

CONTROL PARAMETER VARIATIONS IN AN ANAEROBIC FLUIDISED BED REACTOR SUBJECTED TO ORGANIC SHOCKLOADS

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ABSTRACT

A pilot fluidised bed reactor was subjected to shockloads using wine distillery wastewater. The parameters' response was recorded. Parameters analysed in the gaseous phase were : H_2 , CH_4 , CO_2 and gas production ; in the liquid phase : pH, temperature, TOC and VFA. Different shockloads were applied : short but intensive shockloading, long shockloadings ; temperature variations ; change of nature of substrate (wine distillery wastewater was replaced by glucose). Generally speaking, the parameters' response was short (less than one hour).

The specific parameters of the gaseous phase and pH can be used for the optimal control of the fermentation process.

KEYWORDS

Methanisation ; fluidised bed reactor ; organic shockload ; gaseous phase ; hydrogen ; control.

INTRODUCTION

In the last ten years, a lot of progress has been made by many researchers on the techniques and kinetics of anaerobic digestion, resulting in the development of high performance digesters with relatively short residence time. Nevertheless, the risks of operation failure become more frequent in these short residence time reactors.

As far as the treatment of wastewaters is concerned, this risk is high, especially when the organic load on the digester exceeds the potential capacity of the microbial population. This overload may be due to an excessive increase in the influent organic load or due to a decrease in the activities of the microorganisms in charge of the elimination.

Taking into account the performance at equilibrium, these overloads could be the results of the following phenomena.

Feeding Characteristics

- increase of influent COD (Chemical Oxygen Demand)
- increase of feed rate
- change in the characteristics of influent, a pronounced change in the nature of its organic matter
- variation of pH
- presence of toxic elements (organics, mineral, heavy metals)

Operation Methods

- excessive temperature fluctuations
- working hydraulic variations
- excessive loss of microorganisms

The response of the microbial ecosystems to these disturbances depends on the type of reactor, the wastewater to be treated and the degree of the modification's effects. By shortening the residence time, a rapid detection of these disturbances and intervention is obvious.

An automatic control of a digester would guarantee an optimal operation. Therefore control parameters which can provide a lot of information on the behaviour of the digester and whose measurement can be made by simple and usual methods are of great interest. In the case of an anaerobic digester, the most interesting parameters are in the liquid and gaseous phases. A review of the nature and interest of these parameters was recently made (Moletta, 1989 ; Switzenbaum et al, 1990). The following parameters, which are used for manual control of digesters, could be proposed for an automatic control.

In the liquid phase : volatile fatty acids, pH, alkalinity, chemical oxygen demand (COD), Total Organic Carbon (TOC) and dissolved hydrogen.

In the gaseous phase : Production rate and composition of gas (CH_4 , CO_2 , H_2 , CO).

In this work, the responses of most of these parameters to varying organic shockloads were studied in order to identify the parameter (s) to be closely monitored for an optimal automatic control of anaerobic digestion.

MATERIALS AND METHODS

Digester Description

The study was carried out in an upflow fluidised bed reactor of a total capacity of 22 litres. Bed expansion was created by the pump of the recycling tubing. The expanded working volume was 8.6 litres. The support material was a granular media (BIOLITE) with an effective size of 280 μm diameter. Biomass was estimated by measuring the volatile solids fixed on the support material (Degrémont, 1978).

Substrate Used

The substrate was prepared from a thirty times concentrated wine distillery wastewater (vinasses) of pH 3.4. During each operation, the

wastewater was diluted to the required concentration. The pH was adjusted to neutrality with slaked lime and NaOH 10N in order to be certain that the response of the reactor was only due to organic shockloads and not to influent acidity. In order to improve its fermentability, substrate was supplemented with 530 mg/l of NH_4Cl , 50 mg/l of KH_2PO_4 , 10 g/l of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ and 10 g/l of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$.

Standard Experimental Conditions

The standard operating conditions of the reactor were as follows :
- reactor temperature : 37°C - feeding rate : 5.5 litres/day (or residence time of 4 days) - influent COD : 20 g/l - load : 12.8 kg COD/m³.d. The shockloadings were made relative to these standard operating conditions.

Performances at Standard Experimental Conditions

The average values of the study parameters at standard working conditions were :

- in the reactor : pH = 7.0 - TOC = 0.5 g/l - acetate = 0.045 g/l - propionate = 0.072 g/l

- gas production : 55 l/d - gas composition : CH_4 = 68 % ; CO_2 = 32 % ; H_2 = 44 ppm (v/v) - COD reduction : 94 %

At each shockload, the values of the above parameters were compared with those of the standard working conditions.

Response Time Determination

Liquid phase. This was done by injecting a solution of LiCl (6.7 g of LiCl in 50 ml of wastewater) in the inlet of the reactor. Lithium analysis was done by flame spectrophotometry (CORNING 410), on samples taken from the recycling tubing.

Gaseous phase. Nitrogen gas was injected from the bottom of the reactor (3 l/h). The response time was detected from the gas production rate and gas composition (CO_2 , CH_4 and H_2).

Organic Overload

The following experiments were carried out :

- short (15 minutes) and intensive shockloading at the inlet. The applied organic loading was twenty times greater than the standard experimental loading.

- long shockloading at the inlet :

* During 8 hours, the COD concentration was multiplied by 6. Then the feed was stopped for 4 hours. Finally, the COD concentration returned to standard conditions.

* During 8 hours, the flow-rate was multiplied by 4. Then the feed was stopped for 4.5 hours before returning to the standard running conditions.

* Temperature decrease from 37 to 26°C for 8.5 hours.

* Change of nature of substrate. During 24.5 hours, vinasse was replaced by glucose at the same COD concentration.

Analysis

Several parameters were continuously measured with receptors connected in line. The values were directly fed to a computer (ANALOG DEVICE MACSYM 120) permanently installed by the side of the reactor.

* In the liquid phase, the pH and temperature were measured continuously from the recycling unit.

* In the gaseous phase, the following measurements were done :

- gas production rate : measurement with an impulsive counter (Moletta, 1982);

- percentage of CO₂ and CH₄ : continuous measurement with an infra-red analyser (ULTRAMAT 22P SIEMENS);

- hydrogen content : usually measured every half an hour by an analyser, "Exhaled Hydrogen Monitor" (GAS MEASUREMENT INSTRUMENTS LIMITED). The gas sample analysed was taken automatically by a pump placed in the gas circuit. H₂S was trapped by the passage of the sample through a DRAEGER tube CH 281 01.

The other measurements were manually done from liquid samples drawn from the recycling unit. They were :

- total Organic Carbon : measured by the method of oxidation by Ultraviolet light with a DOHRMAN CARBON ANALYSER DC 80 coupled with an infra-red detector HORIBA PIR 2000 (Landre, 1983).

- Volatile fatty acids : measured by GPC with a chromatography INTERSMAT IGC 120 DFL coupled with an integrator CHROMATOPAC CR 3A (Laroche, 1983).

RESULTS AND DISCUSSION

Response Time Related To Reactor Dimensions and Operating Conditions

The response time in the liquid of the digester was very short (Fig. 1). After 15 minutes, the concentration of lithium increased from 0 to 8 ppm and became maximum after 45 minutes.

The response time in the gaseous phase was also short : after 6 minutes, the gas introduced at the bottom of the reactor was detected at the hydrogen sensor (by dilution of H₂ concentration). In 10 minutes, nitrogen was detected by measurement of gas composition . In this reactor, the response or contact time due to reactor dimensions and operating conditions were very short. The reactor can therefore be considered as completely mixed.

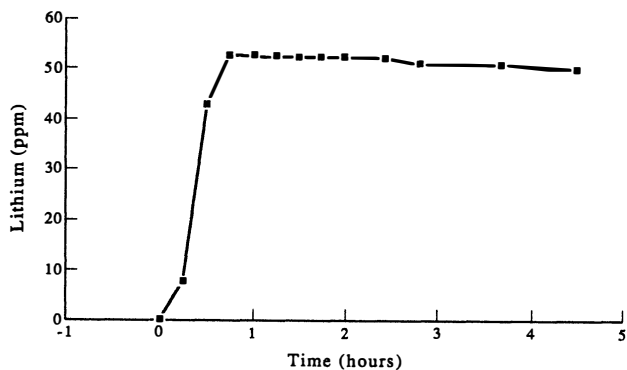


Fig. 1. Lithium concentration in the effluent against time.

Short Shockload

This shockload was simulated by a transitory increase of the inlet COD. The variation of gas production, gas composition and volatile fatty acids are shown in Figure 2.

A rapid and short duration variation of gas composition was observed. It was also observed for the pH which decreased only by 0.2 units.

On the other hand, VFA, which showed immediate response to shockload, had longer time to return to normal concentrations. Hydrogen showed a significant variation, with its concentration increasing by 3.5 times in just half an hour.

These variations (on H_2 , VFA, TOC and gas composition) are similar to the observations made by Mosey and Fernandes (1989) for a reactor, fed with easily fermentable substrate (reconstituted skimmed milk), an hour daily. But their hydrogen response time was shorter; its appearance was noticed only 10 minutes after the shockloading. Many authors have noticed the importance of hydrogen, either in the liquid phase (Whitmore et al, 1987) or in the gaseous phase (Mosey and Fernandes, 1984), as a control parameter in anaerobic digestion. The transitory variation of CH_4 and CO_2 percentages in the gas was due to a relatively greater increase in CO_2 than in CH_4 production rate in the digester.

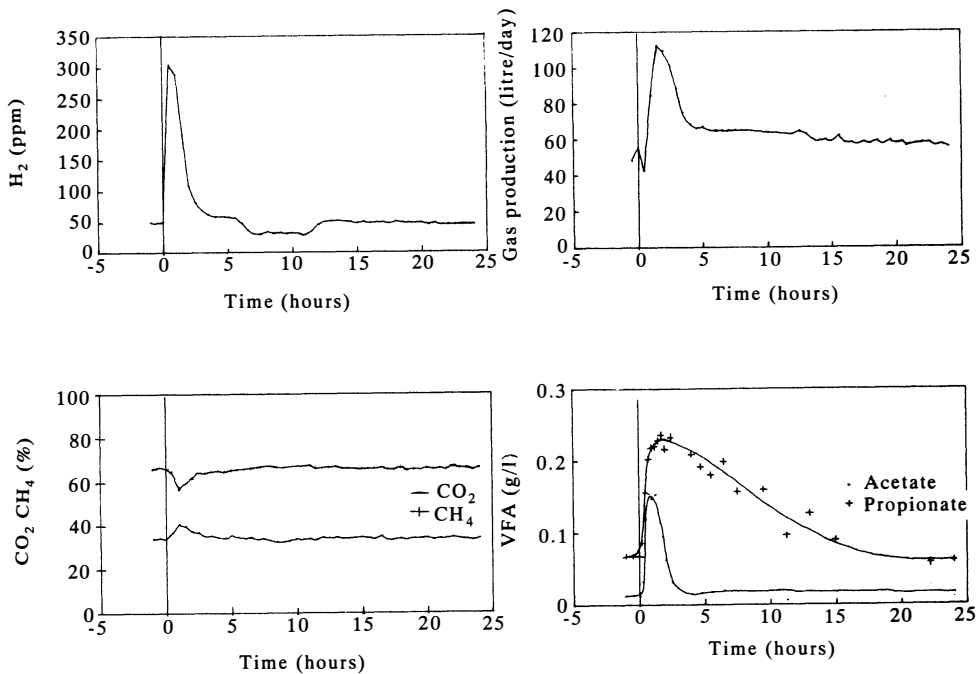


Fig. 2. Variation of VFA, gas production and gas composition (H_2 , CH_4 and CO_2) after a short and intensified shockload (20 times the standard load during 15 min).

Long Duration Shockload

Influence of the influent concentration. The feeding with a six times more concentrated influent than the standard loading gave the same variations as previously, but these variations continued throughout the period of shockloading. Fig. 3 shows the variations of gas composition.

Stopping the feeding immediately after the shockload resulted in a rapid decrease in hydrogen content in the gas. For CH_4 and CO_2 , the return to normal values was slower. This shockload was relatively severe ($75.6 \text{ kg COD/m}^3\cdot\text{d}$) because there were constant accumulations of TOC and VFA during its application. They returned very slowly to their initial values.

A shockload of the same type and duration, using an anaerobic filter treating ice-cream wastewater, produced a similar variation of control parameters (Cayless *et al.*, 1989).

For this type of shockload, Lloyd and Whitmore, (1988) observed a transitory increase of dissolved methane. The methane concentration variation in the liquid phase is therefore different from its variation in the gaseous phase.

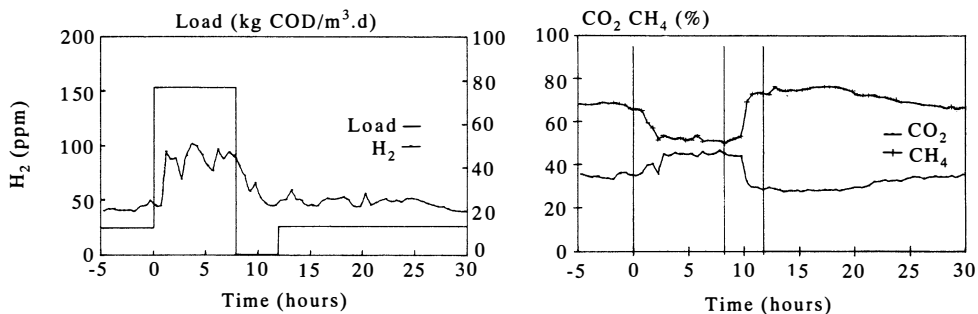


Fig. 3. Variation of gas composition for an 8 hour shockloading, followed by a 4 hour interruption of feeding before a return to standard feeding.

Influence of influent flow rate. Increasing the feeding rate from 5.5 l/d to 22 l/d produced the same variations as previously. For the same type of shockloading, using a chemical industry wastewater, Kennedy and Van den Berg (1982) obtained an increase in the volatile fatty acids concentration and a drop in the methane content of the gas in less than one hour. This type of shockloading produces also a transient increase in CH_4 content and dissolved hydrogen in the reactor (Lloyd and Whitmore, 1988).

Fig. 4 illustrates variations of TOC and gas production. It is important to note that even though a gradual decrease of VFA and TOC was observed in the reactor when the feeding was stopped, the gas flow-rate was relatively high during 3 hours (Fig. 4). After a feed interruption, the

response of gas production can be an indication to detect periods of shockloading in a digester (Stander et al, 1968).

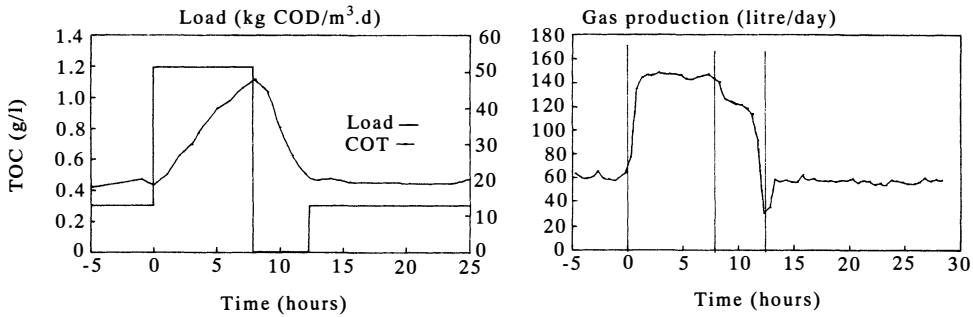


Fig. 4. Variation of TOC, gas production and load for a shockload caused by an increase of the feeding rate by four times.

Influence of temperature drop. Temperature variations result in a drop of microbial activities and can cause a disequilibrium between the flux of organic materials entering the reactor, and the quantity admissible for the microbial ecosystem. The temperature variation and gas composition (CH_4 and CO_2) are presented in Fig. 5. During this experiment, pH dropped only by 0.1 unit, TOC was stable and VFA were not detected. Gas production stabilised at 90 % of its normal value in 3 hours while hydrogen showed a little increase in concentration. It is important to note that, according to preceding experiments, the variation of CO_2 and CH_4 proportions are inverse. This phenomenon might be attributed to a significant dissolution of CO_2 . When the temperature returned to its normal value, gas production increased immediately, but its CO_2 and CH_4 content took a slightly longer time to go back to their normal values.

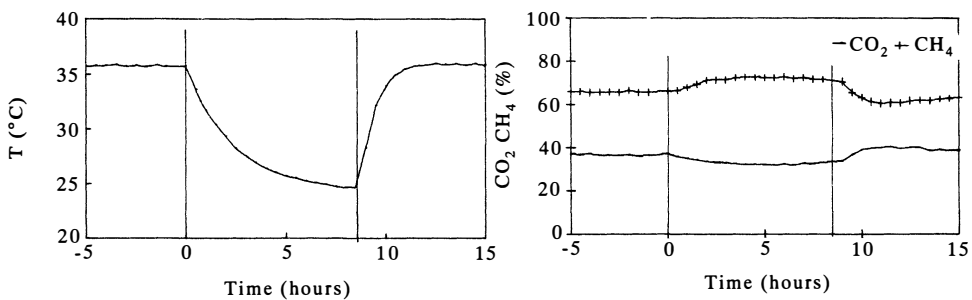


Fig. 5. Gas composition variation (CO_2 and CH_4) when temperature dropped from 35 to 26°C.

In this experiment, it is obvious that the microbial ecosystem did not experience a serious impact. A temperature drop can have severe impacts and can even cause an accumulation of VFA (Cayless *et al.*, 1989) which takes time to go back to initial values.

Influence of substrate change. The change from wine distillery wastewater to a glucose solution produced an organic shockloading on the reactor. Fig. 6 shows the variation of gas production and composition.

The hydrogen content in the gas, which was about 30 ppm before the shockload, increased slightly about ten hours after the shockload (finale concentration : 50 ppm). The other parameters were generally constant. The transitory variation of gas production is supposed to correspond with the time necessary for the microbial ecosystem to adjust its metabolic patterns. The rapid recovery means that the microorganisms present in the ecosystem did not undergo a fundamental modification. The impact of feeding depends on its nature. For example, the performances obtained from granular sludges acclimatized with sucrose can deteriorate when the digester is fed with another carbon source (Morgan *et al.*, 1990).

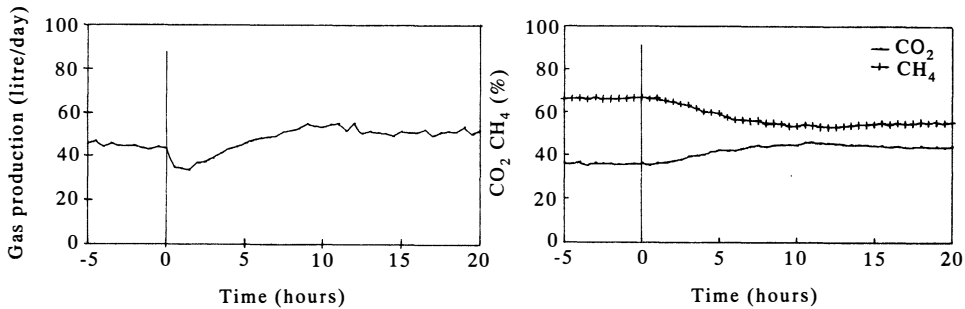


Fig. 6. Variation of gas composition and production rate following a change of feed : the wine distillery wastewater was replaced with a solution of glucose of same COD from $t = 0$.

CONCLUSION

In this work, we examined the response of certain parameters at organic shockloads of different origins, common in industrial plants (the impact of toxic elements in the digester is presently being studied).

The parameters studied rapidly reacted (generally, less than 1 hour after a shockload). They can thus be used as indicators for an automatic control of the process.

In our opinion, the choice of parameters should be based on the simplicity of utilization in a given installation. For a rapid detection of defects, it is necessary to choose many complementary parameters. The presence of hydrogen in the gaseous phase is a very important indicator. Nevertheless, it should be in relation to a close

monitoring of the proportion of CH₄ and CO₂ and of the gas production rate.

The variations of these parameters can be used as an algorithm for the control of the process. The measure of pH in liquid is simple and, knowing its impact on the biological activity, its value can serve as a detector of shockloads.

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