

## Optimisation of biogas production from anaerobic digestion of agro-industrial waste streams in Brazil

M. A. Boncz, L. Pinheiro Bezerra, C. Nobuyoshi Ide and P. Loureiro Paulo

### ABSTRACT

The important Brazilian agro-industry produces significant amounts of wastewater with high concentrations of biodegradable compounds. A lot can be gained if wastewater treatment would take place using anaerobic reactors instead of the anaerobic lagoons generally used now. Apart from preventing methane emissions to the atmosphere this would permit the use of the biogas as a source of energy. To facilitate implementation of this technology also in small and intermediate sized companies a system requiring only minimal maintenance is needed. The need for maintenance by skilled labour can be reduced using an automated process control system, which is being developed. Cassava (manioc, tapioca) processing wastewater has been treated in a lab scale UASB reactor equipped with an on-line monitoring system, to test a control strategy based mainly on pH control. Good results have been obtained treating not only pre-acidified but also treating raw (diluted) cassava processing wastewater.

**Key words** | agro-industry, biogas, cassava, wastewater

M. A. Boncz  
L. Pinheiro Bezerra  
C. Nobuyoshi Ide  
P. Loureiro Paulo  
Department of Hydraulics and Transport,  
Federal University of Mato Grosso do Sul,  
Cidade Universitária s/n,  
79070-900 Campo Grande, MS,  
Brazil  
E-mail: mboncz@nin.ufms.br

### INTRODUCTION

In the Brazilian state of Mato Grosso do Sul, the principal economical activity is the production and processing of agricultural products, like grains, sugarcane, beef and cassava (tapioca, manioc) (Saldanha *et al.* 2003). At the same time, the agro-industries produce large volumes of concentrated, but well biodegradable wastewaters, generally treated using low-tech systems like anaerobic ponds. There is a great potential for using these wastewaters for energy production, when the treatment would occur using UASB or EGSB type bioreactors, not only promising improvements from an environmental (reduction of greenhouse gas emissions and surface water pollution), but also from an economic point of view (given the trend of rising energy prices, and the high local price of electricity). As the local industries involved often lack technically skilled staff for permanently supervising a wastewater treatment plant, it would be beneficial to implement the wastewater treatment and energy generation plant using some degree of automation, thus reducing the need for supervision.

A system for on-line monitoring and control was developed and is being tested using a lab-scale UASB reactor. The system will be applied for the treatment of effluents of several different types of agro industries, like the ethanol distilleries that are being installed in large numbers to supply transportation fuel. Initially, the system has been tested using cassava processing wastewater (“manipueira”). Apart from serving as a demonstration unit when installed *in-situ*, the system is also intended to provide more accurate data on agro-industrial wastewater characteristics over time, as very few accurate data on the amounts and composition of the wastewaters produced in the state are actually available.

Cassava processing wastewater is a high-strength wastewater composed mainly of sugars, which may also contain, depending on the species of cassava processed, a significant concentration of cyanide (Barana & Cereda 2000; Mai 2006). Anaerobic digestion of this wastewater takes place in two steps: acidification of the sugars to form

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volatile fatty acids (VFAs) is followed by a slower methanogenic step, converting the acids formed into methane and CO<sub>2</sub> (Chernicharo 2003). Treatment problems arise from the acidity produced in the acidification step, which runs at a very high rate due to the easily biodegradable sugars; anaerobic treatment of cassava processing wastewater in one step without addition of alkalinity is therefore generally considered not feasible (Barana & Cereda 2000). In some cases cyanide inhibition of the anaerobic sludge may occur although moderate (Colin *et al.* 2007) or even high concentrations of cyanide (up to 25 mg.l<sup>-1</sup>) were shown not to be inhibitive (Annachhatre & Amornkaew 2001).

If the pH is allowed to drop too much, methanogenesis will be inhibited, diminishing the process efficiency and biogas formation rates. This can be avoided using one of three approaches: (i) application of a two-step process with an acidification reactor and buffering, followed by a second digestion step or (ii) applying lower OLRs and (automated) process control to avoid inhibition of the methanogenic biomass in the reactor (Holubar *et al.* 2002). Option (iii), addition of alkalinity by for instance dosing bicarbonate (Paulo *et al.* 2003) is generally considered too expensive.

Objective of this research is to establish a control strategy for anaerobic treatment of agro-industrial wastewaters, optimising COD removal efficiency whilst at the same time maintaining a constant biogas composition to enable its use in energy generation, and reducing the necessity of human supervision of the process.

## MATERIALS AND METHODS

### Batch experiments

Batch experiments were performed according to the procedure outlined by Owen and Stuckey (Owen *et al.* 1979) with 400 ml solution in 500 ml serum bottles with rubber septa. The solutions contained 2.0 g.l<sup>-1</sup> VSS, 2.0 g.l<sup>-1</sup> of COD, and were kept at 30°C in thermostated orbital shakers operating at 50 rpm. After filling the bottles, the headspace was purged for 5 minutes with N<sub>2</sub> (White Martins), or with a mixture of 30% CO<sub>2</sub>/70% N<sub>2</sub> (White Martins) in case a

buffered solution (buffer: 4 g.l<sup>-1</sup> carbonate) was used. Gas production and COD content were monitored at least on a daily basis using the gas displacement method and the titration according to “Standard Methods” (American Public Health Association 1992) respectively.

### Continuous experiments

A 40-litre UASB reactor used for the continuous experiments was equipped with meters for pH (Provicec Dosa-Tronic PH1000 Top with P226C electrode), Redox Potential (Provicec DosaTronic MV1000 Top with P556C electrode) and temperature (made in-house), all connected to a PC through a MCC USB1208-LS data acquisition module. The influent–cassava wastewater periodically collected at a local cassava processing plant, stored at 20°C until use, and diluted before use was pumped to the reactor at a variable rate using a peristaltic pump (Watson Marlow 505S), whilst its turbidity was monitored on-line using Dr. Lange Solitax turbidity meter. The biogas produced was collected, and its flow and composition monitored on-line using a tumbling-cell type gas flow meter (made in-house) and a Madur MadIR CO<sub>2</sub>-detector respectively. The seed sludge for the reactor was collected from a UASB reactor treating domestic wastewater (50%) and from an anaerobic pond receiving wastewater from a slaughterhouse (50%).

The reactor was operated both in continuous and in batch mode. In batch mode, methanogenic activity and sensor output were tested using sodium acetate as the sole substrate. In continuous mode, on-line monitoring and process control was tested, while the reactor was fed with pre-acidified and neutralised cassava-processing wastewater at an OLR of between 0.75 and 1.5 gCOD.l<sup>-1</sup>.d<sup>-1</sup> (experiment I) (COD content ranging from 0.6 to 10 gCOD.l<sup>-1</sup>) and with raw, diluted, cassava processing wastewater at an OLR of around 0.75 gCOD.l<sup>-1</sup>.d<sup>-1</sup> (experiment II) (COD content between 2 and 6 gCOD.l<sup>-1</sup>). Process control during experiment II was carried out using a crude adjustment of influent flow based on pH variations and biogas production.

Analysis of COD, acidity, alkalinity and volatile fatty acids were carried out using titrations as described in “Standard Methods” (American Public Health Association 1992).

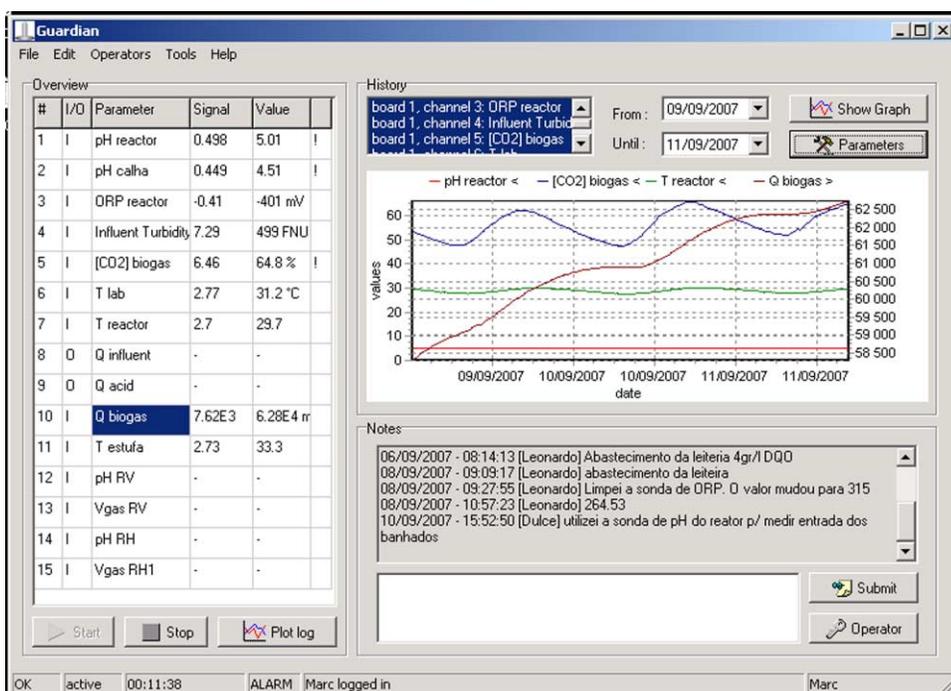


Figure 1 | Screen capture of data acquisition/processing software “Guardian” running.

## Data acquisition

For on-line data acquisition, data processing and process control specialised software (“Guardian 1.2”) was written in-house, built upon drivers for the data acquisition module provided by MCC Computing® (it will support a wide variety of MCC data acquisition boards and modules), and upon interfaces for both MS Access® and MySQL® databases, using Borland Delphi® 7. During the course of the described experiments, the software has been running on a PC running MS Windows 2,000®, but it will run on

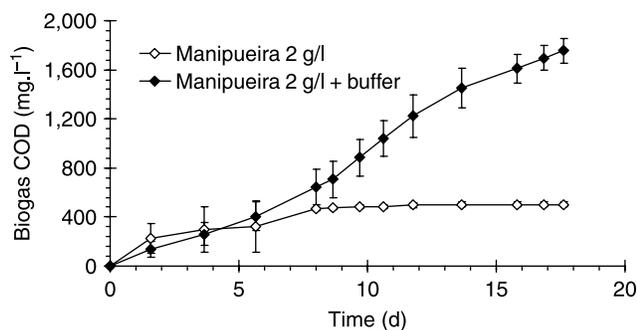


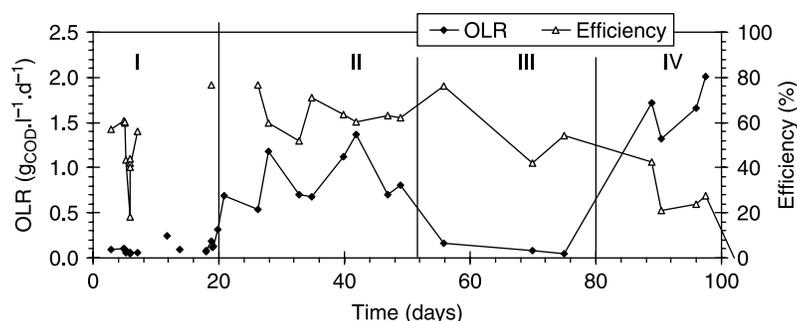
Figure 2 | Methane production from cassava processing wastewater ( $COD_0 = 2 \text{ g.l}^{-1}$ ) with and without buffering of the pH.

systems running MS Windows 98® and MS Windows XP® as well. Amongst its functionalities are -apart from data acquisition, processing and storage- maintaining a laboratory activities log for several identified operators and making data available for a web-based interface to enable monitoring the system from a distance, as well as making archives available in MS Excel® readable format (\*.csv archives).

Analogue output signals from third party equipment providers (like the measuring devices mentioned under “Continuous experiments”) are converted into real measurement units through programmable calibration functions (linear, 2nd order, logarithmic or exponential). Measurements can be supplied in units with their standard deviation due to a repeated sampling procedure. Operational alarm messages can be sent to one or more operators by email as well, if so configured. The software is bilingual (English and Brazilian Portuguese; it will switch interface language depending on the operator logged in), and the amount of available languages can easily be increased. A screen image from the running system is displayed below (Figure 1).

**Table 1** | Overview of experimental conditions applied

Exp.	Influent	Period	Time d	OLR $g_{COD} \cdot l^{-1} \cdot d^{-1}$	pH	Remarks
I	Pre-acidified wastewater	I	0–20	0.1	8.2	Acetate supplied to increase OLR
		II	20–56	0.8	6.2	
		III	56–80	0.1	7.0	
		IV	80–96	1.7	5.7	
II	Raw wastewater	I	52	0.6	6.1	
		II	8	0.5	5.3	

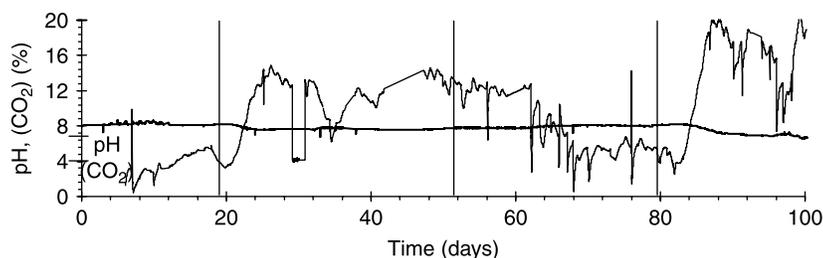
**Figure 3** | OLR and efficiency of UASB reactor fed with pre-acidified cassava processing wastewater.

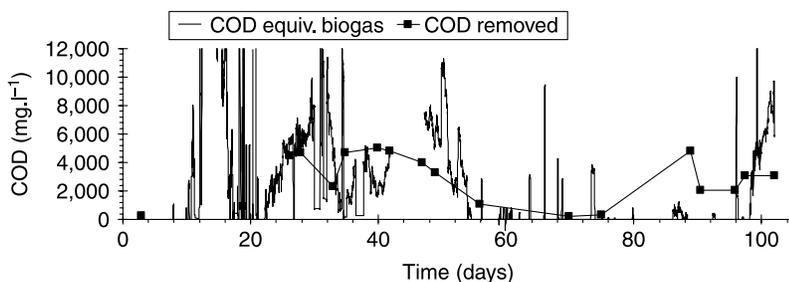
## RESULTS AND DISCUSSION

Both experiments confirm the results from earlier research that degradation of cassava processing wastewater is very well possible, but that the formation of high amounts of organic acids inhibit methanogenesis when no method to stabilise the pH is applied (Figure 2). Where in the non-buffered solution the pH dropped to 5.3, and methane formation was interrupted, the pH in the buffered solution remained stable at 7.2, and all organic matter was converted into methane gas. This illustrates again the importance of avoiding a strong decrease of the pH during reactor operation to avoid methanogenesis inhibition.

Two different continuous experiments were performed in which cassava processing wastewater was fed to the UASB reactor. During a first experiment (experiment I) the wastewater was pre-treated in an acidification reactor before being fed to the UASB, while in the following experiment (experiment II) the wastewater was fed to the UASB directly from refrigerated storage. Table 1 shows the different experimental conditions applied:

Figure 3 shows the organic loading rate and efficiency of the reactor during experiment I. Four phases can be distinguished: I, in which the concentration of COD in the influent is low, as is the OLR, II, in which addition of acetate to the influent results in a much higher OLR, III, in

**Figure 4** | pH and CO<sub>2</sub> concentration in the biogas produced by the reactor when fed with preacidified cassava processing wastewater.



**Figure 5** | Measured COD removal compared to COD removal calculated from  $\text{CH}_4$  production.

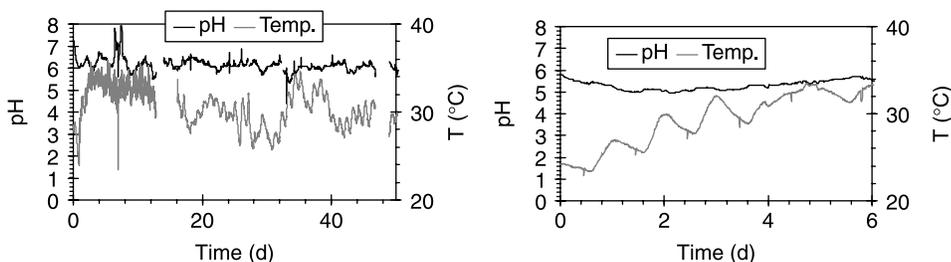
which acetate dosing is stopped again, and IV, in which the influent flow has been increased, causing a reduced efficiency of the pre-acidification reactor and a more concentrated influent for the UASB.

The COD removal efficiency was about 55% in the first three periods, dropping significantly upon introduction of the less well acidified influent. This is reflected in a relatively stable pH and  $\text{CO}_2$  concentration in the biogas, until the start of phase IV where the pH drops and the amount of  $\text{CO}_2$  in the biogas increases sharply (Table 1 and Figure 4). The redox potential measured in the reactor (not shown in the graph for being basically a straight line), stable at 490 mV until day 82, increases to  $-460$  mV in the last

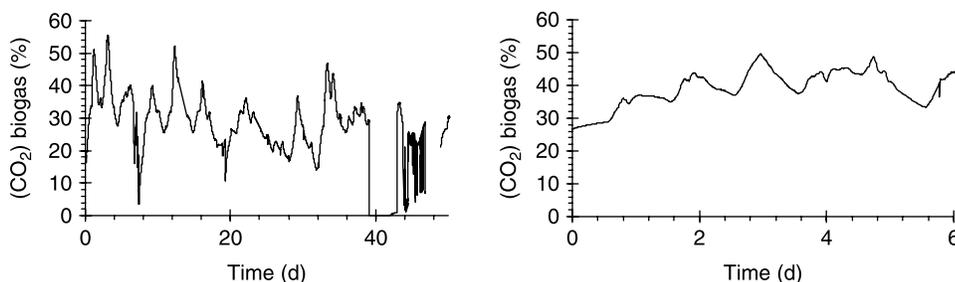
phase of the experiment, indicating anaerobic conditions suitable for methanogenesis during the whole experiment, although less favourable in the last phase.

On-line measured biogas formation (converted to  $\text{mg COD.l}^{-1}$  equivalents of the influent) reasonably matched COD removal (Figure 5), except during phase III, when problems occurred with the sealing of the gas separator.

During experiment II, the reactor was treating raw, diluted, cassava processing wastewater. The process was controlled varying the OLR adjusting the influent flow, in order to stabilise the pH. Removal efficiencies were higher (around 85%) than in the previous experiment, but at the cost of lower organic loading, and operation was much less



**Figure 6** | Variations in pH and operating temperature during experiment II, periods I (left) and II (right). In both graphs the pH is the thicker black line above, and the temperature the thinner grey line below.



**Figure 7** | Variations in biogas composition ( $\text{CO}_2$ -content) during experiment II, periods I (left) and II (right).

stable, with large variations of the CO<sub>2</sub> content of the biogas between 5 and 60%, accompanying smaller changes in the systems pH (Figures 6 and 7). This may be a problem if the intention is to use the biogas as a fuel (reduced caloric value and varying combustion stoichiometry complicating generator operation) and shows stabilisation of the pH of the process to be essential.

In these experiments also a clear effect of operating temperature could be seen, where lower temperatures reduce methane production and acidification, the tendency of decreasing pH indicating that methanogenic activity is more temperature-sensitive than acidification.

Figures 6 and 7 also show a clear relation between operating temperature and biogas composition (CO<sub>2</sub>-content) where lower temperatures seem to correspond to a lower concentration of CO<sub>2</sub> in the biogas.

## CONCLUSIONS

Wastewaters of agro-industries can be a source of energy when digested anaerobically. Apart from process layout, process control has a large influence on process performance, and on the possibility to use the biogas produced in the reactor for energy generation.

The main factors affecting biogas production and composition are operating temperature and process pH, apart from the organic loading. In full scale systems, operating temperature will be a less important factor.

Calibrating the system and defining control strategies using a fast acidifying wastewater like cassava processing wastewater, generally considered difficult to treat in a one-step anaerobic process, gives a good insight in how to deal with process control.

## ACKNOWLEDGEMENTS

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