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Influence of Hydraulic Retention Time in the Treatment of Cane Alcohol Vinasse by UASB Reactor

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ABSTRACT

The influence of the hydraulic retention time (HRT) on the removal efficiency of the organic matter in a UASB reactor was evaluated. Several tests were carried out in a continuous flow, maintaining the organic load rate (OLR) at a constant value of 6 gCOD/L.d and applying several HRTs of 24, 12, 5, 3 and 1 day. The average results show that the removal of organic matter from the applied HRT is in a range of 51 to 63% in COD_{soluble}. The average biogas and methane production for the HRTs of 24, 12, 5, 3 and 1 day were 3.265, 5.283, 4.196, 3.350 and 2.450 L/d and 2.905, 3.815, 3.370, 3.275 and 2.200 L/d, respectively. The yield of methane (Y_{CH4}) presented average values of 0.306 to 0.459 L/g COD_{removed} in the HRTs applied. The reduction of the HRT or dilution of the vinasse influenced the reduction of sulphate toxicity and increased the efficiency of its removal. These changes impede the activity of hydrolytic bacteria, reducing the total volume of biogas and indirectly the risk of an overload from an uncontrolled additional input of organic matter. Under these conditions, the OLR could still be increased, since demonstrated the ability to process more organic matter.

INTRODUCTION

Mohana et al. (2009) showed that dumping vinasse into the environment establishes a hazard and has high pollution potential. The highly coloured components of the vinasse reduce the sunlight penetration in rivers, lakes or lagoons, which in turn decrease both photosynthetic activity and dissolved oxygen concentration affecting the aquatic life. In accordance to Pant & Adholeya (2007), 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). Patel et al. (1996) reported that the dry vinasse or effluent contains about 38-40% inorganic salts of potassium, sodium, magnesium, and calcium in the form of chlorides, sulphates, and phosphates, and about 60-62% organic compounds. Besides a strong pungent smell and intense dark colour, effluent has a large biochemical oxygen demand (BOD) and chemical oxygen demand (COD) in the range of 45 and 100 g/L, respectively. Sharma et al. (2007) indicated that the unpleasant odour of the vinasse is due to the presence of skatole, indole and other sulphur compounds, which are not effectively decomposed by yeast during distillation.

Singh et al. (2013), mentioned about the different anaerobic treatment systems and technologies that are being encouraged because of several advantages, including low construction costs, small land requirements, low sludge production, easy operation and maintenance, and energy generation in the form of biogas.

Vieira & Garcia (1992) concluded in their study, that hydraulic retention time (HRT) is one of the most important parameters affecting the performance of a UASB reactor when used for the treatment of municipal wastewater. In this study the influence of hydraulic retention time (HRT) on the removal efficiency of organic matter from cane alcohol vinasse has been evaluated.

MATERIALS AND METHODS

The UASB reactor (Fig. 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 2.3L. 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 3N NaOH solution. The operation temperature was of $35\pm2^{\circ}$ C using a Polystat® water bath heater circulator.

To inoculate the UASB reactor, granular sludge from a

wastewater treatment plant (WWTP) was used, 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. At the beginning of the experiment, an operation was performed in batches. 600 mL of concentrated sludge was used, which represented an initial concentration of 10.63 g/L of SSV in the mixed liquor in the reactor. The feed utilised was vinasse, coming from a family local distillery that processes about 20,000 litres of daily alcohol from molasses. The production of vinasse is about 20 L per litre of processed alcohol, i.e. 200 m³ per day. The vinasse was transported and maintained at 4°C before its utilisation. Vinasse was diluted with tap water, to obtain the required concentration and reach the HRT established. It was operated in continuous mode, feeding vinasse by a peristaltic pump. The diluted vinasse was neutralized before feeding the reactor, taking a pH of 4-7 with manual addition of 3N NaOH stabilization of the system in each condition.

According to the experiments by Sosa et al. (2014), the importance of HRT and dilution of vinasse in the operation of a UASB reactor is shown. Therefore, in this research different hydraulic retention times (HRT) were applied to determine the influence of HRT on the UASB reactor. The HRT applied were 24, 12, 5, 3 and 1 days. HRT was established by changing the concentration of vinasse. The calculation was performed with the following data: HRT set of 24, 12, 5, 3 and 1 day, the flow rate reactor (Q) (Table 1), the set OLR of 6 gCOD/L.d and the reactor volume (V_R) of 2.4 L. When the result of the concentration of vinasse was obtained, we proceeded to prepare the reactor feed. Gross vinasse diluted with tap water, to obtain the required concentrations and reach the HRT established. Table 1 shows the operating conditions of the UASB reactor with tested HRT.

The stabilization of the system was verified with a daily analysis of the influent and effluent and the following parameters: COD_{T} , COD_{S} , sulphates (SO_{4}^{2-}), total solids (TS), total volatile solids (TVS), total suspended solids (TSS) and volatile suspended solids (VSS), pH, temperature, biogas and CH₄ produced. The followed procedures correspond to those indicated by Mexican standards, and standard methods for the analysis of water and wastewaters (APHA 1995).

RESULTS AND DISCUSSION

The UASB reactor was operated under five different HRTs 24, 12, 5, 3 and 1 day and applying a OLR of 6 gCOD/L·d.

Fig. 2 shows the removal efficiency in COD as a function of HRT. The 24-day HRT had a mean removal value of 51% soluble COD. At the start of the 12-day HRT, a 56% removal was observed, since the system stabilized at 10

Table 1: Operating conditions of UASB reactor with applied HRTs.

Parameter	HRT (days)				
	24	12	5	3	1
OLR (gCOD/L·d)	6	6	6	6	6
Upflow velocity (m/h)	3	3	3	3	3
Flow rate Q (L/d)	0.099	0.207	0.535	0.877	2.108
Temperature (°C)	36 ± 2	36 ± 2	36 ± 2	36 ± 2	36 ± 2

days. After 10 days, mean percentages of 60% removal of soluble COD were presented.

At the start of 5-day HRT, removal values of approximately 60% were still observed. The average value observed was 57% of COD removal. Sunil Kumar et al. (2007) operated an anaerobic hybrid reactor (combination of sludge bed and filter) at laboratory scale in continuous mode to study the anaerobic biodegradation of distillery wastewater. The study showed that an optimal HRT of 5 days and OLR of 8.7 kgCOD/m³·d, was able to obtain a removal efficiency of 79%.

The 3-day HRT was a condition in which the bacteria responded quickly, i.e. there was a stabilization in the reactor. During the first 3 days, values of 60% were obtained, decreasing to 45%. Removal was also obtained above 60% and on average a 60% removal was obtained in soluble COD.

The last HRT applied, which was 1 day, initially showed a little instability, from a value of 80% to decrease to a 29% removal in soluble COD. The average COD removal percentage was 63%.

Anaerobic sulphate reduction increased sulphur concentration, which inhibited the metabolism of methanogenic bacteria and reduced the reactor performance.

Biogas and methane production: To better analyse the metabolism of the granules, a daily production of biogas and methane was estimated in the reactor, as well as the percentage methane contained in the biogas (Fig. 3). The theoretical methane volume value calculated stoichiometrically for an OLR of 6 gCOD/L·d was 2.767 L/d.

For 24-day HRT, biogas average values of 3.265 L and CH₄ of 2.905 L were obtained, i.e. a methane content of almost 90%. The volume of methane generated is very close to the calculated theoretical value.

When applying the 12-day HRT, a value of approximately 6 L/d of biogas was reached. After 10 days of operation, more stable biogas values were obtained, such as an average value of 5.283 L/d and a percentage of CH_4 of 72%. At the beginning of this condition the methane production had values close to 2.7 L/d, and then stabilized around 3 L/d. The average yield during the 12-day HRT period was 3.815 L of methane. When the 5-day HRT was applied, there was



Fig. 1: The UASB reactor.



Fig. 2: Percent COD removal versus HRT.

a slight decrease in volumes, with an average production of 4.196 and 3.370 L/d of biogas and methane respectively. The methane content of this biogas was 80%. By reducing HRT to 3 days, the volumes produced were also reduced to 3.550 and 3.275 L/d of biogas and methane respectively. The proportion of methane remains high with 92% CH₄ in biogas. The last HRT applied was 1 day, in this case 2.450 L of biogas and 2.200 L of methane was obtained. The methane content in the biogas was 90%.

By keeping the OLR at 6 gCOD/L·d, the amount of organic matter introduced into the reactor is constant, so the reduction of biogas and methane production is not explained by reducing the HRT. Although the overall removal efficiency and CH_4 content of biogas were not affected by HRT, we can note an alteration in methanogenic activity.

With respect to biogas, a maximum production value of 5.283 L/d was reached, and then, by reducing HRT, a pro-



Fig. 3: Biogas, CH₄ and CH₄ percentage versus HRT.



Fig. 4: Evolution of methane yield $(\boldsymbol{Y}_{\text{CH4}})$ as a function of HRT.

portional reduction of the total volume of biogas was observed. However, the percentage of methane contained in the biogas increased with the decrease of the HRT, reflecting values of 80 to 92% of CH_4 .

It is considered that by reducing the HRT, the favourable conditions necessary to the hydrolytic bacteria disappeared, reflected in a reduction of extra methane production.

The methane yield was not affected by the TRH changes, presenting values close to the theoretical along the experiment. This confirms the excellent methanogenic activity of the granule.



Fig. 5: Evolution of sulphates as a function of HRT.

Methane yield: To make a more precise evaluation of the methanogenic activity of the granules present in the reactor, the methane yield was used as a monitoring parameter (Michaud 2002, 2005). The methane yield is the volume of methane produced per mass of organic matter, removed at a given temperature. The average reactor temperature throughout the study was 36°C, corresponding to a theoretical production efficiency of 0.366 LCH₄/g COD_{removed}.

In Fig. 4, the evolution of the Y_{CH4} is shown, the 24-day HRT reached an average value of 0.377 LCH₄/gCOD_{eliminated}. In 12-day HRT, a peak in methane yield was initially observed to finally reach an average Y_{CH4} value of 0.459 LCH₄/gCOD_{eliminated}.

From 5-day HRT, in spite of highs and lows, an average Y_{CH4} value of 0.365 LCH₄/ gCOD_{eliminated} can be observed. The next condition applied was that of a 3-day HRT, where an average of 0.306 LCH₄/ gCOD_{eliminated} was elicited. Subsequently a 1-day HRT was applied and the mean Y_{CH4} was 0.371 LCH₄/gCOD_{eliminated}.

The Y_{CH4} presents values very close to the calculated theoretical, so the methanogenic bacteria are working correctly with a metabolism focused on the production of biogas.

On the other hand, by reducing HRT, the conditions no longer favour the hydrolytic bacteria, which means that there is no longer any production of methane from this extra source of organic matter. This phenomenon could explain the reduction of gas production by reducing HRT. If so, this OLR of 6 gCOD/L.d, which appeared to be the upper limit of operation of the reactor, could still be increased.

The behaviour of the sulphate concentrations during the HRT conditions at the inlet and outlet of the UASB reactor is shown in Fig. 5. For the 24-day HRT, the concentrations in SO_4^{-2} were very variable, since at the entrance of the reactor values of 25 gSO_4^{-2}/L were observed until decreasing to values of approximately 15 gSO_4^{-2}/L .

At the reactor outlet, there was also a decrease in values from 15 to 8 gSO₄²⁻/L. Average concentration values obtained were 20 and 13 gSO₄²⁻/L at the reactor inlet and outlet respectively, a removal percentage of 34% of sulphates and a COD/SO₄²⁻ ratio of 6.74.

 COD/SO_4^{2-} ratio is an important control parameter in the anaerobic treatment of this type of effluents. When the COD/SO $_4^{2-}$ ratio is equal to 0.67, theoretically there is sufficient SO_4^{2-} in the medium for the sulphate-reducing bacteria (SRB) to completely remove the organic matter (Rinzema & Lettinga 1988, Alphenaar et al. 1993). On the other hand, when the COD/SO $_4^{2-}$ ratio is higher than the stoichiometric value, there is sufficient organic matter that favours the co-existence of both methanogenic microorganisms (MM) and sulphate-reducing bacteria (SRB).

For a 12-day HRT of sulphates average with an input of 7.44 g SO_4^{2-}/L , output values were obtained from 5.9 to 2.84 with an average of 3.52 g SO_4^{2-}/L . Removal percentage of

 SO_4^{2-} achieved an efficiency of 53% and a COD/ SO_4^{2-} ratio of 9.58. When an HRT of 5 days was applied, we obtained an average removal percentage of 47% and a COD/ SO_4^{2-} ratio of 21.38.

For a 3-day HRT, an average COD/SO₄²⁻ ratio of 18 and a 6% removal rate of sulphates were calculated. The last condition applied was an HRT of 1 day. The average COD/ SO₄²⁻ calculated ratio was 7.10 and an average removal percentage of 80% SO₄²⁻. Under the conditions applied, it was observed that the highest sulphate removal was when applying 1 day HRT. COD/SO₄²⁻ ratio was greater than the stoichiometric value of 0.67 in all conditions. Kalyuzhnyi et al. (2001) and Searmsirimongkol et al. (2011) obtained results similar to those observed here with a percentage of sulphate removal of 80% for an HRT of 5 and 0.85 days.

Reduction of HRT or dilution of vinasse was beneficial by reducing the toxicity of sulphates and increasing the efficiency of their removal.

CONCLUSIONS

The removal efficiency of soluble COD is independent of HRT, for a OLR of 6 g COD/L·d and tested HRT conditions (24, 12, 5, 3 and 1 days). The removal values achieved are up to a little higher than the observed anaerobic biodegradability values of vinasse of 50%. With regard to biogas, a maximum production average value of 5.283 mL/d was reached, however, reducing HRT shows a proportional reduction of the total volume of biogas. On the other hand, as the HRT decreases, the percentage of methane contained in the biogas increased, reflecting values of 80 to 92% of CH₄.

It is considered that by reducing the HRT, the favourable conditions necessary to the hydrolytic bacteria disappeared, reflected in the reduction of extra methane production.

The methane yield was not affected by the HRT changes, presenting values close to the theoretical, confirming the excellent methanogenic activity of the granule methanogenic bacteria. The sulphate removal efficiency in these experiments reached maximum values of up to 80%, as the HRT was decreasing.

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