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Optimization of semi-continuous anaerobic digestion of sugarcane straw co-digested with filter cake: Effects of macronutrients supplementation on conversion kinetics

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ABSTRACT

Anaerobic digestion of sugarcane straw co-digested with sugarcane filter cake was investigated with a special focus on macronutrients supplementation for an optimized conversion process. Experimental data from batch tests and a semi-continuous experiment operated in different supplementation phases were used for modeling the conversion kinetics based on continuous stirred-tank reactors. The semi-continuous experiment showed an overall decrease in the performance along the inoculum washout from the reactors. By supplementing nitrogen alone or in combination to phosphorus and sulfur the specific methane production significantly increased ($P < 0.05$) by 17% and 44%, respectively. Although the two-pool one-step model has fitted well to the batch experimental data ($R^2 > 0.99$), the use of the depicted kinetics did not provide a good estimation for process simulation of the semi-continuous process (in any supplementation phase), possibly due to the different feeding modes and inoculum source, activity and adaptation.

1. Introduction

Sugarcane straw (SCS), also known as tops and trash, accounts for approximately one third of the total primary energy of the sugarcane, and therefore nowadays major efforts are being carried out by many research initiatives to develop feasible options for the conversion of this residual biomass fraction into useful products (Leal et al., 2012; Sindhu et al., 2016).

However, due to the important agronomic benefits provided by SCS blanketing, such as protection against soil erosion, increased biological activity, water infiltration as well as weed and temperature control, its full recovery from the fields for further utilization would be limited to a fraction of the total SCS (Hassuani, 2005; Leal et al., 2013). For this reason, earlier studies have considered only 50% of the total SCS generated to be used as fuel, together or in substitution of bagasse, in the existing co-generation plants at the sugarcane mills or as source of lignocellulosic biomass for second-generation (2G) bioethanol production (Dias et al., 2011; Walter and Ensinas, 2010).

In this context, anaerobic digestion (AD) is a promising alternative strategy for utilization of the full potential of SCS, since part of the organic matter would be converted to methane and/or platform chemicals for value-added products, and the non-degraded material (digestate) could be a valuable source of minerals and organic matter (*i.e.* recalcitrant fraction) for recycling on the fields (Janke et al., 2016a; Leite et al., 2015a,b). Alone or in combination with another conversion pathway (*e.g.* 50% for AD and 50% for 2G bioethanol), the AD of SCS would contribute to the diversification of the product portfolio of the sugarcane mills in a sustainable biorefinery concept and improve their robustness against market fluctuations of the main products.

Although the biogas potential of SCS has been previously assessed in batch tests (De Paoli et al., 2011; Janke et al., 2016b), to our knowledge the semi-continuous AD of SCS has not been studied yet. Only by applying a similar feeding regime used during large-scale applications (semi-continuous) it is possible to have a broader understanding of the process behavior in terms of effect of inhibitors, nutrient deficiencies, optimum organic loading rate (OLR) and hydraulic retention time (HRT) for a satisfactory and efficient conversion of the substrate to methane.

In a recent study on the main characteristics of sugarcane waste for biogas production (Janke et al., 2015), it was shown that SCS has an

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unfavorable balance of macronutrients and trace elements for AD. The C:N:P:S ratio was 600:7:1:1, which represents a much lower nitrogen, phosphorus and sulfur content than the recommended values of 600:15:5:3 (FNR, 2010). These macronutrients are the main cell constituents and if found in lower concentrations it may negatively affect the functioning of the microbial communities, causing an incomplete conversion of the substrate, resulting in lower methane yields. In addition, the trace elements Mn, Co, Zn, W, Se and Ni were also found in concentrations below or close to the minimum range recommended for AD by Kayhanian and Rich (1995) and by Oechsner et al. (2008). Thus, the lack of those elements could potentially affect the efficiency and stability of the process, since trace elements are essential constituents of cofactors and enzymes in AD (Schmidt et al., 2014a).

A potential solution to minimize those drawbacks is the co-digestion of SCS with another waste fraction from the sugar and bioethanol production such as sugarcane filter cake (SFC), because this material is rich in trace elements and has a more favorable balance of macronutrients (600:25:8:2.5) (Janke et al., 2015). However, it is unclear whether SFC could provide enough nutrients for an efficient AD of SCS, since different amounts are generated per ton of sugarcane (TC) between SCS (140 kg TS TC^{-1}) and SFC (35–40 kg FM TC^{-1}), besides of both materials being formed by a lignocellulosic structure that prevents the solubilization of carbon and nutrients for microbial uptake.

In this study, the semi-continuous AD of SCS co-digested with SFC in a substrate mixture of 70% FM of SCS and 30% FM of SFC was performed to assess whether the supplementation of macronutrients (N-P-S) could still provide additional benefits to the process in terms of substrate conversion to methane. Considering typical conditions found in the Brazilian sugarcane mills, this substrate mixture would correspond to approximately 50% FM of the total SCS and 100% FM of SFC generated. Assuming that digestate could provide the same benefits of SCS blanketing, this AD concept could potentially allow the full utilization of SCS without competition for biomass since 50% of SCS would still be available for other conversion pathways, such as co-generation or 2G bioethanol production.

Furthermore, substrate characteristics, such as the total methane/biogas potential as well as the conversion kinetics (*e.g.* first-order reaction constants), are commonly determined based on laboratory batch tests. Although extensive research and standard protocols (*e.g.* VDI 4630) for the conduction of anaerobic batch tests have been optimized and standardized in the past years (Angelidaki et al., 2009; Holliger et al., 2016), the results and significance are still affected by numerous influencing factors, such as the source or pre-treatment of the inoculum used (De Vrieze et al., 2015). In addition, the validity of batch tests to describe continuously operated anaerobic reactors is rarely investigated or proven (Batstone et al., 2009). Therefore, in this study an assessment of the degradation kinetics was also carried-out to compare the performance of the AD process both in batch and semi-continuous feeding mode.

2. Methods

2.1. Substrate and inoculum

Samples of SCS and SFC were obtained from a sugarcane mill in the state of Goiás (Brazil) during the 2014/2015 season, transported to Germany in sealed plastic drums, homogenized by using a milling machine SM 200 (Retsch, Germany) with a 2 mm sieve (only for SCS) and stored at 4 °C until its use. A large-scale biogas plant that uses maize silage and cattle manure as substrate provided fresh digestate, which was used as inoculum for the batch and semi-continuous experiments.

2.2. Batch experiment

The methane yields of SCS and SFC were obtained through biochemical methane potential (BMP) tests according to VDI (2006) using an Automatic Methane Potential Test System II (Bioprocess Control, Sweden) under mesophilic temperature (38 ± 1 °C) during 30 days. Prior to the tests, the inoculum was degassed at 38 °C during approximately 7 days to reduce the non-specific biogas generation. The experiment was carried-out in duplicate by using 600 mL glass bottles (400 mL working volume). The headspace of the reactors were flushed with nitrogen to create anaerobic conditions. To prevent inhibition, the ratio of substrate/inoculum (gVS^{-1}) was set to around 0.25 (*i.e.* 4 times higher amount of inoculum than substrate based on VS).

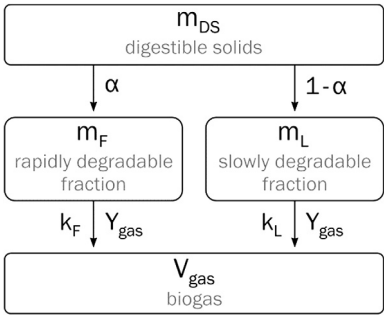

Fig. 1. Components and parameters of the utilized model structure – model C (adopted from Brulé et al. (2014)).

Considering the different model derivations presented by Brulé et al. (2014) an exponential two-pool one-step model (model C) was used to evaluate the methane production kinetics of the batch experiment. This modelling approach differentiates between rapidly and slowly degradable fractions (two-pool) of the available substrate, as shown in Fig. 1. Thus, four model parameters and constants needed to be adjusted to depict the respective measurement results: the total methane potential S_{BMP} (mL gVS^{-1}), the ratio of rapidly degradable substrate to total degradable substrate α and the two first-order reaction constants for the degradation of rapidly degradable substrate k_F (d^{-1}), and slowly degradable substrate k_L (d^{-1}). The model implementation as well as the numeric parameter identification (Levenberg-Marquard algorithm) was performed in the software environment Matlab (Mathworks, USA).

2.3. Semi-continuous experiment

Four lab-scale continuous stirred-tank reactors (CSTR) with 5 L total volume (3 L working volume) were used for this experiment. The reactors were continuously stirred (100 rpm) using a central stirrer with vertical shaped blades to reduce the formation of floating layers. The operating temperature was kept under mesophilic conditions (38 ± 1 °C) by recirculating hot water through the double-walled reactors.

The experiment was carried-out during 300 consecutive days with the same feeding frequency (once per day), co-digestion mixture (8.4 g SCS FM + 3.6 g SFC FM) and water (40 mL) to adjust the solids concentration to 169 g L^{-1} (wet digestion process). Thus, providing to the process a constant OLR of 2.0 gVS L^{-1} d^{-1} and a HRT of 58 days during the entire experiment. For the start-up period (days 0–83) all reactors were kept in parallel until most of the original inoculum was assumed to be washed-out from the reactors. During the nitrogen supplementation phase (days 84–175) 104 mg d^{-1} of urea ($(NH_2)_2CO$) were daily added to the reactors R3 and R4, while the reactors R1 and R2 were kept as control until the end of the experiment. For the nitrogen-phosphorus-sulfur supplementation phase (days 176–300) the reactors R3 and R4 were supplemented with a macronutrient solution composed of urea (104 mg d^{-1}), monopotassium phosphate (47.25 mg d^{-1} of KH_2PO_4) and sodium sulfate (27.50 mg d^{-1} of Na_2SO_4) to balance the C:N:P:S ratio according to FNR (2010).