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# Anaerobic co-digestion of sugarcane press mud with vinasse on methane yield

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#### ABSTRACT

The conversion efficiency of high solids waste digestion as sugarcane press mud (P) may be limited due to hydrolysis step. The option of co-digestion with vinasse, main liquid waste generated from ethanol production, was investigated under batch regime at mesophilic conditions  $(37.5 \pm 1 \,^{\circ}\text{C})$  and the best mixture was evaluated under semicontinuous regime in stirred-tank reactors. The maximum values for methane yield in batch tests were for  $V_{75}/P_{25}$  and  $V_{50}/P_{50}$  mixtures (on basis of the chemical oxygen demand (COD) percentage added in the mixture), with an average value of 246 N mL CH<sub>4</sub> g<sup>-1</sup> COD<sub>fed</sub>, which was 13% higher than that of press mud alone. A highest methane production rate of 69.6 N mL CH<sub>4</sub> g<sup>-1</sup> COD<sub>fed</sub><sup>-1</sup> d<sup>-1</sup> was obtained for the mixtureV<sub>75</sub>/P<sub>25</sub>. During the experiment carried out in CSTR reactors, the organic loading rate (OLR) was increased from 0.5 up to 2.2 g VS L<sup>-1</sup> d<sup>-1</sup>. Methane yields of 365 L CH<sub>4</sub> kg<sup>-1</sup> VS and biogas productivities of 1.6 L L<sup>-1</sup> were obtained in co-digestion, which was 64% higher in comparison to mono-digestion. The performance of the process in mono-digestion was less stable than in co-digestion, with a significant fall of methane yield to 1.8 kg VS m<sup>-3</sup> d<sup>-1</sup>. And a partial inhibition of the methanogenic archaeas when the OLR was increased up to 2.2 kg VS m<sup>-3</sup> d<sup>-1</sup>. The co-digestion of vinasse with press mud is a good option for the treatment of streams at the alcohol-sugar industry.

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#### 1. Introduction

Biogas can be produced from a diversity of organic substrates, through the anaerobic digestion (AD) process. In Cuba, most of the biogas plants are digesting pig and cow manure, and a few are working with wastewaters from industries as alcohol distillery and brewery. The main goal of the biogas plants working currently has been to reduce the environmental organic load before its final disposition. However, energy generation from renewable energy sources is being taken into account by the Cuban government. That is why, other residues with higher biogas potential and alternatives to optimize the AD process will have to be implemented to attain economic viability.

Among them is the press mud, a solid fibrous residue generated during sugar cane juice clarification and filtration. Press mud is characterized (on dry weight basis) by 9–14% wax, oil, and resin, 10–18% protein, 11–17% cellulose, 15–27% hemicellulose and 9–14% lignin (Janke et al., 2016; Leite et al., 2015; López González et al., 2014). Also it contains 0.6–3% P<sub>2</sub>O<sub>5</sub>, 2–7% Ca, 0.2–0.3% Fe,

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http://dx.doi.org/10.1016/j.wasman.2017.07.016 0956-053X/© 2017 Elsevier Ltd. All rights reserved. 0.3–0.4% K, 0.2–0.3% Mg, 0.01–0.02% Zn and 0.04–0.05% Mn (López González et al., 2014; Rouf et al., 2010). However, other required nutrients for methanogens as sulphur, cobalt and nickel are lost or close to the lowest limit recommended (Demirel and Scherer, 2011). The most generalized use of press mud is as a fertilizer/soil improver directly applied on the fields or after composting. Due to difficulties with its transportation and soil capacity, higher amounts of press mud are left in piles and its drainage causes the pollution of nearby water bodies.

On the other hand, vinasse, main residue of alcohol production, is generated in volumes between 9 and 14 litres per litre of ethanol obtained (Christofoletti et al., 2013). It is characterized by a pH between 3.5 and 5.0, a dark brown colour and a high COD ranging between 50 and 150 g g L<sup>-1</sup> (Barrera et al., 2014; Wilkie et al., 2000). Vinasse contains some nutrients essential for bioconversion process such as 0.1–0.3 g L<sup>-1</sup> P<sub>2</sub>O<sub>5</sub>, 3.7–7.8 g L<sup>-1</sup> K, 0.4–5.2 g L<sup>-1</sup> Ca and 0.4–1.5 g L<sup>-1</sup> Mg (López González et al., 2015; Salomon and Lora, 2009). A lack of some important trace elements, as tungsten, manganese, selenium, zinc, cobalt, molybdenum, and copper below to the lowest limit were previously reported by Janke et al. (2015).

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The anaerobic digestion from vinasse and press mud separately (as monosubstrate) have been reported previously reaching methane yields of  $0.344 \text{ m}^3 \text{ kg}^{-1}$ COD removed and 0.250 m<sup>3</sup> kg<sup>-1</sup> COD removed, respectively (Harada et al., 1996; Sánchez et al., 1996). Main difficulties of the anaerobic digestion from vinasse have been attributed to the high salt concentrations (mainly K<sup>+</sup>), high sulfate levels, and the presence of phenolic compounds (Barrera et al., 2014; Driessen et al., 1994), while for press mud the fibrous material contained, as well as wax and proteins difficult its degradability, limiting the conversion efficiency. In a work reported by Janke et al. (2015) the phosphorous addition to balance C:P ratio, and sulphur addition to balance C:S ratio, were recommendations made to improve the anaerobic digestion process from vinasse and press mud, respectively. Thus, the codigestion of both substrates could be an alternative to complement macro-and micronutrient equilibrium, moisture balance and/or dilute inhibitory or toxic compounds. Besides, as press mud cannot be digested unless water or some liquid waste in replacement, and both residues are generated very near, transportation costs and water consumption could be reduced if co-digestion is applied.

In previous studies the co-digestion of press mud with others residues from sugar production have been reported (Janke et al., 2016; Rouf et al., 2010). Rouf et al. (2010) evaluated the biogas production for two mixtures ratio (2:1, 1:1) press mud: bagasse, and press mud: sugar cane straw in batch test. The biogas production was increased by 13% (press mud: bagasse) and 58% (press mud: sugar cane straw) for the same proportion of substrates in the mixture, respect to the press mud in mono-digestion. However, as the methane yield of bagasse and sugar cane straw were not determined, it is not possible to conclude if synergetic effects were contained in the mixtures investigated.

The before finding were different to that obtained by Janke et al. (2016) in a semicontinuo test. Those authors compared the process performance during semi-continuous mono-digestion of press mud versus the option of co-digestion with bagasse. The study was carried out for a mixture ratio press mud: bagasse of 2.33:1, equivalent to press mud (42%) and bagasse (58%) on volatile solid basis. The methane yield attained in co-digestion was 33% lower than the mono-digestion of press mud.

It is well known that the feed regime and high proportion of inoculum used during batch tests do not allow an adequately assessment of possible process inhibition during digestion of the substrates. The effect of the interaction of parameters in the co-digestion such as macronutrients, micronutrients, carbon/ nitrogen ratio, pH/alkalinity and inhibitors/toxic compounds, are better explored during semicontinuos regime. In semicontinuos regime, after a start-up period and stability of the process, where a dairy feeding is kept and the reactor work during long time, the inoculum can be shown adapted, and therefore can be evaluated with more precision the synergetic or antagonist effects of the mixture.

Previously, the co-digestion of vinasse with liquid and solid fractions from thermally pretreated press mud were explored (López González et al., 2015). As result, antagonistic effects were found for most mixtures examined, mainly when vinasse and liquid fraction were mixed. Nevertheless, the effect to blend untreated press mud with vinasse in anaerobic digestion process is not clarified yet.

Therefore, it was screened if the co-digestion of two major byproducts from sugar cane processing could be a novel process route for the industry. The first objective is to evaluate the synergetic and antagonistic effects of the mixture of press mud and vinasse co-digestion on the methane yield and methane production rate using batch tests, and the second is to compare the process performance during semi-continuous mono-digestion of press mud and its co-digestion with vinasse.

#### 2. Methods

#### 2.1. Materials

Fresh press mud and vinasse were provided from "Melanio He rnández" Sugar Mill (Sancti Spiritus, Cuba). Press mud was stored in plastic bags at 4 °C until use. Press mud contained 27.7% and 22.4% of total solids (TS) and volatile solids (VS), respectively. Vinasse (5.6% of TS and 4.1% of VS, on fresh matter) was collected, cooled and kept at -20 °C. The results of the substrates character-isation are shown in Table 1.

#### 2.2. Analytical methods

TS, VS and pH were determined according to standard methods (APHA, 1998). COD analysis for vinasse was carried out by standard closed reflux, colorimetric method 5220 D (APHA, 1998), while for press mud the modified method reported by Raposo et al. (2008) was applied. Total organic carbon (TOC) and Total nitrogen (TN) were determined by TOC analyzer and Kjeldahl method, respectively.

Sugars were determined by gas chromatography (GC Varian 3380) using a previously published methodology (López González et al., 2014). Lignin and structural carbohydrates were analyzed according to the NREL procedure (Sluiter et al., 2008).

#### 2.3. Anaerobic digestion

#### 2.3.1. Mixture design

To evaluate the synergetic and antagonistic effects of press mud (P) and vinasse (V) co-digestion a simplex-lattice design was employed. The design consists of pure blends ( $V_0/P_{100}$ ,  $V_{100}/P_0$ ) and mixture of two components ( $V_{25}/P_{75}$ ,  $V_{75}/P_{25}$ ,  $V_{50}/P_{50}$ ), expressed as COD percentages for each component. The response variables were  $y_{max}$ , ultimate methane yield (N mL CH<sub>4</sub> g<sup>-1</sup> COD<sup>-1</sup><sub>fed</sub>) and  $r_{sCH4max}$ , maximum methane production rate (N mL CH<sub>4</sub> g<sup>-1</sup> COD<sup>-1</sup><sub>fed</sub> d<sup>-1</sup>), determined by fitting the experimental data to Hill model (Eq. (1)) and its derivate (Eq. (2)), respectively.

$$y(t) = y_{\max} \frac{t^b}{K_M^b + t^b} \tag{1}$$

$$\mathbf{r}_{\mathrm{s}(\mathrm{t})} = \mathbf{y}_{\mathrm{max}} \cdot \frac{\mathbf{b} \cdot K_{\mathrm{b}}^{\mathrm{b}} \cdot t^{\mathrm{b}-1}}{\left(K_{\mathrm{b}}^{\mathrm{b}} + t^{\mathrm{b}}\right)^{2}} \tag{2}$$

Table 1

| Chemica | l composition | of press | mud | and | vinasse. |
|---------|---------------|----------|-----|-----|----------|
|---------|---------------|----------|-----|-----|----------|

| Parameters                     | Unit             | Press mud | Vinasse             |
|--------------------------------|------------------|-----------|---------------------|
| TS                             | % FM             | 27.7      | 5.3                 |
| VS                             | % TS             | 80.8      | 78.6                |
| Ash                            | % TS             | 19.2      | 21.4                |
| COD                            | $ m g~kg^{-1}$   | 284.5     | 62.3                |
| рН                             |                  | 6.3       | 4.8                 |
| Protein                        | $g kg^{-1}$      | 30.6      | 3.8                 |
| Lipids                         | $g kg^{-1}$      | 25.6      | 0.03                |
| Sugars                         | $g kg^{-1}$      | 22.5      | 31.6                |
| Cellulose                      | % TS             | 11.3      | NA                  |
| Hemicellulose                  | % TS             | 27.1      | NA                  |
| Lignin                         | % TS             | 9.3       | NA                  |
| TOC                            | ${ m g~kg^{-1}}$ | 122.5     | 21.7                |
| TN                             | $g kg^{-1}$      | 4.7       | 0.6                 |
| N-NH <sup>+</sup> <sub>4</sub> | $g kg^{-1}$      | ND        | $5.6 \cdot 10^{-3}$ |
| Phosphorus                     | $g kg^{-1}$      | 3.1       | 0.1                 |
| Sulfate                        | $g kg^{-1}$      | ND        | 1.8                 |
| C:N ratio                      |                  | 26:1      | 36:1                |

g kg<sup>-1</sup> refers to Fresh matter (FM), total solids (TS), volatile solids (VS), total organic carbon (TOC), total nitrogen (TN).

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