Wat. Sci. Tech. Vol. 25, No. 7, pp. 213–222, 1992.
Printed in Great Britain. All rights reserved.

0273–1223/92 $15·00
Copyright © 1992 IAWPRC

THERMOPHILIC ANAEROBIC DIGESTION OF VINASSE IN PILOT PLANT UASB REACTOR

M. E. Souza*, G. Fuzaro** and A. R. Polegato**

**Biometano Consultoria, Brazil*
***Usina São Martinho, Fazenda São Martinho 14850, Pradópolis, SP, Brazil*

ABSTRACT

Vinasse from sugar-cane alcohol production is generated in great amounts in Brazil and is disposed on soil "in natura". Only a few distilleries use mesophilic anaerobic digestion, so far, for gas recovery and vinasse treatment or conditioning, due to an unfavorable economical balance.

Thermophilic anaerobic digestion was studied in a large-scale pilot-plant (75 m^3 UASB reactor), since vinasses are generated at high temperatures, and no experiments on this subject are reported in the literature, except for a few laboratory assays using pure substrates and other wastewaters.

This work demonstrates the feasibility of sugar-cane vinasse thermophilic anaerobic digestion in UASB reactors.

The reactor was operated for 280 days, as far as possible like a full-scale plant; at applied organic loading rates of 25-30 kg COD/m^3.day, 72% COD removal and 10 Nm^3gas/m^3.day could be achieved. Granulation of the sludge was essential in order to maintain good stability of the process; almost no soda was necessary for pH control.

For full-scale implementation, re-start of the thermophilic process, after several months without feeding, has to be studied yet.

KEYWORDS

Thermophilic Digestion; Anaerobic Digestion; Biogas; UASB reactor; Vinasse.

INTRODUCTION

Brazil produces about 1.2 x 10^{10} ℓ ethanol per year, from sugar-cane, generating large amounts of vinasse (about 1.5 x 10^{11} ℓ/year). The major part of this vinasse is applied in sugar-cane plantations for potassium recovery, without any previous treatment. So, depending on the region it represents serious risks to the environment. Furthermore, such a large amount of vinasse could generate some 1.2 x 10^9 Nm^3 CH_4/year by anaerobic digestion, which can be used as fuel for vehicles or to generate electricity. The feasibility of vinasse mesophilic anaerobic digestion has been proved, using a UASB (Upflow Anaerobic Sludge Blanket reactor) pilot plant, several years ago (Craveiro et al., 1982; Hirata et al., 1988). Nevertheless, only a few full-scale plants have been installed so far in Brazil, due to unfavourable economical balance, caused mainly by low relative costs of diesel and electricity. Furthermore, all these plants use mesophilic UASB reactors, which reach a relatively low maximum organic loading rate in practice (15 kg COD/m^3.day) (Craveiro et al., 1986; Hirata et al., 1988) and require much energy and equipment for cooling the vinasse, once it is generated at 85-90°C.

Considering the high temperature of vinasse and the advantages of thermophilic bacteria (Zinder, 1988) "Usina São Martinho", the major Brazilian alcohol producer (3.4 x 10^8 ℓ/ year), decided to develop, assisted by "Biometano", thermophilic anaerobic digestion in a high-rate UASB reactor. The objective was at least a duplication of the maximum mesophilic organic loading rate (consequently reducing the total reactor volume necessary in the distillery from 40,000 to 20,000 m^3) and decreasing the heat exchange cost. With the produced biogas "São Martinho" could, for instance, generate electrical energy corresponding to 27 MW.

Although the feasibility of thermophilic anaerobic digestion of pure substrates and high strength wastewaters has already been proved in laboratory-scale UASB reactors (Wiegant and Lettinga, 1985; Wiegant et al., 1985), no reports were found in the literature concerning pilot or full-scale experiments, especially treating vinasses. Therefore, it was impossible to apply a thermophilic process, before studies at a larger scale were made.

A large pilot plant with a 75 m^3 thermophilic UASB reactor was constructed, installed and operated as far as possible like a full-scale plant, to allow easy equipment and process scale-up. The first attempt at operation (1988/1989) was not sucessful.

This work presents and discusses the second attempt to operate the pilot plant, during 1990.

MATERIALS AND METHODS

Description of the Pilot Plant

The experiments were carried out in a 75 m^3 (70 m^3 net volume) UASB reactor (height - 6 m and diameter - 4 m), made of epoxy coated carbon steel. Thermal insulation with glass wool was provided. In the reactor there were 12 feed inlet points, 6 sampling points and 4 temperature measurement probes. The UASB reactor was designed according to criteria described before (Souza, 1986). Temperature was automatically controlled at thermophilic range (55-57°C), through the heating or cooling of the influent (vinasse + recycle).

The vinasse was collected continuously, directly from the distillery and sent to feeding tanks (2 tanks of 10 m^3 each). The mixture of vinasse, part of the effluent (recycle) and chemicals (urea, phosphoric acid and soda when necessary) was pumped to the heat exchanger and then to the digester. The sludge wasted with the effluent was collected in a separate tank, after settling, for future use in other units. The biogas was collected, cooled for water and humidity removal, before flow measurement, and sent to a flare.

Figure 1 shows a schematic diagram of the pilot plant.

Characteristics of the Vinasse

In "Usina São Martinho" vinasse is generated from alcohol production, using as substrates, mixtures of sugar-cane juice and molasses. When the vinasse for the pilot plant operation was collected directly from the distillery (in natura), it was available at temperatures around 90°C.

During the major part of the experiments (sugar-cane harvest), vinasse "in natura" was utilized (from May, 30, 1990 until Nov, 7, 1990). However, during inter-harvest (from Feb, 19, 1990 until May, 29, 1990 and from Nov, 8, 1990 until Nov, 25, 1990), concentrated vinasse, after dillution in tap water to reach the mean characteristics of vinasse "in natura", was utilized (Figure 2).

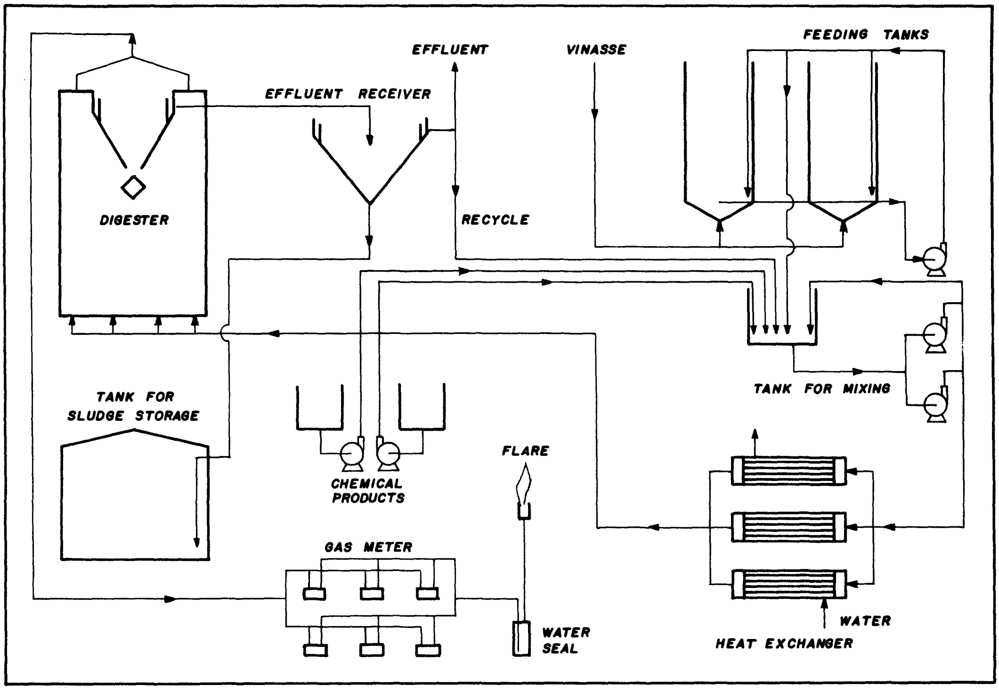

Fig. 1. Schematic diagram of the thermophilic UASB pilot plant.

(1)	(2)	(1)
100 days	162 days	18 days

(1)- Inter-Harvest (operation with concentrated vinasse, after dilution)
(2)- Harvest (operation with vinasse "in natura")

Fig. 2. Periods of operation with different types of vinasse

The mean characteristics of vinasse are presented in Table 1, for the whole period of operation.

TABLE 1. Mean Characteristics of the Utilized Vinasse for the Whole Period of Operation*

pH	COD	VFA	VSS	N	NH_4^+	P	SO_4	K
3.9	31.5	2.4	3.7	0.37	0.04	0.024	0.42	1.3

* Units in g/ℓ, except for pH

Sampling and Analysis

Samples of vinasse and digester effluent were collected hourly, 24 hours per day, flow proportionally composed and preserved for daily analyses. Alkalinity, volatile fatty acids and pH were determined immediately after sample collection and other analyses (COD, TSS, VSS, Total N, NH_4^+-N, Total P, SO_4^{2-}, S^{2-}, K) were made from the composite samples.

All analyses were made according to "Standard Methods" (1980) except for volatile fatty acids, determined according to a simplified potentiometric method (Vieira and Souza, 1981).

Determinations on sludge characteristics were made also immediately after sample collection. Six sampling points in the digester allowed the frequent determination of the sludge profile.

Specific methanogenic activity was determined with 0.65 ℓ flasks, at 56°C, using 10 g VSS/ℓ of sludge and a mixture of acetate, propionate and butyrate (respectively 10;1 and 1 g/ℓ) as substrates, according to Penna (1991), at Escola de Engenharia de São Carlos.

Seed Materials

A mixture of mesophilic digested sewage sludge and cow manure was used as seed sludge, since there was no availability of either granular or thermophilic sludge. A total amount of 1200 kg VSS of sewage sludge and 100 kg VSS of cow manure was used.

Nutrients

Urea and phosphoric acid were used in order to add to the vinasse 7.4 g N/kg COD applied and 1.7 g P/kg COD applied.

Soda

The mean requirement of soda for pH control was only 4 g NaOH/kg COD applied.

Effluent Recycle

Digester effluent was recycled to the system in order to keep a maximum HRT of 2 days and a minimum recycle ratio of 50%.

RESULTS AND DISCUSSION

Thermophilic Culture Adaptation

Figure 3 presents the evolution of the organic loading rate, gas yield factor and COD removal ratio.

In the beginning of the experiment, even applying a very low loading rate (0.2 to 1 kg COD/m^3. day), it took 50 days for the reactor to reach a regular operational condition in terms of gas production, i.e., to promote the adaptation of the thermophilic culture present in the seed sludge to the new conditions. The difficulty was caused by the poor seed sludge used.

Loading Rate Increase, Before Granulation

After the initial adaptation of the thermophilic culture in the seed sludge, 90 more

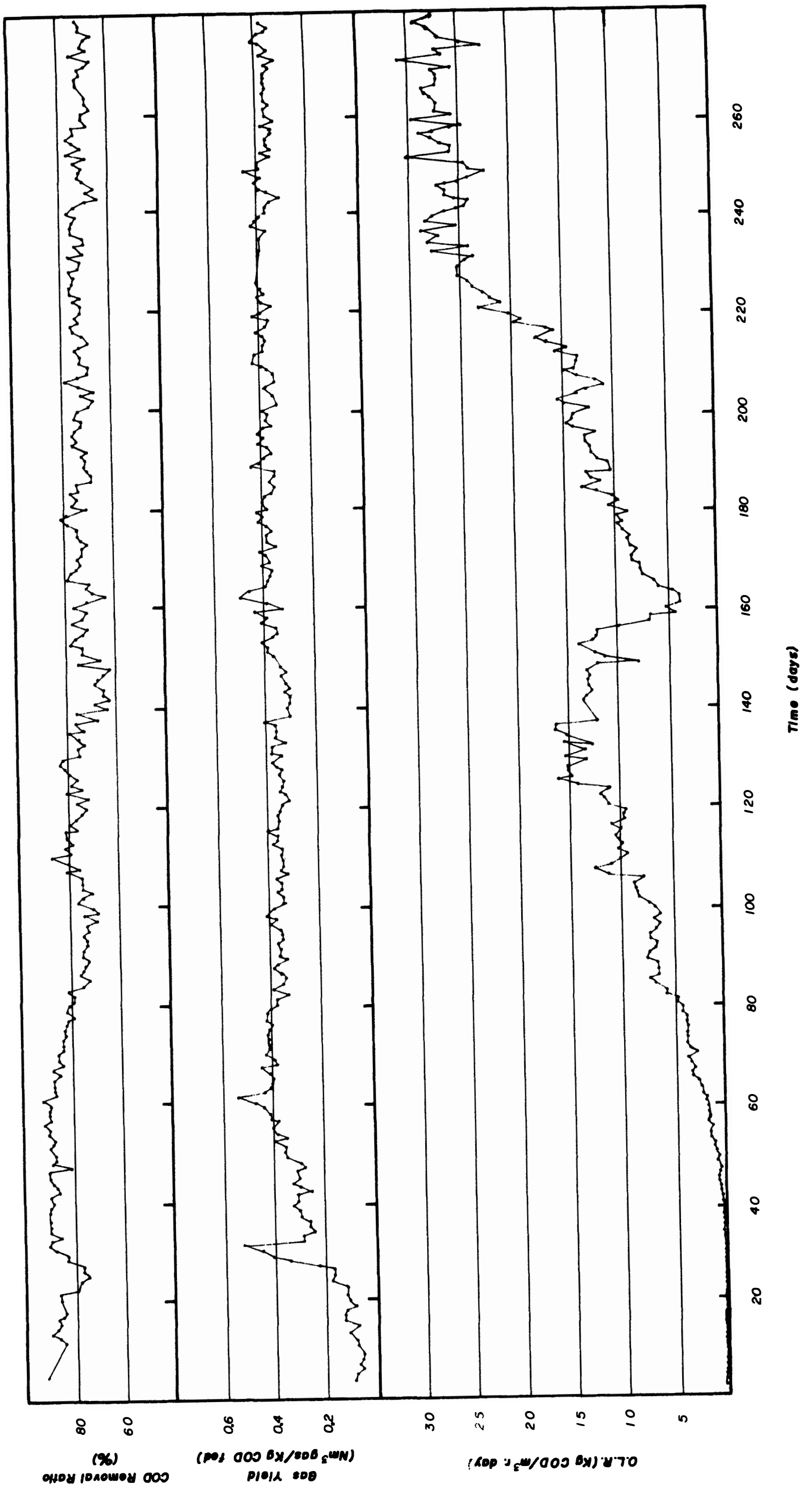

Fig. 3. Evolution of the organic loading rate, gas yield factor and COD removal ratio

days were necessary (from day 50 to day 140) to reach a good organic loading rate (about 15 kg COD/m^3.day), which is in agreement with results obtained in well operating mesophilic UASB reactors treating similar vinasse (Craveiro et al., 1986; Hirata et al., 1988). However, the granulation of the sludge at that time was still very poor, and the sludge quantity reached its minimum value (about 10 kg VSS/m^3). Consequently, the process began to deteriorate, causing an increase in the volatile acids concentration and an effluent pH decrease (Figure 4). Also the COD removal ratio and the gas yield factor decreased (Figure 3).

Therefore, the organic loading rate had to be decreased to less than 5 kg COD/m^3.day, during the next 20 days (between days 140 and 160) of operation, when the process stabilized again.

Sludge Granulation

After re-establishing the process equilibrium, the organic loading rate was again carefully increased, to reach the same values obtained before, i.e, about 15 kg COD/m^3.day, from day 160 until day 210. The behaviour of the process was very good and stable at this time, as can be seen in Figures 3 and 4. The main reason for that was the almost complete granulation of the sludge clearly observed in this period.

Loading Rate Increase, After Granulation

Once the sludge granulated and the concentration of granules increased to about 22 kg VSS/m^3, it was relatively easy to double the organic loading rate (from 15 to about 30 kg COD/m^3.day), in approximately 20 days (from day 210 until day 230), without any increase in volatile acids concentration and without any decrease in effluent pH (using almost no soda), COD removal ratio or gas yield.

Stability of the Thermophilic Process

The main objective of this phase of the research was to reach, in a large pilot-plant UASB reactor operating like a full-scale plant, twice the organic loading rate generally applied in full-scale mesophilic UASB reactors treating vinasse, i.e, 15 kg COD/m^3.day (Craveiro et al., 1986; Hirata et al., 1988).

Furthermore, one of the main problems known about the thermophilic process is its instability. In view of that we kept operating the reactor as long as possible, in an organic loading rate range of 25-30 kg COD/m^3.day, without further increases. This could be maintained for 50 days (from day 230 to day 280), until all the stored vinasse was finished.

During this significant period of operation, the effluent pH, volatile acids concentration, COD removal ratio and gas yield kept almost constant (Figures 3, 4), showing the good stability of the process.

As can be concluded from the comparison between the results obtained before and after granulation, this phenomenon was essential to allow process stability.

It should be noticed that this stability was kept even when operating with variations in flow, pH, COD, SO_4 and other general characteristics of the vinasse.

Otherwise, the mean soda consumption for pH control after granulation was very small (about 4 g NaOH/kg COD fed), independent of the organic loading applied, and probably could be completely avoided, if desired.

Effluent Characteristics

Table 2 presents the mean effluent characteristics during the last 50 days of operation, when the nominal organic loading rate was kept constant. For comparison, the mean characteristics of the vinasse are indicated for the same period.

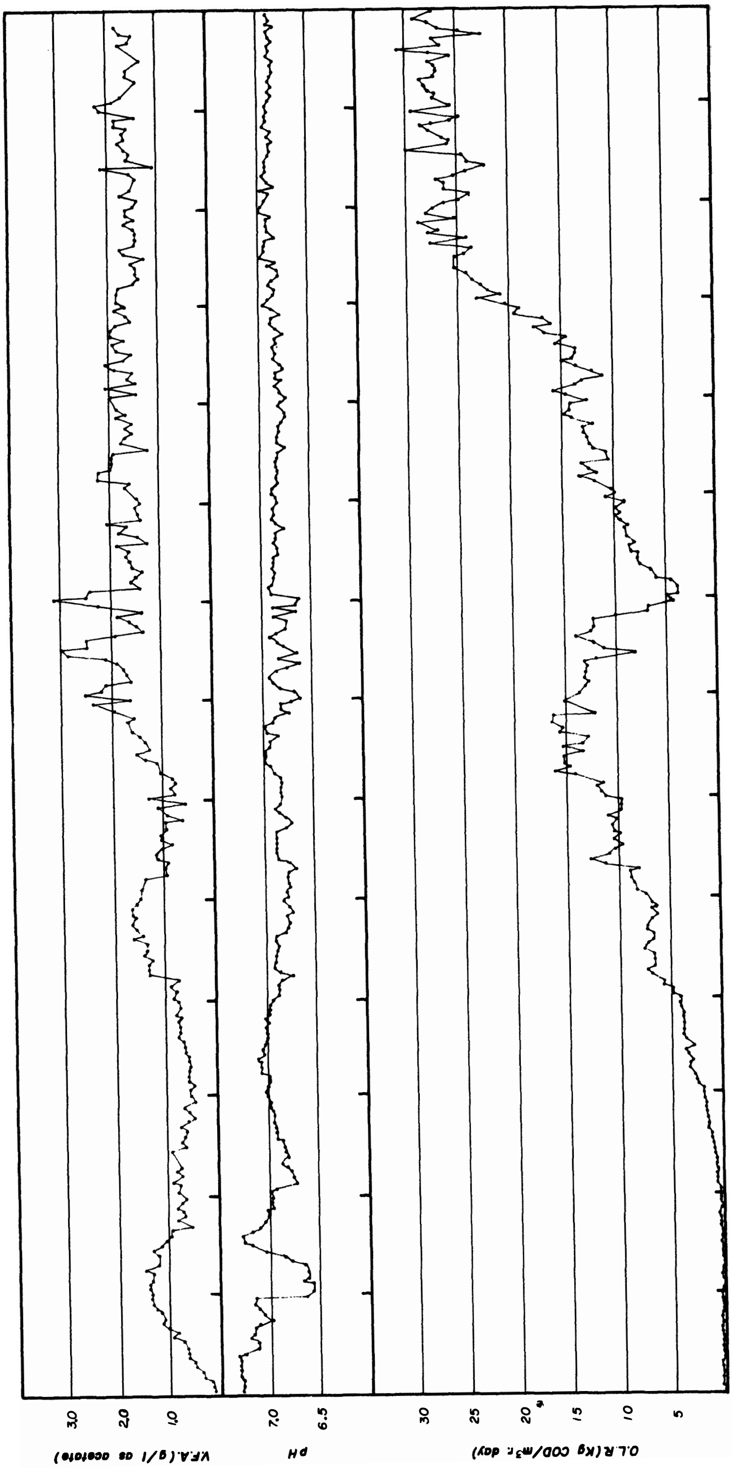

Fig. 4. Evolution of the organic loading rate, effluent pH and volatile fatty acids concentration in the effluent

TABLE 2. Mean Characteristics of Vinasse and Effluent, During the last 50 Days of Operation*

	pH	COD	VFA	VSS	N	NH_4^+	P	SO_4	K
VINASSE	3.7	31.8	2.4	4.6	0.46	0.04	0.027	0.48	1.5
EFFLUENT	6.9	9.0	1.6	4.8	0.66	0.25	0.044	0.01	1.4

* Units in g/ℓ, except for pH

Despite the very low pH of the vinasse, the pH of the effluent reached a very acceptable level, even using almost no soda.

The N, NH_4^+ and P contents of the effluent show that there was excess of these nutrients in the system; probably one could operate the reactor without any addition of N (7.4 g N/kg COD applied was added) and with a lower P addition (1.7 g P/kg COD applied was used).

The high degree of SO_4 removal in thermophilic conditions was surprising, reaching about 98%.

As expected, there was no potassium removal in the process.

In the thermophilic process, the difficulty in reaching a low volatile fatty acid concentration is well known. In fact, the total concentration of VFA in the effluent was not so high (1.6 g/ℓ), considering the high organic loading rates applied in the period (25-30 kg COD/m^3.day), but it was higher than in a mesophilic process.

Furthermore, several chromatographic analyses were made, indicating that the major part of the acids were propionate. The literature also shows the inability of thermophilic bacteria to degrade propionate (Wiegant et al., 1986).

Digester Performance

The digester performance was very good (Table 3), even applying a very high organic loading rate and a low hydraulic retention time.

TABLE 3. Digester Performance*

Loading Rate (kg COD/m^3.day)	26.5
Sludge Loading Rate (kg COD/kg VSS.day)	1.7
HRT (h)	10.8
COD Removal (%)	71.7
Gas Production (Nm^3gas/m^3.day)	9.8
Gas Yield (Nm^3gas/kg COD fed)	0.37
Methane (%)	60

* Mean values for the last 50 days of operation

The sludge loading rate applied during the last 50 days (1.7 kg COD fed/kg VSS.day), was 2 to 3 times greater than the values generally applied for mesophilic granular sludge (Craveiro et al., 1986; Hirata et al., 1988).

Some chromatographic determinations of gas composition were made, at UNESP - Jaboticabal, indicating a CH_4 content of approximately 60%.

The high values of produced gas per reactor volume should be noticed and also the reasonable gas yield and COD removal ratio obtained during the last 50 days of operation.

Specific Methanogenic Activity; Loading Potential

The specific methanogenic activity measured at the end of the experiment, using mixtures of acetate, propionate and butyrate, was exceedingly high (2.14 kg CH_4-COD/kg VSS.day). This value is 3 times greater than the values reported for mesophilic granular sludge (Penna, 1991).

Considering the sludge loading rate applied (1.7 kg COD/kg VSS.day) and the relatively low concentration of sludge present in reactor at the end of the experiment (20 kg VSS/m^3), it is valid to suppose that the maximum possible organic loading rate should be much greater than the values already achieved.

Another measurement of activity was made employing only propionate as substrate; as expected, the obtained values were very low (0.05 to 0.08 kg CH_4-COD/kg VSS.day), confirming the small capacity of thermophilic sludge propionate degradation (Wiegant et al., 1986).

CONCLUSIONS

The feasibility of sugar-cane vinasse thermophilic anaerobic digestion was demonstrated in large-scale pilot-plant UASB reactor.

The high organic loading rates achieved (25-30 kg COD/m^3.day) were about double those normally used in mesophilic UASB reactors treating similar vinasse. Evidence that much greater organic loading rates could be applied in the pilot plant was presented.

In these conditions, high organic matter conversion (72% COD removal) and biogas production rate (about 10 Nm^3gas/m^3.day) were verified and virtually no soda was necessary for pH control.

Granulation of the sludge was very difficult and time-consuming. After granulation of the sludge, no problems regarding the stability of the thermophilic process were experienced, during ten weeks of loading rate increase and seven weeks of operation at the maximum applied loading rates.

Since sugar-cane alcohol distilleries operate only about six months per year, in order to allow full-scale application of the UASB thermophilic process, successful re-start of the plant is essential; this will be the next study in this research.

Very little is known about thermophilic anaerobic bacteria (Zinder, 1988). Studies on this matter will also be conducted during the next step of the research, in collaboration with "Escola de Engenharia de São Carlos - USP".

ACKNOWLEDGEMENT

We would like to thank the Companies and Universities below, which in some manner collaborated for the realization of this work: Cetesb, Sabesp, Unesp - Jaboticabal, USP - Escola de Engenharia de São Carlos.

REFERENCES

Craveiro A.M., Schmidell, W., Villen, R.A. and Hirata, Y.S. (1982). Anaerobic treatment of stillage at low retention time-energy and treatment efficiencies. Proceedings International Fuel Technology Symposium, 5. New Zealand, 3, 149-154.
Craveiro, A.M., Soares, H.M. and Schimidell,W. (1986). Technical aspects and cost estimations for anaerobic systems treating vinasse and brewery/soft drink wastewaters. Water Science and Technology, 18 (12), 123-134.
Hirata, Y.S., Viana, C.E. and Schimidell, W. (1988). Anaerobic treatment of sugar cane stillage in semi-industrial UASB reactor. In: Proceeding of the VIII International Symposium on Alcohol Fuels, Nov. 13-16, 1988, Tokyo, Japan. pp 149-154.
Penna, J.A. (1991, In print). Estudo de Metodologia para Medida de Atividade Metanogênica Específica em Diferentes Lodos Anaeróbios. PhD Thesis, Escola de Engenharia de São Carlos, São Paulo, Brazil.
Souza, M.E. (1986). Criteria for the utilization, design and operation of UASB reactors. Water Science and Technology, 18 (12), 55-69.
Standard Methods for the Examination of Water and Wastewater. (1980). 15th Ed., APHA, AWWA, WPCF, Washington, D.C., USA.
Vieira, S.M.M. and Souza, M.E. (1981). Métodos Analíticos para Acompanhamento da Biodigestão. Energia, 3 (15), 26-36.
Wiegant, W.M., Claassen, J.A. and Lettinga, G. (1985). Thermophilic anaerobic digestion of high strength wastewaters. Biotechnology and Bioengineering, 27, 1374-1381.
Wiegant, W.M., Hennink, M. and Lettinga, G. (1986). Separation of the propionate degradation to improve the efficiency of thermophilic anaerobic treatment of acidified wastewaters. Water Research, 20, 517-524.
Wiegant, W.M. and Lettinga, G. (1985). Thermophilic anaerobic digestion of sugars in upflow anaerobic sludge blanket reactors. Biotechnology and Bioengineering, 27, 1603-1607.
Zinder, S.H. (1988). Conversion of acetic acid to methane by thermophiles. Anaerobic Digestion 1988 (Adv. Water Pollution Control no 5) ed. F. R. Hall and P. N. Hobson.