

## Anaerobic Digestion of Vinasse cane alcohol: The influence of OLR by a UASB reactor

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**Abstract:** An Anaerobic Sludge Blanket (UASB) reactor was used to study the treatment of distillery effluent. Vinasse was used to feed the reactor, although its Chemical Oxygen Demand (COD) concentration varied during the experiment, the volume utilized to feed the reactor was adjusted to maintain constant Organic Load Rate (OLR). The UASB reactor was operated with OLR 1, 2, 4 and 6 gCOD/L-d. Removal efficiencies of 76, 64, 63 and 51% respectively were observed. The reactor responded with progressive decreases of efficiency with each increase of OLR, the total mass removed increased. An average biogas production of 1.400, 1.872, 2.17 and 2.172 L to each OLR of 1, 2, 4 and 6 gCOD/L-d, respectively was observed. The methane content in biogas was 63, 68, 86 and 89% each OLR tested. Methane production is also followed with values of .892 L to OLR 1 gCOD/L-d, 1.264 L to OLR 2 gCOD/L-d, 1.876 L to OLR 4 gCOD/L-d and 2.900 L to OLR 6 gCOD/L-d. The UASB reactor operating in continuous mode, it was necessary to evaluate the best conditions for this type of waste. The treatment of distillery effluents using a UASB reactor is feasible and is an alternative to treat these wastes in the alcohol industries

**Keywords:** Anaerobic digestion, organic load rate (OLR), UASB reactor, Vinasse, Biogas

### I. INTRODUCTION

The sugar and distillery industries are the most important agro-industries for economic development in Mexico and several countries for the production of sugar and alcohol. However, alcohol industry has been proved as one of the industries, which consume large amount of water and energy, produce numerous organic pollutants, and cause serious contamination [1]. All distilleries produce an effluent commonly known as “vinasse,” an amount equal to 10–15 times that of the volume of alcohol [2]

According to the origin of the raw material and the fermentation / distillation used for obtaining alcohol, is the high content of COD, total nitrogen, total phosphorus effluent among other parameters. The vinasses disposal into the environment is hazardous and has high pollution potential. The highly colored components of the vinasses reduce sunlight penetration in rivers, lakes or lagoons, which in turn decrease both photosynthetic activity and dissolved oxygen concentration affecting aquatic life. In accordance Pant and Adholeya [3] the brown colour is due to phenolics (tannic and humic acids) from the feedstock, melanoidins from Maillard reaction of sugars (carbohydrates) with proteins (amino groups)

[2] Patel et al. reported that the dry vinasse or effluent contains about 38–40% inorganic salts of potassium, sodium, magnesium, and calcium in the form of chlorides, sulfates, and phosphates, and about 60–62% organic compounds. Besides a strong pungent smell and intense dark color, effluent has a large biological oxygen demand (BOD) and chemical oxygen demand (COD) in the range of 45 and 100 g/L, respectively.

[4] Mohana et al. indicate that the unpleasant odor of the vinasse is due to the presence of skatole, indole and other sulphur compounds, which are not effectively decomposed by yeast during distillation.

A number of technologies have been explored for reducing the pollution load of distillery effluent. Biological treatment of distillery is either aerobic or anaerobic but in most cases a combination of both is used. Various physicochemical methods such as adsorption, coagulation–flocculation, and oxidation processes have also been practiced for the treatment of distillery effluent [3].

Among anaerobic technologies available, based clearly in the granular sludge blanket is the most interesting. The UASB concept is one of the most notable developments in anaerobic treatment process technology, conceived in the late 1970's in the Netherlands by Professor Lettinga Gatzte Wageningen.

The key features of the UASB process that allow the use of high volumetric COD loadings compared to other anaerobic processes. In this study the influence Organic Load Rate (OLR) with the removal efficiency was evaluated.

## II. MATERIALS AND METHODS

### 2.1 UASB reactor

The UASB reactor (Figure 1) consisted of a glass column 53 cm high, with 7.5 cm internal diameter and six sampling points along its length, the reactor had a total volume of 2.3 L. The reactor was fed with vinasse using a peristaltic pump and the vinasse was maintained at 20°C in a container during the feeding to the reactor. The hydrodynamic conditions and upflow was maintained by recycling using a Masterflex® peristaltic pump. An inverted conic gas-solid-liquid separator was installed in the upper part of the reactor, after the biogas passed through a Mariotte Flask containing to (3N NaOH) solution. The operation temperature was of 35±2°C using a Polystat® water bath heater circulator

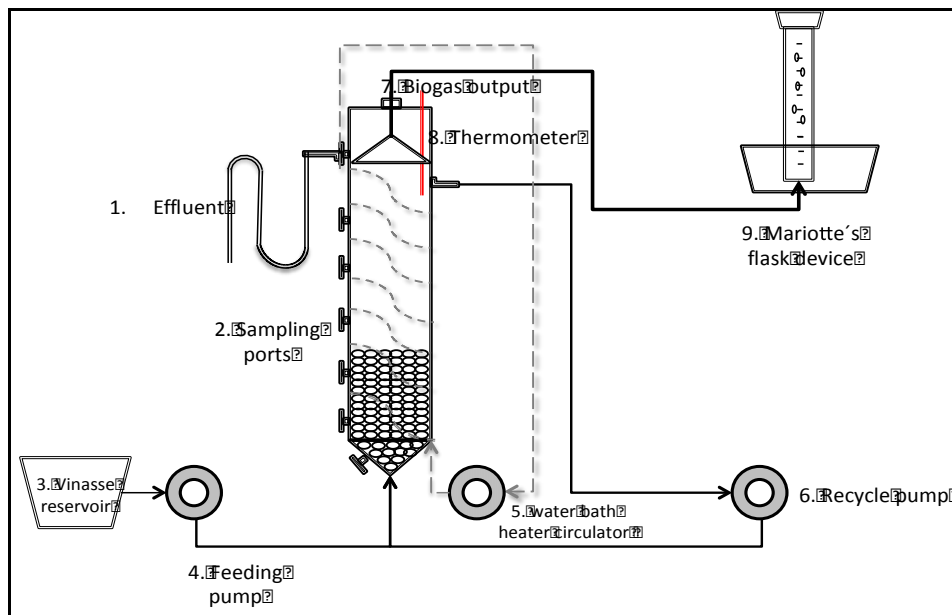


Figure 1. UASB reactor

### 2.2 Experimental procedure

To inoculate the UASB reactor, there were used granular sludge from a Wastewater Treatment Plant (WWTP), which is a mixture of urban wastewater (UW) and Industrial Wastewater (IW), from various local industries such as; brewery, paper, production of drugs, chemicals, steel mills, petrochemical and others. In the beginning of the experiment, an operation was performed in batches. 600 ml of concentrated sludge were used, which represented an initial concentration of 10.63 g/L of VSS in the mixed liquor in the reactor. A solution of inorganic medium adds (Table 1)

Table 1. Modified inorganic medium

Modified Kawahara		Stock solution volume/100 ml of reactor
KH <sub>2</sub> PO <sub>4</sub>	4.05 g/l	15 ml Stock solution of KH <sub>2</sub> PO <sub>4</sub>
K <sub>2</sub> HPO <sub>4</sub>	8.385 g/l	4 ml Stock solution of K <sub>2</sub> HPO <sub>4</sub>
NH <sub>4</sub> Cl	7.95 g/l	4 ml Stock solution of NH <sub>4</sub> Cl
CaCl <sub>2</sub>	1.125 g/l	4 ml Stock solution of CaCl <sub>2</sub>
MgCl <sub>2</sub> .6H <sub>2</sub> O	1.0 g/l	4 ml Stock solution of MgCl <sub>2</sub> .6H <sub>2</sub> O
FeSO <sub>4</sub> .7H <sub>2</sub> O	5.6 g/l	4 ml Stock solution of FeSO <sub>4</sub> .7H <sub>2</sub> O

Note: Original Inorganic medium [5]

### 2.3 Substratum

The vinasse used throughout the entire study, came from a family local distillery that processes about 20,000 liters of daily alcohol from molasses. The production of vinasse is about 20 L per liter of processed alcohol, i.e. 200 m<sup>3</sup> per day. This vinasse are downloaded and processed in an anaerobic lagoon system. The vinasse was sampled monthly during the project; in the Table 2, the average characterization of the vinasse used for the project is summarized. It was operated in continuous mode, feeding vinasses by a peristaltic pump. The vinasse was neutralized before feeding the reactor, taking a pH of 4-7 with manual addition of 3N NaOH. Initial OLR 1 gCOD/L·d was applied, with an average concentration of vinasse of 108.33 and 97.47 gCOD/L total and soluble, respectively. OLR gradually increasing values were performed at 2, 4, up to a maximum value of 6 gCOD/L·d observing the stabilization of the system in each condition. In Table 3, the UASB reactor operations conditions are shown. Gradual increases were performed according to the recommended values by Lettinga and Hulshoff [6]

Table 2. Characterization of the vinasse

Parameter	Average	Maximum	Minimum
pH	4.14	4.44	4.03
Conductivity (µs/cm)	21.17	29.80	7.73
Tot-COD (g/L)	128.63	217.71	57.59
Sol-COD(g/L)	108.48	156.07	36.13
TTS (g/L)	80.12	113.98	17.85
VTS (g/L)	58.11	81.67	11.81
TSS (g/L)	6.83	15.24	1.08
VSS (g/L)	5.42	11.78	0.96
N-Organic(g/L)	0.25	0.65	0.08
TKN (g/L)	0.28	0.69	0.12
N-NH4 (g/L)	0.03	0.05	0.003
Total phosphate (g/L)	0.08	0.15	0.01
Sulfates (g/L)	9.36	14.64	5.03

Table 3. Average operating conditions of the UASB reactor with proved OLR

	OLR (gCOD/L·d)			
	1	2	4	6
Upflow velocity (m/h)	2	2-3	2,5-3	2,5-3
HRT (days)	109	58	30	25
Flowrate (L/d)	0,022	0,042	0,108	0,099
Temperature (°C)	36±2	36±2	36±2	36±2

### 2.4 Performed analyzes

To verify the stability of the system, the following parameters were analyzed daily, influent and effluent: COD<sub>Total</sub>, COD<sub>Soluble</sub>, sulfate (SO<sub>4</sub><sup>2-</sup>), total solids (TS), volatile total solids (VTS), total suspended solids (TSS) and volatile suspended solids (VSS), pH, temperature, biogas and CH<sub>4</sub> produced. For the analytical determination of soluble compounds previously samples were centrifuged (6000 rpm for 30 min.). The procedures correspond to those reported by Mexican standards, and standardized methods for the analysis of drinking and Wastewater [7]

### III. RESULTS AND DISCUSSION

#### 3.1 Process Performance

The percentage of COD<sub>soluble</sub> removal function of time can be seen in Figure 1. At the beginning of the experiment, was observed an upper 98% removal during the first 20 days. It is up to day 50 and up to a length approximately equivalent to 1 HRT that is beginning to show a stable clearance values over 70%. Initially the OLR of 2 gCOD/L-d values for removal of approximate 80% this is still observed for 72 days after the 1 OLR gCOD/L-d, ie equivalent to the period of 1 HRT. The observed average value of COD removal percentage is approximately 60%. By applying OLR of 4 gCOD/L-d due to a change in the concentration of our vinasse during a period of approximately 50 days, we obtain data clearance 40 to 60%. Once stabilized feeding conditions, a removal of COD<sub>soluble</sub> average of 60% is reached.

During OLR of 6 gCOD/L-d is observed a greater variability in the percentage of COD removal. Excluding the period in batch mode to recover the activity of the reactor, the average removal efficiency of soluble COD was 51%. We can observe that while the reactor responds by a progressive reduction to each increase efficiency OLR the total mass removed increases.

The response of the reactor and the biodegradability of the effluent is considered that further increases OLR could irreversibly affect the operation of the reactor. However it is clear that the concentration of vinasse and consequently, HRT plays an important role in the operation and efficiency of the UASB reactor. This directly impacts the Hydraulic Retention Time (HRT), which has values of 118, 59, 41, 24 days for the OLR 1, 2, 4 and 6 gCOD/L-d respectively.

These facts still position the UASB technology in a suitable and appropriate process for the treatment of sugar cane alcohol vinasses.

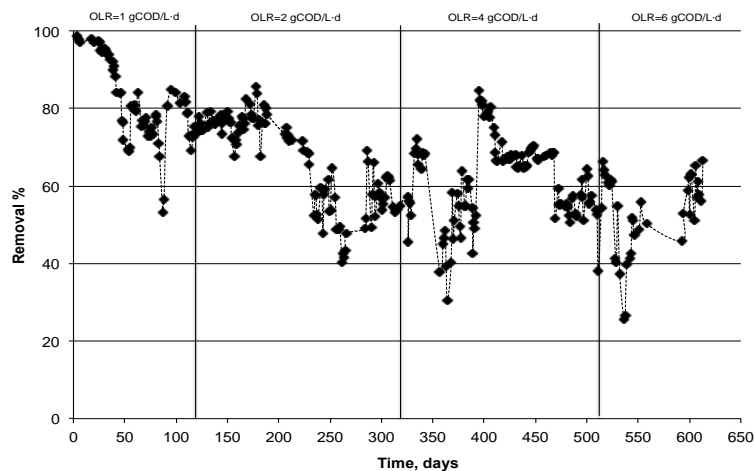


Figure 2. COD<sub>soluble</sub> removal versus time

#### 3.2 Biogas production

The biogas production, CH<sub>4</sub> and CH<sub>4</sub> percentage is illustrated in figure 3. To an OLR 1 gCOD/L-d average and stable biogas production and methane with values of 1.4 L and 0.892 L respectively was observed. The presented biogas contained 63% of methane. Biogas production and methane observed reflect a good development of the granule and a good metabolism by this. Furthermore, the average theoretical production of CH<sub>4</sub> is 0,646 L, i.e. lower than actually occurred, this may have two explanations. First, the sensitivity of the biogas production volumetric system. However, the measured values would be lower than the theoretical, in this case. For this reason, a second alternative is required. According E. Houbron, et al [8] a solids residence time of more than 20 days, promotes liquefaction of solid organic wastes. Indeed given the amount of solids in the vinasse and HRT 118 days, the conditions for hydrolysis-solubilization of this organic matter are presented and allow additional methane production. With respect to the OLR 2 gCOD/L-d, the average production of biogas and methane had values of 1.87 L and 1.26 L respectively. The methane gas is 68%, which represents an interesting quality of biogas from the energy standpoint. The theoretical value calculated methane is 1 L. This value is still lower than what actually occurred. Under these operating conditions the HRT of 59 days is still suitable to hydrolysis-solubilization of solid waste and the generation of an extra amount of methane. By increasing OLR 4 gCOD/L-d proportional increases were observed in the production of biogas and methane.

The average production of biogas and methane was 2.17 L and 1.87 L, respectively. The methane content of the biogas is very interesting; values increased to 86%. The average theoretical methane production represents the 2.15 L, higher than the measured value. Under this OLR of 4 gCOD/L·d, HRT is approximately 40 days whereby the condition for hydrolysis of the solids is reduced. The difference between real and calculated methane production corresponds to the balance of the catabolism and anabolism of the microorganisms in the reactor, under these operating conditions. With OLR 6 gCOD/L·d an average biogas and methane production of 3.26 L and 2.9 L, respectively was observed. Actual production of methane is close to the theoretical value calculated from 2,767 L. The average percentage of CH<sub>4</sub> content in the biogas was 89%. Biogas constant values when applying OLR 4 and 6, even with the variability of HRT were maintained.

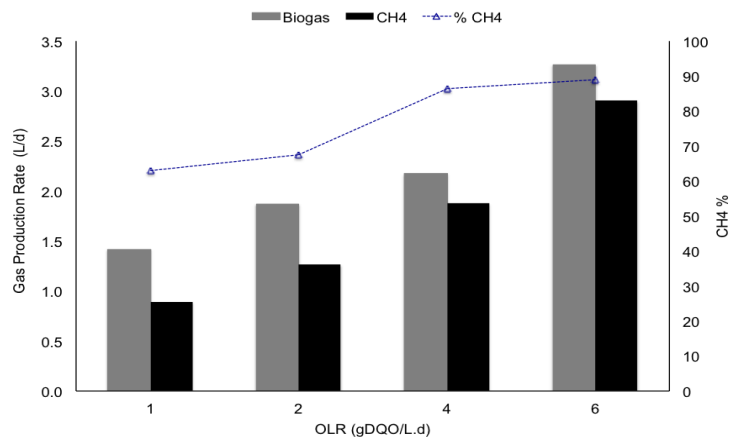


Figure 3. The biogas production, CH<sub>4</sub> and CH<sub>4</sub> percentage versus Organic load rate (OLR)

### 3.3 Methane yield

To make a more accurate methanogenic activity of granules in the reactor evaluation, the methane yield was used as a monitoring parameter [9] [10]. The methane yield is the amount of methane produced per mass of organic matter, removed at a given temperature. The average temperature of the reactor throughout the whole study was 36°C, corresponding to a theoretical 0.396 production efficiency  $LCH_4/gCOD_{removed}$ .

In Figure 4, the methane yield evolution versus OLR is showed. For OLR 1 g COD/L·d a  $Y_{CH_4}$  average of 0.504  $LCH_4/gCOD_{removed}$  was obtained. The methane yield real value exceeds the theoretical value, involved the long HRT, which allows it to carry out a hydrolysis-solubilization of the solids, which generates an extra supply in soluble COD. For OLR 2, 4, and 6 gCOD/L·d, methane yield values are very close to the theoretical value. As we discussed previously, the presence of solids and long HRT generate favorable hydrolysis-solubilization conditions, which may explain an additional methane production. These optimal values of methane yield confirm the excellent activity of the granules to treat vinasse. However, an optimization of the HRT or dilution vinasses, may eventually reduce the toxicity of the substrate, increase its removal at higher OLR and also eliminate hydrolysis-solubilization.

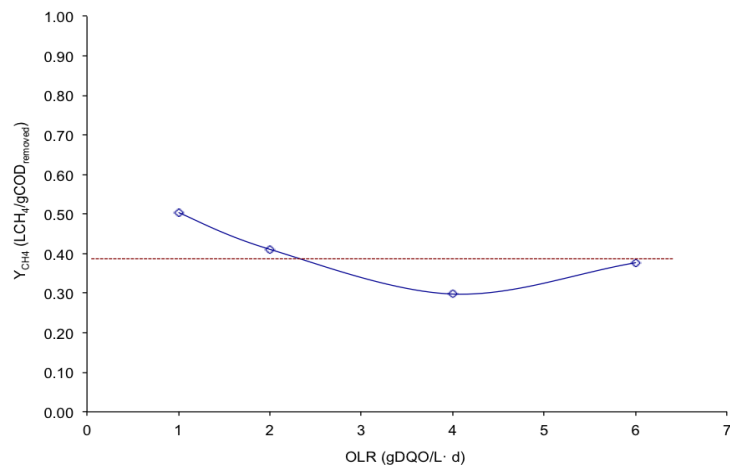


Figure 4. The Methane yield ( $Y_{CH_4}$ ) evolution versus Organic load rate (OLR)

Several authors reported the operation of UASB reactors at higher loads of 10 gCOD/L-d [11] [12] [13]. However all reported strong reduction removal efficiency by increasing the load. Moreover, it is reported [14], that dilute effluent removal efficiencies increase. Now, while diluting a vinasse at laboratory does not represent major problems, in situ requires a huge source of water. Given the laws in practice is unthinkable, use clean water to dilute vinasse, so the only option would be to reuse the treated effluent. Another alternative could be mixing the waters of vinasse with urban wastewater. But, as Toze [13] mentioned, there are a number of risk factors to reuse. Certain factors are short term and vary in their degree of risk, depending human, animal and environmental factors (pathogens, microbes), while others have a long term impact, which increase with continued use of wastewater recycling (eg, saline effects on soil). Therefore, dilution of vinasse as charged is not as viable in situ solution.

#### IV. CONCLUSION

By operating the UASB reactor in continuous mode, it was possible to evaluate the best operating conditions for this type of waste (vinasse). For a range of operation OLR 1-6 gCOD/L-d quite a favorable response is observed with respect to the performance of UASB reactor, as presented soluble COD removal percentages of 51 to 76%, similar efficiencies to those reported literature. The OLR 6 gCOD/L-d for the UASB reactor fed with vinasse represents the limit of its capacity. However, with increasing OLR increased biogas production and methane is generated. Elevated HRT applied, allowed hydrolysis-solubilization of solid, generating an extra production of methane, which is reflected in the higher theoretical value  $Y_{CH_4}$ . Once the reactor stabilized, catabolism was the dominant biochemical process. Treating cane alcohol vinasse using an UASB reactor is feasible, however the high concentration of organic matter imposes high HRT.

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