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EVALUATION OF A FOUR YEAR EXPERIENCE WITH A FULLY INSTRUMENTED ANAEROBIC DIGESTION PROCESS

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ABSTRACT

For several years, a 1 m^3 fixed bed anaerobic digestion process has been operated for the treatment of distillery vinasses. This reactor has been fully instrumented with the following variables available on-line : pH, temperature, liquid and gas flow rates, gas composition (*i.e.*, CH_4, CO_2 and H_2), concentration of bicarbonate, chemical oxygen demand, total organic carbon, volatile fatty acids and partial and total alkalinity, these last four variables being measured twice by different techniques (*i.e.*, using a TOC analyzer, a titrimetric sensor and an infrared spectrometer). The purpose of this paper is to compare the respective benefits of advanced instrumentation for the monitoring of wastewater treatment processes in general, and for anaerobic digestion in particular. It will also provide some statistical analysis of the time required to operate a fully instrumented wastewater treatment process. It is indeed well admitted in the literature that instrumentation is usually the main limitation step for using closed-loop control. However, it is our opinion that, in a near future, this situation will change. This point is discussed based on our four years practical experience.

KEYWORDS

Anaerobic digestion; instrumentation; on-line sensors.

INTRODUCTION

The anaerobic digestion process is based on a complex ecosystem of anaerobic bacterial species that degrade the organic matter. It presents very interesting advantages compared to the traditional aerobic treatment : high capacity to degrade difficult substrates at high concentrations, very low sludge production, low energy requirements and possibility for energy recovery through methane combustion.

At the industrial scale, anaerobic digestion processes have been widely used since the late seventies and today, more than 1,300 digesters are referenced world-wide (Totzke, 1999). Many of these processes are applied for the treatment of residues from agro-industrial (*i.e.*, sugar, corn processing…) and beverages industries (*i.e.*, beer, wine, canning, distilleries, …) although many other types of wastes from other origins are considered (*e.g.*, petrochemical, wood processing, …).

However, many industrials are still reluctant to use anaerobic treatment plants, probably because of the counterpart of its efficiency : it can become unstable under some circumstances. Disturbances like variations of the process operating conditions can lead to a destabilization of the process due to accumulation of intermediate toxic compounds resulting in biomass elimination and several weeks to several months are then necessary for the reactor to recover (see for example (Steyer *et al.*, 2001a)). During this period, no treatment can be performed by the unit. It is therefore a great challenge for instrumentation and control sciences to make this process more reliable and usable at industrial scale. The first step – and maybe the most important one – is to be able to follow dynamically (*i.e.*, using on-line sensors) the key process variables.

When studying more carefully industrial applications of anaerobic digestion processes, one can notice that, most of the time, their management is rudimentary. The sensors are indeed very basic (*i.e.*, temperature, pH, liquid and gas flows) and the processes are mostly run in open loop. This situation contrasts with recent results obtained in activated sludge processes where evidence has been brought that they could be optimized and that efficient pollutant removal could be achieved (Thornberg and Thomsen, 1994).

Within the last ten years, a great effort has been done to improve instrumentation for anaerobic digestion processes (Vanrolleghem, 1995) with specific attention to hydrogen in the gas phase (Archer *et al.*, 1986; Pauss and Guiot, 1993; Guiot *et al.*, 1995; Strong and Cord-Ruwisch, 1995) and VFA and bicarbonate concentrations (Rozzi *et al.*, 1985; Hawkes *et al.*, 1993; Hawkes *et al.*, 1994; Guwy *et al.*, 1994).

In order to highlight the benefits of using on-line sensors for anaerobic digestion processes, this paper presents a fully instrumented pilot scale reactor that is operated for the last four years treating industrial raw distillery effluents. This paper is organized as follows. First, the process and the related on-line sensors are presented. In a second part, a typical example of data recorded on-line is detailed and analysis of time requirements for the operation of the process will be discussed.

THE ANAEROBIC DIGESTION PROCESS AND ITS INSTRUMENTATION

An up-flow anaerobic fixed bed process of 1 m^3 was used in the present study. Its schematic layout is shown in Figure 1. The reactor is a circular column of 3.5 m in height, 0.6 m in diameter and has a total volume of 0.982 m^3. The support used (*i.e.*, Cloisonyl: 180 m^2/m^3 specific surface) fills 0.0337 m^3 leaving 0.948 m^3 of effective volume. The support creates 135 m^2 of surface.

Three storage tanks (27 m^3 each) are connected to a home-made 0.2 m^3 dilution tank (see Figure 2) by a piping system of 0.1 m of diameter and 60 m long. The effluents are raw industrial wine distillery wastewaters obtained from local wineries in the area of Narbonne, France. Neither sterile nor homogeneous, this wastewater has changing characteristics as depicted in Table 1.

Connected to the dilution tank is a remotely controllable peristaltic pump which ensures the desired influent flow rate fed into the reactor. Fresh substrate is mixed with the recycled liquid just before entering the heat exchanger (which regulates the temperature to 35°C). The heated liquid is then introduced at the bottom of the reactor where it is homogenized by the mixing pump.

Two pH measuring and regulating systems (Hanna instruments) are used (*i.e.*, in the dilution system and in the recirculation loop). They are made of a pH sensor, a PID hardware controller, an NaOH storage tank and a dosing pump. The addition of the soda can be performed either in the dilution system or in the recirculation loop just before the heat exchanger.

In the liquid output of the reactor, there is degassing system and a gas evacuating system. The liquid from the top of the reactor is collected by overflow in a receiving vessel. Some of this liquid is recycled at about 150 l/h and the rest is sent to the sewer. The input and recycling liquid flow rates are measured by two electromagnetic sensors (Khrone) with normalized analog outputs (4-20 mA).

Table 1. Characteristics of the wine distillery wastewaters

Component	Range
Total COD (g/l)	[12 – 34]
Soluble COD (g/l)	[7.6 – 31]
Volatile Fatty Acids (g/l)	[1.5 – 10]
Total Suspended Solids (g/l)	[2.4 - 10]
Volatile Suspended Solids (g/l)	[1.2 – 5.4]
Phenol (mg/l)	[90 – 275]
pH	[4.5 - 5.2]

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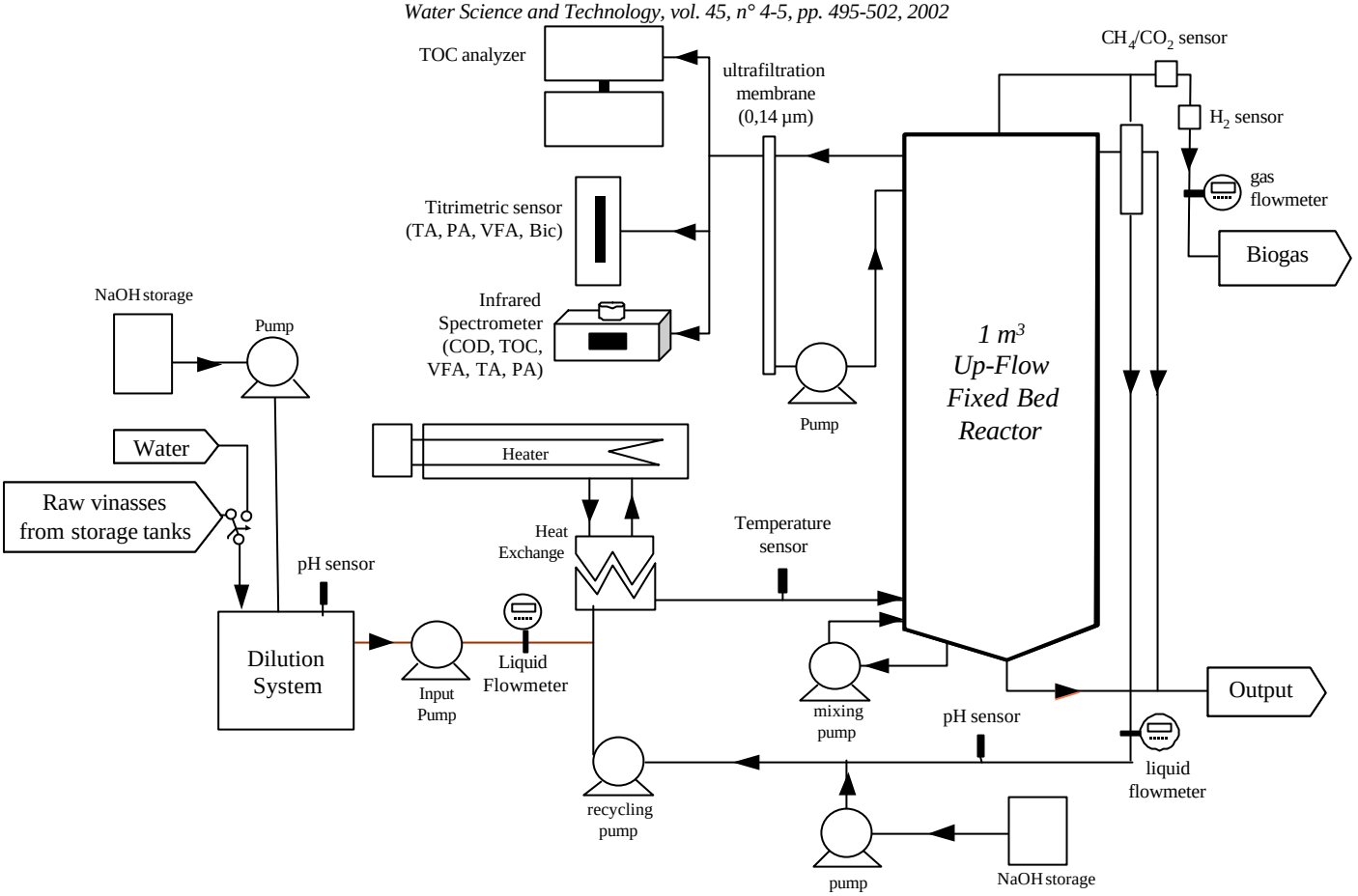

Figure 1. Schematic layout of the up-flow anaerobic fixed bed treatment plant.

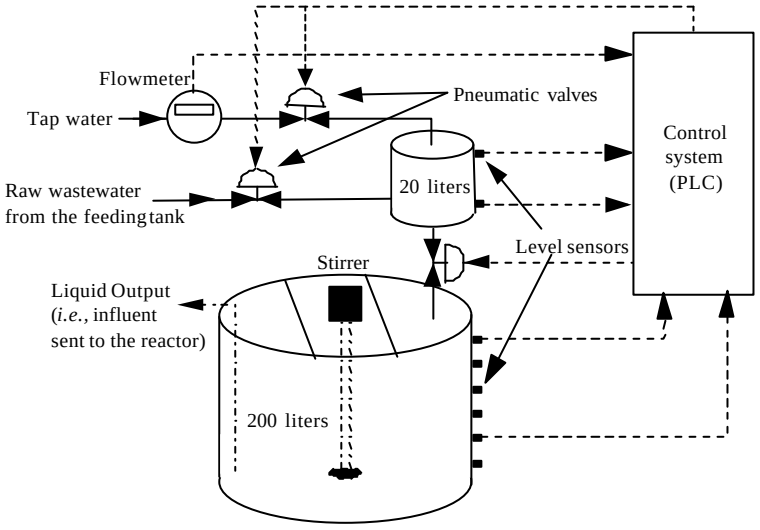

Figure 2. Schematic representation of the dilution system for the feeding of the reactor

The gas analyzing loop (see figure 3) is composed by a dryer which eliminates the humidity by cooling down the gas. The gas flowmeter is located at the output of this loop. It uses an electromagnetic floater to measure the produced gas continuously. Hydrogen concentration in the gas phase is measured by an AMS 6400 H_2 analyzer (Pekly Hermann-Moritz). This sensor is very sensitive to H_2S contained in the gas, so a "purafil" trap, which changes its color when becoming saturated with H_2S, was added. A peristaltic pump was also installed to ensure a fixed gas flow rate throughout the cell (electrochemical cell). It uses capillary diffusion, which has the advantage of a low temperature coefficient, a direct indication of the concentration (in ppm) and consequently, the influence of pressure over the measure is low. The Ultramat 22P sensor (Siemens) measures the CO_2 and CH_4 percentage composition of the analyzed gas. This sensor works on the principle of the nondispersive absorption of infrared light, that is a one beam method with a two-layer optopmneumatic detector.

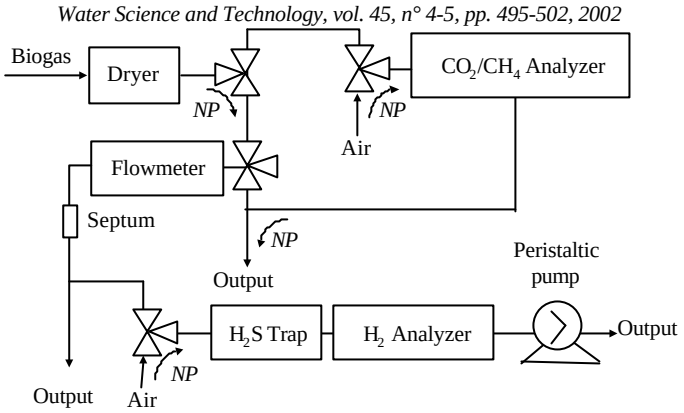

Figure 3. Schematic description of the output gas flow pattern (NP = "Normal Position").

At the top of the reactor, an extra loop has been installed with an ultra-filtration module (a 10 mm diameter tubular ultrafiltration membrane from Tami Industries with a cut off of 0.14 µm and a surface of filtration of 0.045 m^2). The membrane gave continuously 0.5 l/h of filtered sample with only one maintenance cycle required every two weeks on average. Three on-line advanced sensors are connected to this loop :

- a Zellweger analytics autoTOC 1950 allows the measurement of the Total Organic Carbon (TOC). This equipment has a measurement range from 0 to 2000 ppm performing a continuous analysis of the sample,
- an on-line automatic titration equipment developed in our laboratory is also connected to the ultra-filtration loop. This titrimetric sensor allows us to have partial and total alkalinity measurements every 3 minutes if required. It also estimates on-line the bicarbonate and Volatile Fatty Acids (VFA) concentrations in the output of the reactor (*Cf.* Figure 4 for a comparison of on-line measurements of VFA using the titrimetric sensor and manual off-line analysis by gas chromatography),
- last but not least, a Fourier Transform InfraRed (FT-IR) spectrometer (*i.e.*, Avatar spectrometer of the Nicolet company) working in the mid-infrared range (*i.e.*, wavelength between 2.5 and 25 µm) was modified in our laboratory (*i.e.*, to remove temperature effects in particular) and connected to the ultrafiltration loop. We demonstrated that such a sensor could provide us with precise and reliable measurements of soluble Chemical Oxygen Demand (COD), TOC and VFA concentrations as well as total and partial alkalinity (Steyer *et al.*, 2001b).

All these sensors are connected to an input/output device and managed by a freely available software developed in our laboratory to perform advanced control law calculations as well as process supervision in link with Matlab®. Two other computers are used : one for the monitoring of the titrimetric sensor and one for the FT-IR spectrometer.

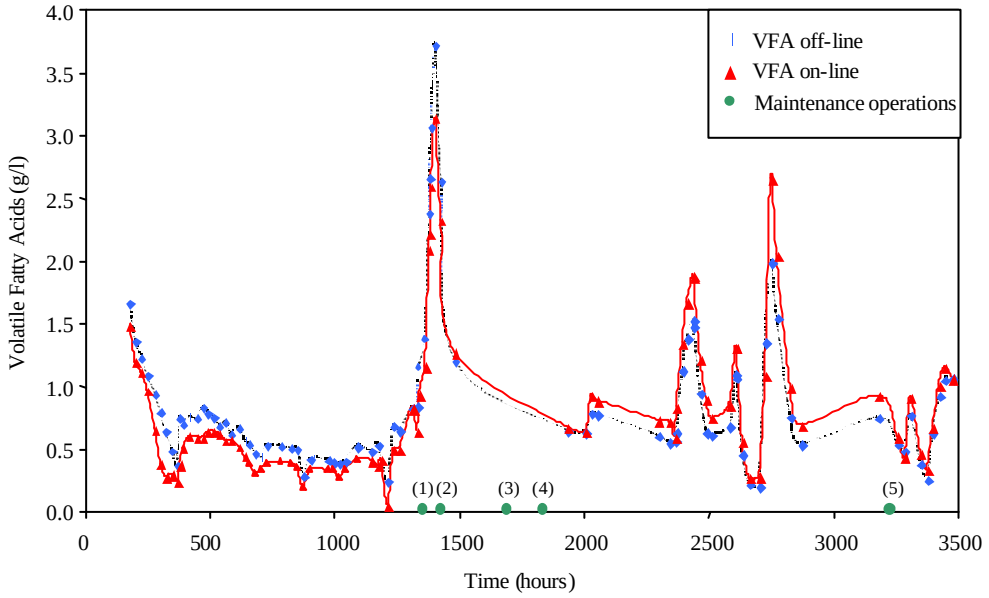

Figure 4. Comparison over a 5 month period of volatile fatty acids measurements using manual off-line analysis by gas chromatography and automatic on-line measurements provided by the titrimetric sensor (the main manual operations for maintenance of the titrimetric sensor are shown on the time axis)

RESULTS

All the measurements are recorded every 2 minutes even though the H_2 sensor, the titrimetic sensor and the FT-IR spectrometer perform series of measurements every half hour. This was chosen because of practical requirements (*e.g.*, the H_2 signal has to go down to zero before another measurement) or to lower the maintenance effort (*i.e.*, for the titrimetic sensor and the FT-IR spectrometer) while being fully compatible with the dynamics of the process.

A typical week of data recording is presented in Figure 5 together with the main events and their related effects that occurred during this 200 hour period (see numbers in circles in Figure 5). The first column presents the typical measurements in industrial applications (*i.e.*, pH in the input and in the reactor, influent liquid flowrate, temperatures in the reactor and in the heater). The four first curves in the second column illustrate what could considered as advanced instrumentation nowadays in industrial applications (*i.e.*, recirculation liquid flow rate, output gas flow rate, CO_2/CH_4 percentage in the biogas, H_2 in the biogas and pressure at the top of the reactor). Finally, the last curve of the second column and the third column show the results obtained with the on-line TOC analyzer, the titrimetric sensor and the FT-IR spectrometer. Events during this 200 hours period are the following :

1. First, a bad regulation of the reactor due to a disconnection of the pump that circulates the heated water in the heat exchanger can be noticed for the first 24 hours. Temperature of the reactor was then not regulated at 35 °C as it is supposed to be. However, because of the not too long period of time, no effect on other variables could be noticed,
2. A problem appeared in the feeding system (*i.e.*, no more influent was sent to the dilution system because of a mechanical device was "stuck", the pH sensor in the dilution system was then above the liquid and the pH regulation system added too much soda). The pH in the influent went up to 12 but a safety rule was then automatically activated to protect the process by stopping the input liquid flow rate (for more information about the building of these safety rules, see (Steyer *et al.*, 2001a)). Again, because of the short duration of this problem, no effect was noticed on the overall process. However, if an appropriate safety rule was not activated, it could have largely affected the process (*i.e.*, stop of the process for about a month as illustrated in (Steyer *et al.*, 2001a))
3. A leak appeared in the feeding pipe (*i.e.*, between the dilution system and the input pump) and then, no influent was introduced in the reactor but air instead. Because it appeared during the day, this problem could be solved rapidly and it only had small effects on the overall process (see for example the output gas flow rate and the gas composition). Also, because of the system configuration, the recirculation liquid flowrate decreases rapidly when no influent is added to the reactor (the ultrafiltration loop takes indeed samples continuously) and when the recirculation flow rate is equal to zero, temperature regulation cannot be performed.
4. Foam appeared at the top of the reactor and it disturbs both the output biogas measurements but also the pressure and sometimes and CO_2 and CH_4 measurements. This is usually solved by adding automatically anti-foam (see at t=30 h and t=45 h) but this is not always the case. For example, at t=130 h, the electrical contact was not made between the foam and the level sensor (*i.e.*, the foam was then not "dense" enough) and no anti-foam was added. The consequence is quite catastrophic since then, some foam entered the pipes and at t=150 h, the connected sensors (*i.e.*, the output gas flowmeter, the pressure, the CO_2/CH_4 analyzer and the H_2 analyzer) were out of order and a manual cleaning of these sensors was necessary.
5. Several changes of the input liquid flow rate were performed on purpose to disturb the process (*i.e.*, at t=50, 78, 125 and 155 h) and to analyze their effects on the other variables (see for example the decrease of the pH in the reactor and the increase of the hydrogen at t=50 h because of the overloading of the reactor). Effects on the COD, TOC, VFA and alkalinity can also be noticed in the last column of Figure 5.
6. This point is due to clogging of the recirculation loop that can be either slow (*i.e.*, between t=55 h and t=90 h) or fast (*i.e.*, at t=125 h). This is to be solved rapidly to avoid too large disturbance of the hydrodynamics of he reactor.
7. Cleaning of the ultrafiltration membrane. This has to be performed every two weeks at the maximum to avoid clogging of the membrane.

8. Software problem on the computer specifically in charge of the FT-IR spectrometer. This happens sometimes without any clear explanation of the reason. The consequence is just that no measurement can then be performed by the spectrometer during this period and the solution is just to reboot the computer.
9. Weekly cleaning of the FT-IR spectrometer (*i.e.*, between t=145 h and t=155 h) and of the titrimetric sensor (*i.e.*, between t=175 h and t=195 h). These operations can be performed automatically or manually. Usually, they do not last too long (*i.e.*, less than half a hour) but then, it was decided to run the cleaning over night.
10. Problems with the TOC on-line analyzer. Its measurement range is indeed between 0 and 2 g/l and when it gets closer to the higher limit, the signal becomes noisy (*i.e.*, between t=60 h and t=80 h) and even saturated when it reaches the limit (*i.e.*, between t=150 h and t=160 h).

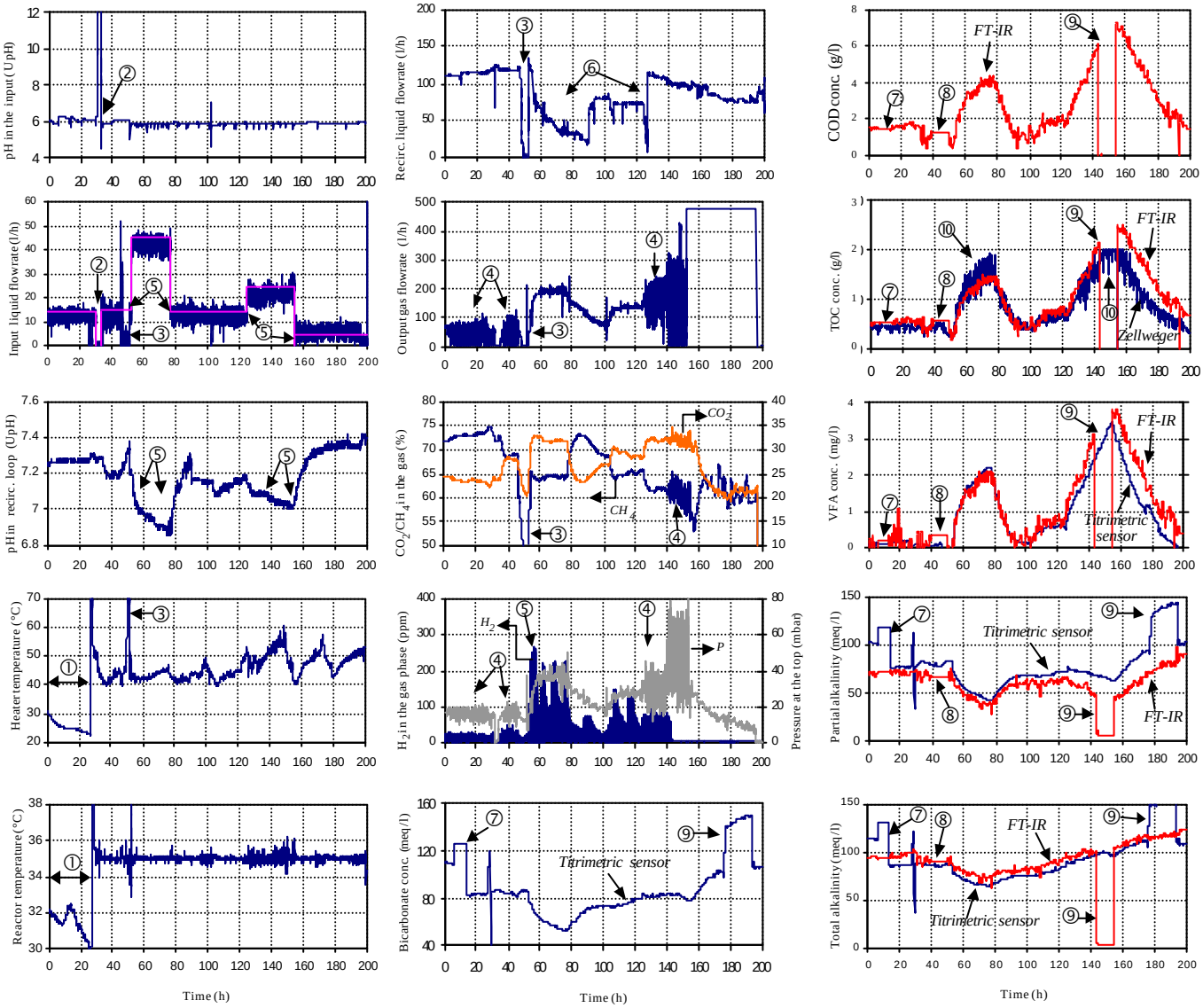

Figure 5. An example of data recorded on-line on the pilot scale anaerobic digestion process used in the present study. Numbers in circles indicate the main events that occurred during this 200 hour period

DISCUSSION

The process used in the present study has been operated since February 1997 with, from the beginning, typical sensors used in industrial applications (*i.e.*, input liquid flow rate, temperature and pH in the reactor, gas flow rate). Few weeks after, we decided to add additional sensors :

- temperature of the heater : indeed, when the heating system is working well, the signal of the reactor temperature – because constant at 35 °C – is not enough to forecast temperature problems before they

occur. However, by measuring the actuator signal (in this case, the temperature of the heater), we can predict either a problem in the regulation system itself (*e.g.,* the temperature heater is saturated to its maximum value) or a clogging of the heat exchanger (*e.g.,* the temperature heater slowly increases until it reaches its maximum value).

- recirculation flow rate : it is very important to know the hydrodynamics inside the reactor since it can largely affect the behavior of the overall process. Adding a flowmeter in the recirculation loop allowed us to explain many phenomena that could not be explained without this measurement.
- pH in the input : in our case, the main reason for measuring the pH in the input was to insure better treatment performance of the process by increasing the pH before entering the reactor (distillery wastewaters have indeed a low pH).

These sensors provided us with very useful information but they were not enough to monitor our anaerobic digestion process in an optimal and reliable way. We thus decided to add sensors in the gas phase (*i.e.,* biogas composition in terms of CO_2, CH_4 and H_2) because reliable, easy to implement and not too expensive. As discussed in many papers in the scientific literature, these sensors were demonstrated to be very interesting to predict evolutions like the overloading of the process. However, they provide us with information only from the end of the reaction scheme (*i.e.,* the biogas is indeed the final product of the anaerobic digestion process) and thus, they were not enough to accurately predict events like VFA accumulation before they occur.

Our attention was then focused on instrumentation systems that would allow us to "get closer" to the microorganism activity. For the last three years, we then implemented an on-line TOC analyzer available on the market and a titrimetric sensor that was developed in our laboratory. These sensors fulfilled perfectly our main objective : to control a system that is widely recognized as very sensitive and very difficult to operate. The knowledge of TOC, VFA, bicarbonate concentrations and total and partial alkalinity indeed allowed us to avoid destabilization of the anaerobic digestion process while maintaining very high removal efficiency.

However, the two above sensors are based on automation of chemical reactions and as such, they require maintenance operations that limit their application in an industrial context. On the other hand, the last sensor (*i.e.,* the FT-IR spectrometer) is only based on spectral analysis of a sample. As a consequence, it does not require any chemical to be added and very low maintenance effort : implemented on-line for almost one year, we never had any problem in operating this sensor (it requires indeed a maintenance effort similar to the one required by pH probe !) and it provided us with very reliable multi-parameter measurements (*i.e.,* COD, TOC, VFA, total and partial alkalinity). Its only drawback is its heavy calibration procedure – quality of the on-line measurements are indeed closely correlated to this step – but our last results (not shown here) demonstrate that this calibration could be reduced to be fully compatible with industrial requirements.

From a more general perspective, it is worth noting that an anaerobic digestion process – when fully instrumented – is not difficult to operate in optimal conditions but it requires on average 2 hours per day for the maintenance of the process. However, this requirement is mainly due to typical problems in the wastewater treatment field (*e.g.,* pipe clogging, foam, …) and not related to the use of advanced sensors like the TOC analyzer, the titrimetric sensor or the FT-IR spectrometer (which mostly induce careful maintenance of the ultrafiltration membrane). Yet this effort is worth doing since it shows – dynamically – the process evolutions and it allows one to efficiently and optimally manage the overall anaerobic digestion process (see for example (Harmand and Steyer, 2001b)).

CONCLUSION

The wastewater treatment field suffers from a major lack of sensors, both reliable and highly informative. The problems with current monitoring technology is that a difficult choice must be made between either reliable, low information content sensors (*e.g.,* temperature and pH probes, gas flowmeters) and, alternatively, high information content but usually recognized as fragile measuring devices (*e.g.,* TOC, COD and VFA sensors for anaerobic digestion processes).

However, it is our belief that the situation will change in the next future. Indeed, advanced sensors still require maintenance effort that could be difficult to handle at the industrial scale but it is the only solution if one wants "to get closer" to the microorganisms and to explain situations that cannot be explained using traditional manual off-line analysis. In addition, it offers very large perspectives for the efficient and optimal monitoring of the processes.

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