).d\(^{-1}\) MWW, buffered to pH 7 and without buffer, respectively. The leachate was collected from the bottom exit of the reactor (figure 1). Biogas was measured by volume displacement in a brine column to avoid gas dissolution. The first batch runs were inoculated with 0.4 L of granular sludge (52 g VSS.L\(^{-1}\) and a
methanogenic activity of 0.6 kg CH₄-COD kg⁻¹ VSS d⁻¹) obtained from a UASB reactor treating a malting plant wastewater. Successive batches were inoculated with 10% of the residual solids of the previous batch.

**UASB reactor.**

Leachates from BATB reactors and MWW (300 mgCOD L⁻¹), were treated in 2.2 L (D=8.4 cm y H=40 cm) UASB reactors inoculated with 0.5 L of the granular sludge (52 g VSS L⁻¹ and a methanogenic activity of 0.6 kg CH₄-COD kg⁻¹ VSS d⁻¹). Mixtures of leachates and MWW, were adjusted of 11 to 28 g COD L⁻¹ and operated at different hydraulic retention times (HRT); 1, 1.25 and 1.5 days. Biogas was measured as described in section 2.

**Analysis of reactors**

Figure 1 shows the setup of the BATB and UASB reactors. A batch of OFMSW is trickling down with a small amount of MWW (Sw = 0.3 g.L⁻¹) to solubilize solids and the leachate rich in soluble COD is diluted with MWW (F_L.S_L + Fw.Sw = F_Uo.S_Uo) to feed a UASB reactor to produce methane.

\[
V \frac{dS_L}{dt} = F_L (S_{W0} - S_L) - Vr_S
\]

\[
\frac{dS_L}{dt} = -D S_L - k (S_{W0} - S_L) - Vr_S \quad (1)
\]

Where
- \(V\) = volume of the OFMSW [L],
- \(S\) = COD concentration [mg COD L⁻¹],
- \(F\) = leachate flow (L d⁻¹),
- \(D\) = the dilution rate = FL.V⁻¹ [t⁻¹],
- \(k\) = the dissolution specific constant [t⁻¹],

Indexes: \(^*\) at the OFMSW, L at the leachate, 0 initial, w at the MWW.
Integration of equation 1 with \((Sw-SL)=-SL\) (as \(Sw\) is negligible compared with \(SL\) taking as boundary conditions; when \(t=0\), \(SL=S^*o\), results in:

\[
S_L = \frac{S^*o}{D-k} (D \ast \exp(-D \ast k) \ast t) - k
\]  

(2)

The performance of the UASB reactor at different organic loading rates \((B_v=SUo/HRT)\) is measured as the COD removal efficiency \((\eta)\) which in turn is a measure of the COD removal rate \((r=\eta COD B_v)\) and a plot of several sets of values is used to determine the maximum loading rate at which the \(r\) decreases beyond an acceptable value.

Statistical analysis

The specific dissolution constant \((k)\) was estimated by fitting equation 2 to experimental data using Polymath 4.1. Data from the different runs were analyzed by one way ANOVA with Tukey-Kramer Comparison Test at \(P < 0.05\).

Analytical methods

The pH was determined with potentiometric equipment HANNA HI 255. Alkalinity, COD, TSS, VSS and FSS, were determined by standard methods, [19]. COD in OFMSW was measured in 1 g ground samples, suspended in distilled water (0.1L), and processed in the same way as the leachate samples. For VFA analysis, a 1.5 mL sample was taken and centrifuged at 1,120 G for 15 min. The supernatant was filtered. A 950 µL aliquot was acidified with 50 µL of HCl 2.2 M. and determined with a HP 5890 gas chromatograph with flame ionization detector (FID) with an AT 1000 capillary column using nitrogen as carrier gas at 4.5 mL min\(^{-1}\), injector temperature 250°C, detector temperature 200°C, with a ramp of 25°C min\(^{-1}\).

Biogas composition was determined using a gas chromatograph (GowMAC 580 series), with a thermal conductivity detector (TCD) operating at the following conditions: Column, detector and injector temperatures: 140ºC, 190ºC, and 170ºC, with a ramp of 25°C min\(^{-1}\). The column was packed with Carbosphere 80/100, using as Helium as carrier gas at 40 psi pressure, flow rate: 25 mL min\(^{-1}\), polarity: 120 mA.

RESULTS AND DISCUSSION

OFMSW characterization

Table 1 shows the OFMSW characterization of thirteen loads to the three BATB reactors. A COD value (1.57 kgCOD.kg\(^{-1}\) VS) with a concentration of 180 g COD.L\(^{-1}\) is found at the solids bed which has an adequate moisture content which allows for an anaerobic degradation, [20]. With the low void space only a small volume of water can trickle down the bed without the risk of flooding it and with no need for bulking agents to enhance percolation as suggested Xu et al. [10].

| Parameter      | Units | OFMSW  |
|----------------|-------|--------|
| Initial pH     |       | 5.5 ± 0.2 |
| Void space     | %     | 14 ± 1.16 |
| Density        | g cm\(^{-3}\) | 0.88 ± 0.03 |
| Moisture       | %     | 84.6 ± 2.2 |
| TS             | g kg\(^{-1}\) | 154 ± 0.4 |
| VS             | g kg\(^{-1}\) | 130 ± 0.3 |
| FS             | g kg\(^{-1}\) | 24 ± 0.1 |
| COD            | g L\(^{-1}\) | 180 ± 60 |
| Proteins       | g kg\(^{-1}\) | 15 ± 5 |
| Carbohydrates  | g kg\(^{-1}\) | 27 ± 17 |
| C/N            |       | 14.87 ± 1.77 |

Hydrolysis and acidogenesis of the OFMSW

Figure 2 shows the dissolved COD of the OFMSW leachates of the three reactors \((R_w, R_wL_7\) and \(R_wL\)). Three phases can be distinguished, an initial lag phase of 10 days, a decaying phase of approximately 20 days and a final stabilization phase of 15 days. After an initial COD in leachate \((S_L)\), lower than the \(So^*\) of solids (180 g L\(^{-1}\)), there is a lag of about ten days where the leachate concentration does not decrease. This is probably due to a large amount of soluble material, already
present in the organic wastes, which slowly washes away due to the small amount of water used. This lag time might be reduced by mechanically dewatering the solid. After this moment, the material which is solubilized due to hydrolysis and acidogenesis, start to leach away and SL starts to decrease (equations 1 and 2) until about 30 days after which, it remains constant. The prediction of SL with equation 2, using the mean of the calculated dissolution constants (Table 2), is plotted for each reactor. The fit suggests that this balance can be used to study this kind of reactors.

![Figure 2](image-url)

**Figure 2.** Leachate COD from BATBR at three different conditions. Rw leached with MWW, RwL7 recycling 10% of leachate and buffered at pH 7 and RwL same as the latter but without pH buffer. (□ first, ◇ second, △ third, ◆ fourth, ⬤ fifth runs, plot of equation 2 using the average k)

Table 2 shows average values of other important results: leachate pH, VFA content, biogas production ($Q_{bg}$) and the removal efficiencies of solids ($\eta_{TS}$) and COD ($\eta_{COD}$). The effect of recycling part of the leachate can be assessed by comparing the pH of $R_w$ and $R_{W_L}$ reactors. The lower pH of the latter negatively impacted on VFA and $CH_4$ formation. By recycling only 10% in $R_{W_L7}$ four times as much VFA as in $R_{W_L}$ was obtained.
Table 2. Response variables of hydrolysis and acidogenesis of OFMSW in BATB reactor

| Run    | Leachate |          | OFMSW bed |          |          |
|--------|----------|----------|-----------|----------|----------|
|        |          | pH      | VFA COD$^{-1}$ | k (d$^{-1}$) | $\eta_{TS}$ (%) | $\eta_{COD}$ (%) | $Q_{bg}$ (L L$^{-1}$ d$^{-1}$) | $y_{CH4}$ (%) |
|        | Initial  | Final    | VFA        |          |          |          |          |          |
| Rw,1   | 4.31     | 4.2      | 26%        | 0.019    | 53       | 42       | 0.15     | 91       |
| Rw,2   | 4.21     | 4.06     | 10%        | 0.038    | 49       | 70       | 0.07     | 92       |
| Rw,3   | 4.53     | 4.23     | 9%         | 0.031    | 38       | 61       | 0.08     | 87       |
| Rw,4   | 4.16     | 5.13     | 12%        | 0.025    | 41       | 54       | 0.14     | 97       |
| Rw,5   | 5.69     | 6.19     | 9%         | 0.023    | 51       | 51       | 0.08     | 92       |
| Av.    | 4.58     | 4.76     | 13.3%      | 0.028    | 46.4     | 55.6     | 0.1      | 91.8     |
| Rwl7,1 | 4.29     | 7.06     | 24%        | 0.039    | 48       | 50       | 0.27     | 91       |
| Rwl7,2 | 4.31     | 6.2      | 25%        | 0.022    | 58       | 51       | 0.37     | 93       |
| Rwl7,3 | 4.75     | 6        | 20%        | 0.024    | 49       | 55       | 0.27     | 91       |
| Rwl7,4 | 6        | 6        | 17%        | 0.021    | 53       | 46       | 0.42     | 89       |
| Rwl7,5 | 5.6      | 6.56     | 18%        | 0.059    | 56       | 42       | 0.39     | 86       |
| Av.    | 4.99     | 6.36     | 20.8%      | 0.033    | 52.8     | 48.8     | 0.344    | 90       |
| Rwl2,1 | 5.13     | 3.7      | 3%         | 0.024    | 47       | 56       | 0.09     | 100      |
| Rwl2,2 | 5.3      | 4.5      | 10%        | 0.019    | 59       | 45       | 0.15     | 100      |
| Rwl2,3 | 4.9      | 4.2      | 4%         | 0.019    | 52       | 38       | 0.13     | 100      |
| Av.    | 5.11     | 4.13     | 5.4%       | 0.021    | 53       | 46       | 0.123    | 100      |

In all experiments pH seems to have no effect on solids destruction, COD removal and the dissolution constant but it impacted on VFA [21, 22] and methane production. In Rw reactor with pH values around 4.13 only carbon dioxide was produced, while 10% CH$_4$ in biogas is obtained in reactors with external buffer RwL7 (pH 6.36) and Rw where pH increases to 6.12 without external buffer, production of CH$_4$ was less (8%). It is reported elsewhere, [11] that pH affects the hydrolysis first-order constant (k) and that high TS removal and COD are enhanced by neutral pH [15]. The opposite was found in this work.

It is only acidogenesis and methanogenesis that are affected by pH values, although Dinamarca et al., obtained more than 25 g VFA L$^{-1}$ in pH uncontrolled reactors [4].

As hydrolysis takes place, VFA increase while pH decreases to their pKa values; if higher than 6, acidogenesis will be faster than in treatments with lower pH values thus obtaining better COD removal, similar result reported by Elango et al. But on the other hand, there is no significant difference on solids destruction. Under neutral conditions (RwL7), for the five batches evaluated in each BATB reactor, it was possible to obtain high VFA formation (more than 20% of degraded COD), more than 49 % solids and 42% COD reduction ($\eta_{TS}$ and $\eta_{COD}$) and biogas production of more than 0.3 L L$^{-1}$ d$^{-1}$ with $y_{CH4}$ greater than 7% with solids retention times of around 33 days.

**Leachate treatment in a UASB reactor.**

Figure 3 shows biogas production and COD removal of the BATB reactor leachates diluted with wastewater from the university campus. There were five runs at different hydraulic residence times (HRT), 1, 1, 1.5, 1.25, and 1.25 d and different inlet concentrations. The first run was a set up period at 1 day HRT and SUo = 20 g L$^{-1}$ (and is not considered in the regression), (Figure 4). At the highest loading rates (run II), there are an accumulation of VFA, mainly propionate, which is the most inhibitory for the methanogenic activity, [12, 13, 23] so the HRT was increased to 1.5 days to bring down the load (Bv).

The leachates treated in UASB reactor, at loading rates similar to those reported by [16, 17], showed removal efficiencies of COD, over 90%, without the addition of nutrients to the leachate and methane composition in the biogas increased to 80%.
CONCLUSIONS

Two phases anaerobic digestion to treat OFMSW; the hydrolytic-acidogenic phase in a BATB reactor speeds up the dissolution of organic solids, reducing 50% of total solids and COD in less than 30 days and produces a leachate with high COD concentrations. Leachate recirculation and pH do not influence hydrolysis rate or efficiency of solids but only VFA and methane production are enhanced with pH higher than 4.8. These results suggest that there is no need to adjust pH or recycle leachate as reported elsewhere because there is no improvement in solids destruction but enhances methane production, which is not desirable at this stage, because it would be difficult to collect it in a continuously fed reactor.

A first order dissolution equation describes the profile of the organic matter recovered in the leachate proving that there is no difference in the k values validating that no pH control in needed thus giving way to a more simple process than previously reported.

The leachate can be treated, diluted with municipal wastewaters, in a UASB reactor up to OLR of 28 g COD L⁻¹ d⁻¹ and producing 11 g CH₄ COD L⁻¹ d⁻¹. Considering that a normal loading rate for MWW treatment is 2 g L⁻¹ d⁻¹, there is enough capacity in MWW anaerobic reactors to treat these wastes. Overall, the process yields 0.2 kg CH₄ COD kg⁻¹ TS fed (0.63 kW*hr kg⁻¹ TS fed).
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