Anaerobic digestion of organic solid wastes: process behaviour in transient conditions

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Abstract This paper presents the study of the variations of the process parameters during transient conditions when substrates with different biodegradability were fed to a pilot-scale anaerobic digester. The two substrates used in the experiments were the mechanically selected organic fraction of municipal solid wastes and a blend of mechanically selected and separately collected organic fraction of municipal solid wastes. Two transient conditions were studied: the mesophilic–thermophilic passage and the increase of the organic loading rate in thermophilic conditions. It was shown that enhanced fractions of biodegradable substrates determined a greater perturbation of the system and needed longer time to reach a new stable condition. The volatile fatty acids concentration and their indirect measure, alkalinity determined upon pH 4, was the more useful parameter to understand the process behaviour because of its sensitivity to the process variations.

Keywords Anaerobic digestion; biodegradability; mechanical selection; municipal solid waste; separate collection; transient condition

Introduction
Anaerobic digestion of organic wastes is now a reliable technology, as confirmed by the growth of full-scale applications in Europe in the last decade (Mata-Alvarez et al., 2000). At present, more than one million tonnes per year of organic wastes (wet weight) are digested in dedicated industrial plants worldwide. These wastes are converted to biogas and stabilised residual matter (Verstraete et al., 2000). According to De Baere (2000), in the early 1990s anaerobic digestion of biowaste and mixed and grey wastes were similar (about 100,000 tons/year for each) while biowaste treatment has been prevailing in recent years, reaching levels of 900,000 tons/year in 2001. This fact is due to the introduction of source and/or separate collection of the organic fraction of municipal solid wastes (OF-MSW) in most of the urbanised areas of the European Union. Therefore, an increase in the biodegradability of the treated substrates has to be expected and, hence, overloading situations in anaerobic digesters can be more frequently observed and have to be properly managed. Therefore, particular attention is needed in evaluating the variations of the process parameters in transient conditions for substrates characterised by different biodegradability. In fact, the ability to manage a biological process is also related to the proper control of transient conditions and not only to the steady-state performances. Despite the lack of knowledge on this issue, research studies on anaerobic digestion processes generally focus on yields and values of stability parameters in steady-state conditions and only a few papers about transient conditions have been published in the last decade (Pavan et al., 1994; Ahring et al., 1995). Generally, the start-up phase has been considered (Lepisto and Rintala, 1995; Fernandez et al., 2001; Bolzonella et al., 2003). In this paper the passages between different steady-state conditions when substrates with different biodegradability are digested will be considered. The paper aims to focus attention on the most useful
parameters of the process during transient conditions. To do this, both literature (Pavan et al., 1994) and original data were considered. Two particular transient conditions were studied: a change in the organic loading rate (kgVS/m$^3$ reactor day) and a change in reactor temperature (from mesophilic to thermophilic conditions). These situations were chosen since they are frequently encountered when managing full-scale digesters and because thermophilic processes have been prevailing over mesophilic ones in recent years (De Baere, 2000).

**Methods**

The experimental work considered the anaerobic digestion of two different types of substrates: the mechanically selected OFMSW (MS-OFMSW) coming from municipalities of the north-east of Italy and the separately collected OFMSW (SC-OFMSW) coming from fruit and vegetable markets. In the first phase of the experimentation, only the MS-OFMSW was fed to the pilot-scale reactor, whereas in the second phase a blend of mechanically and separately collected OFMSW was fed (75% MS-OFMSW + 25% SC-OFMSW, on wet weight). Typical characteristics of the two substrates are reported in Table 1.

In this study, the semi-dry anaerobic digestion process was applied (Cecchi et al., 1991, 1993). When treating both substrates the total solids (TS) content in the digester feeding was about 20% but, in the second case, a more biodegradable substrate was used (TVS/TS 60%). Experimental details of the reactor and analytical methods can be found in Pavan et al. (2000). Besides biogas yields, also the variations of the stability parameters of the process (e.g., pH, alkalinity, VFA and biogas composition) were considered during the transient conditions for the two tested substrates. The following transient situations were studied:

- thermal transient: moving from mesophilic (37°C) to thermophilic (55°C) temperatures in the digester;
- organic loading transient: moving from low (6–7 kgVS/m$^3$ d) to high OLR (9–10 kgVS/m$^3$ d) in a thermophilic environment (55°C).

The operational conditions of the reactor and the typical performances obtained in final steady-state conditions are summarised in Table 2.

**Results and discussion**

**Thermal transient: moving from mesophilic to thermophilic conditions**

Firstly, the thermal transient condition was analysed. When treating both substrates the temperature change was performed very quickly (a few days) while stopping the digester feeding for a week. When passing from mesophilic (30–40°C) to thermophilic (50–60°C) conditions the process is highly stressed, since the thermophilic bacteria are initially absent in the reactor (van Lier et al., 2001). In this experimentation, the imbalance of the food chain within the bacterial consortia was clearly shown by the volatile fatty acids increase: VFAs passed from about 200 to 1,200 mgCOD/l when treating the MS-OFMSW (Figure 1).

<table>
<thead>
<tr>
<th>Parameter</th>
<th>MS-OFMSW</th>
<th>MS-OFMSW + SC-OFMSW</th>
</tr>
</thead>
<tbody>
<tr>
<td>TS, g/kg</td>
<td>223</td>
<td>201</td>
</tr>
<tr>
<td>TVS, %TS</td>
<td>49</td>
<td>60</td>
</tr>
<tr>
<td>COD, g/kg</td>
<td>117</td>
<td>120</td>
</tr>
<tr>
<td>N, g/kg</td>
<td>12</td>
<td>14</td>
</tr>
</tbody>
</table>

Table 1 Characteristics of substrates used in the experimental work

MS-OFMSW: mechanically selected OFMSW; SC-OFMSW: separately collected OFMSW
That is, methanogenic bacteria were not working properly. Gas chromatographic analysis showed that among the different VFAs the acetic acid represented 80% of the total and showed the larger variations of concentration after the stressing of the process, while other acids (from C3 to C6) did not vary significantly.

Obviously, the change of the organic loading was also important. This was gradually increased after the temperature change, passing from 0 to 7.5 kgVS/m³/day in 10 days. The other parameters did not show particular variations. Alkalinity determined at pH 4 showed a little increase when VFA reached high values, while alkalinity at pH 6 was constant. This determined a decrease in the buffer capacity of the system. Consequently pH showed a little decrease (Figure 1) from 7.6 to 7.2 after day 195. Considering the GPR value it decreased from 1.5 to 0.35 m³/m³/day (see Figure 2), due both to the temperature change and the interruption of feeding. Then, it slightly increased again to 2.0 m³/m³/day in about three weeks and after day 195 remained constant. The SGP value showed a slow but constant increase for a couple of weeks to a final stable value of 0.22 m³/kgVS (Figure 2). Methane presence in biogas increased from 52 to 60% when increasing the reactor temperature (Figure 2).

When the blend of MS- and SC-OFMSW was used as feeding, the situation appeared quite different. Also in these conditions the GPR value showed a decrease, passing from about 1 m³/m³/day to 0.5 m³/m³/day and then down to 0.25 m³/m³/day (days 187–191 in

**Table 2**  Operational conditions and yields in steady-state conditions

<table>
<thead>
<tr>
<th></th>
<th>MS-OFMSW</th>
<th>MS—SC-OFMSW</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steady-state operational parameters</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Temperature, °C</td>
<td>Meso</td>
<td>Thermo</td>
</tr>
<tr>
<td>OLR, kgVS/m³/day</td>
<td>7.5</td>
<td>7.0</td>
</tr>
<tr>
<td>HRT, day</td>
<td>14.7</td>
<td>14.6</td>
</tr>
<tr>
<td>Steady-state performances</td>
<td></td>
<td></td>
</tr>
<tr>
<td>TVS, % removal</td>
<td>23</td>
<td>34</td>
</tr>
<tr>
<td>GPR, m³/m³/day</td>
<td>1.4</td>
<td>2.5</td>
</tr>
<tr>
<td>SGP, m³/kgVS, fed</td>
<td>0.20</td>
<td>0.43</td>
</tr>
<tr>
<td>CH₄, % biogas</td>
<td>52</td>
<td>61</td>
</tr>
</tbody>
</table>

MS-OFMSW: mechanically selected OFMSW; SC-OFMSW: separately collected OFMSW; Meso: mesophilic; Thermo: thermophilic, Low: low OLR, High: high OLR

**Figure 1** Variations of stability parameters during the passage from mesophilic to thermophilic operational conditions treating MS-OFMSW
Figure 3), when moving from the mesophilic to the thermophilic range of temperature. After that, the value increased in 10 days to typical values for these substrates: 2 m³/m³ day.

In total, some 30 days were needed to recover the reactor stability after the temperature change. The CH₄ content in biogas (as %) slightly increased, passing from 55% to 75% in 15–20 days after the temperature change. This value was clearly higher than the one previously determined treating mechanically sorted MSW (70% rather than 60%) probably because of the biodegradability of the rated substrate. Concerning the variation of the volatile fatty acids, one notes that it was very rapid: in fact, it increased and passed from 2,000 to 3,000 mgCOD/l the day after the temperature was increased and took some 40 days to come back down to some 2,000 mgCOD/l.

The VFAs concentration was clearly higher when treating more biodegradable substrates: 2,000–3,000 mgCOD/l rather than 1,000 mgCOD/l. According to the VFAs profile, alkalinity determined at pH 4 also increased to a value of 6–8 gCaCO₃/l and then decreased to 3 gCaCO₃/l after day 225. On the other hand, alkalinity at pH 6 showed a stable value, in the range 1.5–3 gCaCO₃/l.

Figure 2 Variation of reactor performances during mesophilic–thermophilic passage

Figure 3 Parameters variations during the passage from mesophilic to thermophilic operational conditions treating a blend of MS-OFMSW and SC-OFMSW (75%+25% on wet weight)
When the VFAs concentration decreased, after day 220, the difference between alkalinity determined at pH 4 and 6 was clearly reduced. These values were similar to those previously observed treating MS-OFMSW. The pH value showed a slight but constant increase, interrupted only between days 195 and 205, when it passed from 7.5 to 7.0. However, its value was always in the normal range.

**Loading transient: increasing the organic loading rate in thermophilic environment**

Treating the MS-OFMSW during increasing the OLR from 6 to 10 kgVS/m³d (Figure 4), gave only little variations in biogas production and composition (see Figure 5).

In particular, the GPR passed from an average value of 2.3 to 3 m³/m³d, while the methane content showed a low decrease, from 65 to 55%. The TVFA concentration showed a sharp increase, from 100–200 to 1,200 mgCOD/l, after day 275 when the OLR increased. The same behaviour was observed when increasing the reactor temperature. The value then decreased to 500 mgCOD/l and reached a stable concentration of some 600 mgCOD/l after one week. Alkalinity, determined at pH 4 and 6, showed little variations between days 280 and 290 and then increased to final values of 7 and 3 gCaCO₃/l, respectively. The variation of alkalinity determined at pH 4 was more evident and is to be related to the VFAs increase. The pH varied slightly around a value of 8.5.

However, except for TVFA, only little variations of the parameters were observed. When treating the blend of MS- and SC-OFMSW, the increase of the organic load from 3 to 9 kgVS/m³d (see Figure 6) in the thermophilic environment determined a first immediate increase in VFA concentration in the reactor (see Figure 7): they passed from some 1,500 mgCOD/l to 3,000 mgCOD/l in a few days, and after some 30 days decreased and remained constant at 2,000 mgCOD/l.

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**Figure 4** Progressive OLR increase when feeding the MS-OFMSW

**Figure 5** Parameters variations during OLR increasing feeding MS-OFMSW

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Alkalinity determined at pH 4 and 6 showed a little increase in the first days and then decreased for more than 30 days until it reached a stable value: alkalinity at pH 4, in particular, passed from 8 to 4 gCaCO₃/l in 30–40 days, while alkalinity at pH 6 passed from 4 to 3 gCaCO₃/l.

Alkalinity at pH 4 showed a similar pattern compared to VFA since it is an indirect measure of that parameter. A relation between the two parameters shown as a VFA decrease of 1 gCOD/l determined a decrease of alkalinity of 4 g/l as CaCO₃. The pH value showed a decrease after day 15, determined by the VFAs increase around day 20, and then reached a stable value of 7.3–7.4.

Concerning biogas production and composition, it was evident that there was a progressive increase of the GPR: this passed from 0.2 to 2.1 m³/m³d and then dropped down to 1.2 m³/m³d after day 40 when feeding was interrupted for a week because of technical problems. Then, it rapidly increased again and reached a stable value of 2.3 m³/m³d. The SGP value showed the same trend and reached a stable value of 0.26 m³/kgVS. The CH₄ content in biogas showed no variation during the OLR increase but then passed from 75 to 65% after day 40.

**Rates of variation of the different parameters**

In order to determine the rate of variation of the different parameters, the linear regressions of the data were performed: the slope of the linear regressions is the rate of variation for the different parameters. Table 3 summarises the rates of the variations obtained in the different tested conditions. The intercept values were not considered since they depend on the specific process conditions.

According to the reported data, it is evident that the VFA concentration showed the
greater variations, followed by total alkalinity determined upon pH 4 and the gas production rate (GPR). The first two parameters could be used to verify the variation and the recovery of the process imbalance whereas the GPR could be useful only when the temperature and the organic loading rate were increased. The same could be said for the methane percentage in the biogas. Alkalinity determined upon pH 6 generally showed minimal variations, whereas pH variations were similar to the instrumental errors. Moreover, pH variations were shown to be late if compared with other parameters’ variations.

On the basis of the reported rates it was shown that the higher was the biodegradability of the substrate (i.e., the blend of MC and SC-OFMSW) the higher were the rates when a perturbation was introduced in the process. This was particularly clear for VFAs and alkalinity at pH 4, but not for other parameters.

Data correlations ($r^2$) were quite good (generally $>0.80$) for the parameters GPR, VFA concentration and total alkalinity determined upon pH 4.

Considering the “decreasing” rates of the parameters it was shown that these were generally low. This means that a long time was needed to recover the stability of the process when a perturbation was introduced, especially for substrates with a high biodegradability.

According to these results the importance of the parameters to be monitored to verify the process stability is the following:

VFAs concentration $>$ alk (at pH 4) $>$ GPR $>$ methane content $>$ alk (at pH 6) $>$ pH

Therefore, according to the results reported in Ahring et al. (1995), it is confirmed that VFA concentration is a good parameter to evaluate the variations of the stability conditions in perturbed processes. However, in this study, the acetic rather than the propionic or butyric acid, was found to be the most important parameter for the process monitoring.

**Conclusions**

The main conclusions deriving from the experimental work are the following: firstly, the changes of the main parameters during transient conditions were strictly dependent on the biodegradability of the treated substrates. The higher the biodegradability the higher were the variations: the substrates with a higher content of biodegradable matter showed the greatest variations of the stability parameters of the process (VFA, alkalinity, biogas production and composition). The time needed to recover a new steady state after a

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**Table 3 Rates of variation of the different parameters in the tested conditions**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>MS-OFMSW Temperature</th>
<th>Organic loading</th>
<th>MS – + SC-OFMSW Temperature</th>
<th>Organic loading</th>
</tr>
</thead>
<tbody>
<tr>
<td>VFA gCOD/l.d</td>
<td>Increasing</td>
<td>+61</td>
<td>+138</td>
<td>+665</td>
</tr>
<tr>
<td>gCOD/l.d</td>
<td>Decreasing</td>
<td>-121</td>
<td>-73</td>
<td>-72</td>
</tr>
<tr>
<td>Alk pH 4 gCaCO$_3$/l.d</td>
<td>Increasing</td>
<td>+0.49</td>
<td>+0.04</td>
<td>+1.62</td>
</tr>
<tr>
<td>gCaCO$_3$/l.d</td>
<td>Decreasing</td>
<td>-0.07</td>
<td>-0.20</td>
<td>-0.07</td>
</tr>
<tr>
<td>Alk pH 6 gCaCO$_3$/l.d</td>
<td>Increasing</td>
<td>nd</td>
<td>+0.02</td>
<td>+0.05</td>
</tr>
<tr>
<td>gCaCO$_3$/l.d</td>
<td>Decreasing</td>
<td>nd</td>
<td>-0.05</td>
<td>-0.09</td>
</tr>
<tr>
<td>pH</td>
<td>Increasing</td>
<td>+0.03</td>
<td>+0.02</td>
<td>+0.02</td>
</tr>
<tr>
<td></td>
<td>Decreasing</td>
<td>-0.07</td>
<td>-0.03</td>
<td>nd</td>
</tr>
<tr>
<td>GPR m$^3$/m$^3$/d.d</td>
<td>Increasing</td>
<td>+0.08</td>
<td>+0.03</td>
<td>+0.06</td>
</tr>
<tr>
<td>m$^3$/m$^3$/d.d</td>
<td>Decreasing</td>
<td>nd</td>
<td>nd</td>
<td>-0.03</td>
</tr>
<tr>
<td>CH$_4$ %/d</td>
<td>Increasing</td>
<td>+0.2</td>
<td>+0.4</td>
<td>+0.6</td>
</tr>
<tr>
<td>%/d</td>
<td>Decreasing</td>
<td>nd</td>
<td>nd</td>
<td>nd</td>
</tr>
</tbody>
</table>

nd: not determined, because of no significant variations
perturbation was more or less the same for the tested substrates, the thermal and the loading perturbation. Secondly, the volatile fatty acids concentration and its indirect measure, the alkalinity determined at pH 4, are the best monitoring parameter: their variance is sharp and can be easily verified after a perturbation. According to the reported results, the significance of the stability parameters was the following:

VFAs concentration > alk (at pH 4) > GPR > methane content > alk (at pH 6) > pH

Finally, the knowledge gained is of particular importance when considering the process behaviour during the transient conditions or the start-up phase of the process.

References