Liquefaction and methanization of solid and liquid coffee wastes by two phase anaerobic digestion process

E. Houbron*, A. Larrinaga** and E. Rustrian*

* Universidad Veracruzana, Facultad de Ciencias Químicas, 94340 Orizaba, Ver., México
** Instituto tecnológico de Orizaba, 94340 Orizaba, Ver., México (E-mail: houbon@prodigy.net.mx)

Abstract This study attempted to investigate the feasibility of volatile fatty acid (VFA) production from coffee pulp hydrolyse, and further to determine the potential of methanization of both the pre-acidified effluent and the coffee wastewater. The experiments were carried out in 2 completely mixed reactors, each one with a working volume of 4 litres. Coffee pulp was used as substrate in the acidogenic reactor and different mixtures of pulper and wash-water and pre-acidified effluent in the methanogenic one. The acidogenic and methanogenic reactors were operated at an organic loading rate of 5 COD g.l⁻¹.d⁻¹ and 0.5 COD g.l⁻¹.d⁻¹. The total, soluble and VFA's effluent COD concentrations of the acidogenic reactor present average values of 57.75, 17.00 and 13.92 g.l⁻¹ respectively. Under these experimental conditions, 23% (COD based) of coffee pulp was hydrolysed with a rate of 1.32 gCOD.l⁻¹.d⁻¹ and the soluble fraction was transformed to VFA’s with an acidification efficiency of 82%. Total VFA’s concentration reached a value of 13.9 gCOD.l⁻¹, and acetate, propionate, butyrate and valerate represented 52%, 28%, 9% and 11% respectively of the liquid phase COD. In the methanogenic reactor, COD removal and methanization of fresh coffee wastewater, pre-acidified effluent and both combined occur with an efficiency of 85% to 95% respectively, with a characteristic biogas composition of 80% CH₄ and 20% CO₂. These results show that a humid coffee “Beneficio” processing daily 23 tons of cherry coffee (fresh fruit), equipped with a two stage anaerobic digestion process could generate at least 1,886 CH₄ m³.d⁻¹. This represents an increase in methane production by a factor 3 to 5 compared to a “Beneficio” using anaerobic digestion only for the treatment of its wastewater.

Keywords Coffee; liquefaction; pulp; two stage anaerobic digestion; volatile fatty acids

Introduction Coffee production represents an important economical activity in several tropical countries. With an annual production of 5.5 millions of quintals (each quintal corresponds to 245 kg of cherry coffee, 57.5 kg of seed parchment coffee or 45.4 kg gold coffee) Mexico is the fourth world producer after Brazil, Colombia and Indonesia. Veracruz State is the second geographical area where coffee production is concentrated in Mexico and most of the coffee is transformed through the “Beneficio de cafe”. The environmental impact of the coffee activity comes from the important generation of solid and liquid waste. One option for the industry to treat the wastewater of this industry is anaerobic digestion with a COD removal efficiency over 70% (Monroy et al., 2000; Valdes et al., 1999). In this case, the main objective of the anaerobic process is to decrease the environmental impact produced by the coffee industry but not to generate energy. Indeed, the energy contained in the biogas is too low compared to the one required for the drying stage of coffee beans. Consequently, the biogas is not valorised in most of the “Beneficio” equipped with anaerobic treatment. Generally, coffee solid waste (pulp) is deposited near the “Beneficio” producing visual and olfaction contamination, and also serious environmental problems with land and water pollution. As a treatment and valorisation alternative, coffee pulp could be used to produce bio-fertilizer, food additive for animals or fuel (Perraud, 1995; Farinet and Pommares, 1999). Until now, all these experimental techniques were not extensively applied since they do not represent economical attractive...
solutions, and coffee pulp is still a great environmental problem, because the new technique does not present an economically attractive solution. Recently, many works have reported the benefit of the two stage anaerobic digestion for organic solid waste treatment (Lopez et al., 1998; Houbron et al., 1999).

The objective of this work is to valorise the solid wastes of the wet coffee processing using a two stage anaerobic digestion to increase the overall biogas production level. More particularly, the main objectives of this study were to evaluate the feasibility of the VFA (Volatile Fatty Acid) production from the hydrolysis-acidification of the coffee pulp and determine the potential of methanization of both, the pre-acidified effluent and the coffee wastewater.

Materials and methods

System configuration

A schematic diagram of the experimental system is shown in Figure 1. The experiments were carried out in 2 completely mixed reactors with a working volume of 4 litres (total volume 5 litres). They were connected to a Marriott flask system to measure methane production.

The acidogenic reactor was maintained at 35°C, and its pH was controlled and regulated at 6 with a Miltron Roy Liquitron Series DP5000 pulse output controller coupled to a Miltron Roy Electronic Metering Pump Series A971-351FI. Triturated fresh coffee pulp diluted 3 times (with tap water, volume per volume) was used as substrate.

The methanogenic reactor was thermostated at 32°C, and the pH was controlled and regulated at 7, with the system mentioned above. Fresh wastewater (Pulper and wash water) from the “Don Pepe coffee Beneficio” (Ixhuatlan del café, Veracruz) and pre-acidified effluent from the acidogenic reactor were used as substrate (Table 1).

Operation mode

Seed sludge used was collected from river sediment from the “Beneficio” discharge point. The respective initial TSS concentration in both reactors was 15 g.l\(^{-1}\). Both reactors were started up in batch mode. To reach a steady state in the acidogenic one, a first batch of 30 days cycle, and then 8 batches of 3 days cycles in each one were applied. To reach a steady state period in the methanogenic reactor, a first batch of 24 days cycle, and then 150 batches of 1 day cycle each were applied. The pH and Organic Loading Rate (OLR) were
used as selective pressure factors. For each reactor the applied pH described above cor-responded to the optimum pH of the involved bacteria. For the acidogenic reactor the OLR applied was 5 g COD.l\(^{-1}.d\^{-1}\) to limit the growth of the methanogenic bacteria and favour the acidogenic one. During the steady state period, to achieve coffee pulp hydrolysis, the acidogenic reactor was operated under SBR (Sequencing Batch Reactor) mode, with a feed volume of 0.4 l.d\(^{-1}\), and without a sedimentation period. These conditions imposed a constant SRT (Solid Retention Time) and HRT (Hydraulic Retention Time) of 10 days. Mixing was made with a magnetic stirrer at 40 rpm.

During the steady state period, the methanogenic reactor was also operated in the SBR mode with an hour of sedimentation. An OLR of 0.5 g COD.l\(^{-1}.d\^{-1}\) was applied to favour the growth of the methanogenic bacteria. This reactor was operated under six different conditions, as shown in Table 2. Each period corresponded to a mixture of coffee wastewater and pre-acidified effluent, which simulated the evolution of the effluent of a coffee “Beneficio” equipped with a two stage anaerobic digestion process. Acidogenic and methanogenic reactors were operated separately.

**Sampling and analytical techniques**

System performance parameters routinely assayed included COD and solids analysis on influent and effluent samples. All these analyses were conducted in accordance with *Standard Methods* (APHA, 1995). The Volatile Fatty Acid (VFA) (Acetic, Propionic, Butyric, Isobutyric, Valeric and Isovaleriac Acid) analyses were carried out on a gas chromatography Varian star CX 4000, with a FID detector, coupled with an integrator Varian and equipped with a Megaboro DB-FFAP column.

Biogas composition was analysed on a gas chromatography BUCK 310, with a TCD detector, equipped with a Packed Buck CTR-1 column and coupled with Peaksimple software for data acquisition and control.

Usually solids as TS, g/kg are used for the balances in the organic solid waste studies. In this paper COD was elected to follow the organic solid waste degradation from solid until gas state, because the COD parameter could be directly measured or calculated in the same unit (mg COD/l) in the solid, liquid and gas state.

**Table 1** Coffee pulp, wastewater and acidified effluent characteristics

<table>
<thead>
<tr>
<th></th>
<th>Total COD (g.l(^{-1}))</th>
<th>Soluble COD (g.l(^{-1}))</th>
<th>TSS* (g.l(^{-1}))</th>
<th>VSS* (g.l(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Triturated pulp</td>
<td>129.2</td>
<td>36.3</td>
<td>142.4</td>
<td>132.4</td>
</tr>
<tr>
<td>Pulper water</td>
<td>18.1</td>
<td>15.3</td>
<td>1.9</td>
<td>1.6</td>
</tr>
<tr>
<td>Wash water</td>
<td>3.5</td>
<td>2.3</td>
<td>1.9</td>
<td>1.7</td>
</tr>
<tr>
<td>Acidified effluent</td>
<td>57.7</td>
<td>17.0</td>
<td>33.2</td>
<td>25.6</td>
</tr>
</tbody>
</table>

* TSS: total suspended solids; VSS: volatile suspended solids

**Table 2** Operating conditions of the methanogenic reactor

<table>
<thead>
<tr>
<th>Period</th>
<th>Conditions</th>
<th>OLR CODg.l(^{-1}.d^{-1})</th>
<th>Cycle</th>
<th>% coffee wastewater(^{(3)})</th>
<th>% acidified effluent(^{(3)})</th>
<th>Total COD(_{tot}) g.l(^{-1})</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>CW(^{(1)})</td>
<td>0.48</td>
<td>1–12</td>
<td>100</td>
<td>0</td>
<td>4.8</td>
</tr>
<tr>
<td>II</td>
<td>CW(^{(1)})+AE(^{(2)})</td>
<td>0.49</td>
<td>13–26</td>
<td>70</td>
<td>30</td>
<td>4.9</td>
</tr>
<tr>
<td>III</td>
<td>CW(^{(1)})+AE(^{(2)})</td>
<td>0.62</td>
<td>27–36</td>
<td>50</td>
<td>50</td>
<td>6.2</td>
</tr>
<tr>
<td>IV</td>
<td>AE(^{(2)})</td>
<td>0.27</td>
<td>37–50</td>
<td>0</td>
<td>100</td>
<td>2.7</td>
</tr>
<tr>
<td>V</td>
<td>AE(^{(2)})</td>
<td>0.50</td>
<td>51–103</td>
<td>0</td>
<td>100</td>
<td>5.0</td>
</tr>
<tr>
<td>VI</td>
<td>CW(^{(1)})</td>
<td>0.48</td>
<td>104–151</td>
<td>100</td>
<td>0</td>
<td>4.8</td>
</tr>
</tbody>
</table>

CW\(^{(1)}\): Coffee wastewater; AE\(^{(2)}\): Acidified effluent; \(^{(3)}\): COD Based; ini*: initial
Results and discussion

Acidogenic reactor

The evolution of the total COD concentration at the entrance and exit of the acidogenic reactor is shown in Figure 2. A steady state period was reached under these conditions after 10 days of operation. This observation confirms the excellent acidogenic bacteria acclimation into the start-up period. Influent and effluent concentration stabilized at total COD concentration of 65.45 g.l\(^{-1}\) and 57.75 g.l\(^{-1}\) respectively (Table 3). In the influent, soluble and particulate COD represent 19.5% and 80.5% respectively, without VFAs. In fact, the solid fraction of coffee pulp is represented by the particulate COD, which has a concentration of 52.72 g.l\(^{-1}\).

Total COD removed is relatively low with an average value of 12%. This confirms the correct acidogenic activity. Analysis of the COD’s transformation, and its different forms are shown in Figure 3.

Total, soluble and VFA effluent COD concentrations of the acidogenic reactor, as shown in Table 3, presented average values of 57.75, 17.00 and 13.92 g.l\(^{-1}\) respectively. For this acidified effluent, soluble and particulate COD represent 29.5% and 70.5% respectively.

Particulate COD concentration of effluent was reduced to 40.7 g.l\(^{-1}\). Under these experimental conditions, 23% (based on particulate COD) of coffee pulp could be hydrolysed and solubilized in the acidogenic reactor with a hydrolytic rate of 1.32 g.l\(^{-1}\).d\(^{-1}\). These results confirm that hydrolytic bacteria were present and that the inoculum used represents an interesting bacteria source. In this study, OLR was the fixed parameter, and the absence of the sedimentation period generated a SRT and HRT of 10 days. Therefore, an optimisation of these parameters should improve the coffee pulp hydrolysis.

The liquefied fraction is also transformed by acidogenic and acetogenic bacteria. In fact, the soluble fraction was transformed to VFAs with an excellent acidification efficiency of 82%. The acidified effluent presented an average concentration of VFA-COD of 13.9 g.l\(^{-1}\). This value presents an interesting potential for an anaerobic methanization. The evolution of the different VFA forms present in the effluent is shown in Figure 4.

It appears from Figure 4 that no VFA was accumulated in the reactor. An average VFA production rate of 1.39 g.l\(^{-1}\).d\(^{-1}\) (VFA-COD) was observed (Table 3). This confirmed the good work of the acidogenic bacteria which transform soluble COD to VFAs and that of the acetogenic bacteria, which transform propionic (Pr), butyric (Bu) and valeric (Va) acid into acetic acid (Ac). Under these experimental conditions, Ac, Pr, Bu and Va represented 52%, 28%, 9%, and 11% of the total VFA-COD respectively. This effluent with more than 50% of acetic acid should be readily biodegradable under methanogenic conditions. The ratio obtained between the VFA is similar to that reported in the literature for the hydrolysis of different organic solid waste (Kida and Sonoda, 1994; Rustrian et al., 1999; Houbron et al., 1999).
Table 3 shows that the major part of influent soluble COD (7.7 g.l\(^{-1}\)) was transformed to methane and biomass while the remaining fraction (5 g.l\(^{-1}\)) was still in a soluble form in the effluent. The methane production occurred with a rate of 0.51 g.l\(^{-1}\).d\(^{-1}\) (COD-CH\(_4\)).

Particulate COD was reduced and transformed into the soluble form (12 g.l\(^{-1}\)). So, the 17 g.l\(^{-1}\) of effluent soluble COD corresponds to the sum of 12 g.l\(^{-1}\) from particulate COD and 5 g.l\(^{-1}\) from the remaining initial soluble COD. Hydrolysis and solubilization rate of the coffee pulp were 1.32 g.l\(^{-1}\).d\(^{-1}\) (COD). Therefore, VFA production occurred with a higher rate of 1.39 g.l\(^{-1}\).d\(^{-1}\) (COD), which suggests that hydrolysis may be the limiting step.

The respective reduction of TSS and VSS, was about 20 and 33% with a rate of 0.85 and 1.27 g.l\(^{-1}\).d\(^{-1}\) respectively. These values confirm the 23% of particulate COD reduction. This experiment demonstrates that coffee solid waste could be hydrolysed and acidified in an acidogetic reactor with a significant efficiency.

To evaluate the methanization feasibility of the acidified effluent, it was used to feed the methanogenic reactor as shown in the next experiences.

**Methanogenic reactor**

An OLR of 1 g COD.l\(^{-1}\).d\(^{-1}\) was applied during the 150 days of the start up period of the methanogenic reactor, fed only with coffee wastewater. Under this condition, COD removal, methanization efficiency of total and soluble COD were 58%, 91.4% and 97.7% respectively. Specific methane production rate of 0.45 g COD\(_{CH4}\)/g VSS.l\(^{-1}\).d\(^{-1}\) and methane yield of 0.35 l CH\(_4\)/g COD\(_{rem}\) were observed (theoretical Y\(_{CH4}\) = 0.39). These results indicated that coffee wastewater could be treated by anaerobic digestion with an efficiency similar to those reported in the literature (Kida and Sonoda, 1992, 1994; Dinsdale et al., 1996).

For the study of the methanization of both pre-acidified effluent and coffee wastewater the OLR was reduced to 0.5 COD g.l\(^{-1}\).d\(^{-1}\). Evolution of total influent and effluent
COD and removal efficiency during the 6 operating conditions (Table 2) are presented in Figure 5.

During the first period (100% coffee wastewater), the effluent presented a residual COD concentration, while total and soluble COD removal presented an efficiency of 85% and 90% respectively, with a methanization efficiency of 99% and a methane yield of $0.38 \text{ l CH}_4 \cdot \text{g COD}_{\text{rem}}^{-1}$. These values are representative of an excellent methanization of the coffee wastewater and will be considered as reference for the other conditions tested. An addition of 30% of pre-acidified effluent during period II, did not change either composition of the treated effluent or removal efficiency. In period III, pre-acidified effluent was increased to 50%, which generated an OLR increment of 30%. Under this overload condition, general efficiencies of the methanogenic reactor were still stable and maximal. Pre-acidified effluent did not generate methanization problems or reactor acidification. A portion of the coffee wastewater was retired during period IV and the methanogenic reactor started to be fed with pre-acidified effluent only. Decrease of COD removal efficiency was closely related to the reduction of OLR to $0.27 \text{ g COD l}^{-1} \cdot \text{d}^{-1}$. Nevertheless, the quality of the treated effluent, the methanization efficiency and the methane yield were not changed. Moreover during period V, when OLR was increased to $0.5 \text{ g COD l}^{-1} \cdot \text{d}^{-1}$ with the same influent, all the parameters remained similar to those observed during period I. Methane production of the pre-acidified effluent, as described above, required at least the activity of acetogenic and acetoclastic bacteria. These results seem to indicate that the species of bacteria were not affected by the substrate changes. Pre-acidified effluent produced methanogenic behaviour similar to that obtained with fresh coffee wastewater. This exhibits the excellent biodegradability and methanization of this substrate. Both effluents could be treated in the same reactor. Period VI was a repetition of period I when the methanogenic reactor was fed with 100% coffee wastewater. With a COD removal of 90%, methanization higher than 90% and a methane yield of $0.38 \text{ l CH}_4 \cdot \text{g COD}_{\text{rem}}^{-1}$, the efficiencies of the methanogenic reactor continued to be similar to those of the reference period. Furthermore, reduction of the solid concentration in the influent during this period permitted us to obtain a best-treated effluent as shown in Figure 5. This observation shows the interest of two stage anaerobic digestion where the solids were treated in a first reactor, and the liquids with a high content of soluble COD are treated in a methanogenic reactor, and most especially in a high performance one.

During all periods, VFAs were not detected in the treated effluent of the methanogenic system. This showed the non-toxicity of both influents and the perfect activity of the acetogenic and acetoclastic bacteria. Methane production rate presented constant values of 2 g

![Figure 5](https://iwaponline.com/wst/article-pdf/48/6/255/423631/255.pdf)
CODCH₄.d⁻¹ (0.5 g CODCH₄.l⁻¹.d⁻¹) with an OLR of 0.5 g COD.l⁻¹.d⁻¹, and 1 g CODCH₄.d⁻¹ (0.25 g CODCH₄.l⁻¹.d⁻¹) with an OLR of 0.25 g COD.l⁻¹.d⁻¹. These values are representative of an excellent methanization efficiency of both pre-acidified effluent and coffee wastewater. Moreover they give us an excellent reference for this type of completely mixed reactor. In comparison, Dinsdale et al. (1996) obtained a similar rate but with a UASB reactor and reported digester failure and acidification in the reactor. Kida and Sonoda (1992, 1994) reported rates of 2 to 18 g CODCH₄.l⁻¹.d⁻¹ with a fluidised bed reactor.

Specific methane production rates higher than 0.5 g CODCH₄. g VSS⁻¹.d⁻¹ were obtained when coffee wastewater was used. These values are representative of the excellent acclimation of the bacteria from river sediment at the “Beneficio” discharge point. Values lower than 0.3 g CODCH₄. g VSS⁻¹.d⁻¹ obtained with pre-acidified effluent could be explained by the lack of acclimation period of the bacteria to this specific substrate. In comparison with fresh coffee wastewater, bacteria had 150 days to acclimate themselves.

Moreover, biogas composition during all periods was constant and representative of a methanization process with an 80% CH₄ and 20% CO₂.

In this study, methanization of fresh coffee wastewater, pre-acidified effluent and mixture of them presented efficiencies of over 90% most of the time. This demonstrates that hydrolysed and acidified coffee pulp can be valorised into. Hence, the methanization kinetics could be optimised by using a high rate methanogenic reactor such as an Upflow Anaerobic Sludge Blanket (UASB) reactor or Expanded Granular Sludge Blanket (EGSB) reactor.

**Conclusions**

This study showed that 23% of coffee pulp could be hydrolysed in the acidogenic reactor with a rate of 1.32 g COD.l⁻¹.d⁻¹ at an HRT and SRT of 10 days. But this significant value is not a limit, and optimal hydrolysis conditions have to be studied.

The acidified pulp presented 30% of the COD under soluble form, with a VFA concentration of 13.9 g COD.l⁻¹ which represented an acidification efficiency of 82%. The soluble fraction had the following VFA-COD profile: 52% of Ac, 28% of Pr, 9% of Bu, and 11% of Va.

The COD removal and methanization in the treatment of fresh coffee wastewater, pre-acidified effluent and both combined occur with an efficiency of 85% and 95% respectively with a rate of 0.5 g COD-CH₄.l⁻¹.d⁻¹ and a methane yield of 0.381 g⁻¹ CODrem. This rate can easily improve by using an UASB or EGSB reactor. Also a specific acidogenic and methanogenic reactor coupled configuration has to be tested.

Treatment of the coffee pulp using a two stages anaerobic digestion process could increase the methane level production at least by a factor 3.

In fact, the two stage anaerobic digestion represents double benefits for the wet process of the “Beneficio de cafe”. The first one is to obtain an energetic self-sufficiency, and the second, is to offer an integral solid and liquid waste management solution with the possibility of river remediation using sludge sediment as inoculum.

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References


