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Thermo-chemical pre-treatment to solubilize and improve anaerobic biodegradability of press mud

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HIGHLIGHTS

- The best pretreatment conditions (100 °C, $Ca(OH)_2$) on press mud were determined.
- COD solubilization and VFAs production were relationship to pretreatment severity.
- Both COD solubilization and methane yield were increased for pretreated press mud.
- Higher solubilization did not mean a biodegradability increase.

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ABSTRACT

Different pre-treatment severities by thermo-alkaline conditions (100 °C, $Ca(OH)_2$) on press mud were evaluated for different pre-treatment time and lime loading. COD solubilization and the methane yield enhancement were assessed. The biochemical methane potential was determined in batch assays under mesophilic conditions (37 ± 1 °C). The best pre-treatment resulted in a surplus of 72% of methane yield, adding 10 g $Ca(OH)_2$ 100 g^{-1} TS^{-1} for 1 h. Pre-treatment also increased the COD solubilization, but the optimal severity for COD solubilization as determined by response surface methodology did not ensure the highest methane production. Inhibitory effects on anaerobic digestion were noticed when the severity was increased. These results demonstrate the relevance of thermo-alkaline pre-treatment severity in terms of both lime loading and pre-treatment time to obtain optimal anaerobic biodegradability of lignocellulosic biomass from press mud.

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1. Introduction

Sugar is one of the most important components of the human diet. It stimulates cell processes in addition to its capacity to facilitate food intake by improving taste and texture. Sugar is produced in over 130 countries and the global production now exceeds 150 million tons a year. Approximately 70% of this production is derived from sugar cane plantations; the remaining 30% is obtained from sugar beet and other sugar crops (ISO, 2011). Sugarcane press mud is one of the main residues in the production process. When the juice is completely separated (clarification process), the residual mud is filtered to extract the suspended matter, including insoluble salts and fine bagasse and press mud is obtained. It represents around 2.8–4.5% of weight on crushed sugarcane basis (Velarde et al., 2004). Merely in Cuba, around 43.5×10^3 tons of press mud are generated yearly from the sugarcane industry, becoming an important high strength stream.

As a general rule of practice in sugar-producing countries, press mud is currently spread onto the soil and used as fertilizer, whether as raw material or after composting. Properties like high organic matter content and the presence of essential macro and micro nutrients make this readily available biomass attractive for bio-conversion processes. Anaerobic digestion is one of the most common technologies which can be suitable to treat sugarcane press mud. Previous studies have reported methane yields of 0.25 L CH_4 g^{-1} COD^{-1} removed and 0.154 L CH_4 g^{-1} press mud (Sánchez et al., 1996; Rouf et al., 2010). Biogas production rates even exceeded those mentioned when press mud was co-digested with cow dung, bagasse or cane pith (Rouf et al., 2010).

As press mud contains up to 85% of insoluble organic matter (Sánchez et al., 1996) with high fiber (lignocellulose) content, its

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biodegradability is expected to enhance as a result of applying different pre-treatments. It has been shown that pre-treatment of lignocellulosic biomass can accelerate the hydrolysis process and improves the biogas production (Pavlostathis and Giraldo-Gomez, 1991). At present, there are different pre-treatment methods including mechanical, physical, thermal, and chemical (i.e., alkali, acidic, oxidative), as well as biological methods (Hendriks and Zeeman, 2009; Carlsson et al., 2012). Among these, lime pre-treatment is a promising one, as levels of anaerobic digestion inhibitors (like furfural, hydroxymethylfurfural and soluble phenolic compounds) released during or after alkali pre-treatment are low and the pH value is increased thereby counteracting the acid production in the reactor. Moreover, lime pre-treatment solubilizes lignin, removing approximately 33% of lignin and nearly 100% of the acetyl groups. The action of lime is slower than other chemicals but its low cost and safe handling makes it a very attractive option (Wyman et al., 2005).

From literature revision, only one paper aimed to treat press mud by alkaline pre-treatment with $Ca(OH)_2$ (López et al., 2005). The experiments were conducted at 30 °C with continuous agitation, attaining solubilization levels of 14.5% when 3.18 g L^{-1} during 7 h were added. Methane production was increased regarding control samples. In addition, studies concerning a proper combination of thermal and alkaline pre-treatments applied to other biomass such as bagasse, waste activated sludge, spent microbial biomass and dewatered pig manure, were performed with positive results (Chang et al., 1998; Tanaka et al., 1997; Penaud et al., 1999; Rafique et al., 2010). Thermo-alkaline pre-treatment is usually carried out at temperatures ranging from 100 to 150 °C with a lime loading of approximately 0.1 g $Ca(OH)_2$ 100 g^{-1} TS^{-1} (Chang et al., 1998).

The objective of the present paper is to verify the potential of a thermo-alkaline pre-treatment at 100 °C in order to enhance the anaerobic digestion of press mud in terms of methane yield augmentation. The effects of the lime loading and pre-treatment time on chemical oxygen demand (COD) solubilization and volatile fatty acids (VFAs) production were determined for different experimental conditions.

2. Methods

2.1. Materials

Fresh press mud was provided from the "Melanio Hernández" Sugar Mill (Sancti Spiritus, Cuba) during 2011 harvest. Press mud was air-dried, milled and sieved to a particle size of less than 2 mm. It was subsequently stored in plastic bags at 4 °C until use.

2.2. Analytical methods

Total COD (tCOD), total solids (TS), volatile solids (VS), ashes and pH were determined according to standard methods (APHA et al., 1995). Samples for soluble COD (sCOD) and volatile fatty acids (VFAs) were centrifuged at 6000 rpm for 15 min in a Hermle Z300 centrifuge. After centrifugation, only the supernatant was used for the analysis of sCOD and VFAs. COD analysis was carried out by standard closed reflux, colorimetric method 5220 D (APHA et al., 1995). VFAs (acetic acid, propionic acid, butyric acid, valeric acid and iso-valeric acid) were determined by gas chromatography (GC) using an Agilent 7820A with a split/splitless injector at 250 °C, Econocap EC 1000 column, oven temperature program: 80 to 120 °C and FID detection, with helium as a carrier gas.

Potassium, iron, calcium, cobalt, copper, sodium, manganese, magnesium and zinc ion contents were analyzed by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) using a Varian Vista-MPX spectrometer.

Elemental analysis (C, H and N) were quantified using a Perkin Elmer 2400 Series II CHNS/O Elemental Analyzer. Elemental oxygen content was calculated by subtracting measured weigh percentages of C, H and N from the dry matter. The remainder was assumed to be composed of elemental oxygen. Sulfate, phosphate and chloride were determined by ion chromatography.

The neutral detergent fiber (NDF), acid detergent fiber (ADF) and acid detergent lignin (ADL) were analyzed according to the method of Van Soest et al. (1991). Hemicellulose and cellulose content were calculated as the difference between NDF and ADF, and ADF and ADL, respectively.

The lower heating value was determined in a standard bomb calorimeter.

2.3. Pre-treatment conditions

The press mud (15 g) was presoaked at 30 °C in a $Ca(OH)_2$ solution using an orbital shaker for15 min at 150 rpm. Different conditions of lime loading (LL) and pre-treatment time (t) (Table 1) were used according to a Central Composite Design (CCD) setup. Afterwards the samples were put in an oven at 100 °C and manually shaken every 30 min. A constant water loading value of 10 g water g^{-1} TS^{-1} press mud was used for all experiments (Chang et al., 1998). Schott bottles of 500 mL were used as reactors. After the fixed pre-treatment time, the reactors were placed in ice water for 5 min to stop the reaction. The best trials in terms of improved COD solubilization (S_{COD}) were anaerobically digested. S_{COD} was calculated as sCOD/tCOD and expressed in %.

To express the impact of the pre-treatment for both t and LL, a severity factor was calculated as defined by Silverstein et al. (2007) (Eq. (1)), where Mo is the modified severity parameter, t is the residence time (min), C is the chemical concentration (wt.%), T_r is the reaction temperature (°C), T_b is the base temperature (100 °C) and n is an arbitrary constant obtained with the best model fits while keeping the log value positive for S_{COD} behavior in relation to the severity factor (Silverstein et al., 2007).

$$\mathrm{Mo} = C^n \cdot t \cdot \exp\left((T_r - T_b)/14.75\right) \quad (1)$$

2.4. Central composite design (CCD) and evaluation by response surface methodology (RSM)

To determine the influence of the LL and t over COD solubilization, a CCD (Montgomery, 2005) setup was designed. Experiments were carried out in two triplicated blocks, one for factorial points and another for axial points. In addition, eight center points were included along the experimental blocks to provide orthogonality and to estimate the experimental error (Montgomery, 2005). In total, 32 trials were run (Table 1).

Response surface modeling was used to determine the best pre-treatment conditions as well as to explore COD solubilization

Table 1
CCD setup. Triplicate experiments are indicated with the appropriate abbreviation. Empty cells are not tested in the CCD.

LL[a]	Time (h)				
	0.59	1	2	3	3.41
2.76			A2_3		
4		A1_4		A3_4	
7	A0_7		A2_7		A3_7
10		A1_10		A3_10	
11.24			A2_11		
Total runs	3	6	14	6	3

[a] g $Ca(OH)_2$ 100 g^{-1} TS^{-1}.

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