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Total solids content drives high solid anaerobic digestion via mass transfer limitation

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1. Introduction

The production of waste including municipal solid waste (MSW) has significantly increased in recent years. For example, in France, MSW generation has more than doubled in less than 50 years, from 175 kg year⁻¹ capita⁻¹ in 1960 to 354 kg year⁻¹ capita⁻¹ in 2006 (ADEME, 2009). Different technologies are used for MSW treatment, such as incineration or composting, but anaerobic digestion (AD) processes are particularly interesting in terms of organic matter reduction by converting organic compounds into biogas. The methane content in biogas represents a source of renewable energy.

Based on the total solids (TS) content of solid waste, three main types of technologies have been developed: wet ($\leq 10\%$ TS), semidry (10–20% TS) and dry ($\geq 20\%$ TS) processes. Dry technologies, also called "high-solid" anaerobic processes, are attractive because the quantity of water added to the raw waste is substantially reduced, and consequently, the digester size is minimized. However, difficulties in operating dry anaerobic digestion at both laboratory and industrial scales are related to the high concentration of total solids. Fernández et al. (2008) showed that methane production started at day 14 in a reactor with 20% TS and at day 28 in a reactor with 30% TS in mesophilic dry anaerobic batch reactors treating the organic fraction of municipal solid waste. The total methane production was 17% lower at 30% than at 20% TS. This result is consistent with the one obtained by Forster-Carneiro et al. (2008), which

ABSTRACT

The role of the total solids (TS) content on anaerobic digestion was investigated in batch reactors. A range of TS contents from 10% to 35% was evaluated, four replicates were performed. The total methane production slightly decreased with TS concentrations increasing from 10% to 25% TS. Two behaviors were observed at 30% TS: two replicates had similar performances to that at 25% TS; for the two other replicates, the methane production was inhibited as observed at 35% TS. This difference suggested that 30% TS content corresponded to a threshold of the solids content, above which methanogenesis was strongly inhibited. The Anaerobic Digestion Model No. 1 (ADM1) was used to describe the experimental data. The effects of hydrolysis step and liquid/gas mass transfer were particularly investigated. The simulations showed that mass transfer limitation could explain the low methane production at high TS, and that hydrolysis rate constants slightly decreased with increasing TS.

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showed better performance of anaerobic reactors operated at 20% TS compared to 25% and 30% TS. It was suggested that a high TS content could reduce substrate degradation and, therefore, biogas production (Fernández et al., 2008). Le Hyaric et al. (2011) used a microbial metabolic intermediate, propionate, as substrate to study specifically the methanogenic activity in dry reactors. The specific methanogenic activity increased linearly by a factor of 3.5 when the moisture content increased from 65% to 82% (corresponding to 35–18% TS, respectively).

A high TS content also affects the physical properties of the digested solid waste. Water distribution was investigated by Garcia-Bernet et al. (2011a) in biowastes and associated digestates sampled in industrial dry AD plants. Even if the proportion of the hydration and vicinal water fractions is small $(0.1 \text{ g}_{water} \text{ gDM}^{-1})$ compared to the total amount of water, the capillary water fractions of the digestates ranged from 2 to 2.5 g_{water} gDM⁻¹: this fraction can represent up to 60% of the total water for a digestate having a TS = 20%. As a consequence, for dry AD systems, the high solids content strongly affects the rheological behavior of the digestates (Battistoni et al., 1993; Battistoni, 1997; Garcia-Bernet et al., 2011b). Digested media are visco-elastic materials characterized by high yield stress levels, and yield stress increases with TS content according to an exponential law (Garcia-Bernet et al., 2011b). Digesters are thus difficult to mix and homogenize. Indeed, Karim et al. (2005) showed that, when the concentration of TS increases in reactors, mixing becomes more important for improving the production of methane. Nevertheless, under unmixed conditions, transport is likely governed by diffusion processes, which





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are strongly related to the porosity of the media and, thus, to the water content. Therefore, the diffusive transport resistance of soluble compounds (substrate or by-products) may have a strong influence on anaerobic digestion performance in dry systems.

The aim of this study is to assess the impact of the total solids content on anaerobic digestion of solid waste. Anaerobic batch reactor experiments were performed with TS concentrations ranging from 10% to 35%. The methane production performances were assessed. To better evaluate the impact of the water content on anaerobic digestion, the Anaerobic Digestion Model No. 1 (ADM1) was then used to describe the experimental data. The objective of the model application was to test some hypotheses in order to understand why the TS content affects the global anaerobic digestion performance. The effects of hydrolysis step and liquid/gas mass transfer were more particularly investigated.

2. Methods

2.1. Substrate characterization

2.1.1. Van Soest fractionation

The content of cellulose, hemicellulose and lignin-like fractions in the substrate was analyzed according to the Van Soest procedure (Van Soest, 1963). After shredding and sieving, 1 g of cardboard was placed in a Fiberbag system (Gerhardt Germany). The sequential fractionation procedure was performed for six samples as follows: (1) The soluble compounds were obtained by extraction with a neutral detergent ($30 \text{ g L}^{-1} \text{ C}_{12}\text{H}_{25}\text{Na04S}$; 18.61 g L⁻¹ C₁₀H₁₄N₂Na₂O₈, 2H₂O; 6.81 g L⁻¹ Na₂B₄O₇, 10H₂O; 4.56 g L⁻¹ Na₂HPO₄; 10 mL L⁻¹ C₆H₁₄O₄) at 100 °C for 60 min; (2) the hemicellulose-like compounds were extracted by an acid detergent ($20 \text{ g L}^{-1} \text{ C}_{19}\text{H}_{42}\text{NBr}$; 26.7 mL L⁻¹ H₂SO₄, 95–97%) for 60 min at 100 °C; (3) the lignin-like compounds were obtained by removing the cellulose-like compounds for 3 h with H₂SO₄ (72%). The remaining fraction corresponds to lignin. At the end of each step, the extracted samples were washed with deionized water and oven-dried at 100 °C before the transition to the next step.

2.1.2. The biochemical methane potential

The biochemical methane potential (BMP) of the cardboard was assessed according to Angelidaki and Sanders (2004). The assays were conducted in triplicate (and one blank) in 600 mL serum bottles at 35 °C, with a working volume of about 400 mL. The serum bottles were filled with synthetic growth medium containing nutrients and trace elements, and inoculated with granular sludge from a mesophilic anaerobic digester of a sugar factory. The final sludge concentration in the bottles was 20 gVS L⁻¹. The bottles were loaded with 1 g of cardboard (corresponding to 0.77 gVS). Biogas production and composition were measured daily. The methane production is expressed under standard condition and accounts for the variation of the gas content in the headspace of the reactors. The calculated BMP accounts for the global methane production measured with the blank assay.

2.2. Batch preparation and operating conditions

A compact cardboard with a density of 1.42 kg m⁻³, branded "Cartonnages Michel", was used as a substrate since cardboard represents usually the largest proportion of organic compounds in municipal solid waste (21.5%) after putrescible waste (32.2%) (ADEME, 2009). The cardboard was shredded using a cutting mill SM-100 and sieved at 2 mm.

Experiments were carried out in 600 mL batch flasks with a working volume of 100 mL. A mixture of cardboard, water, inocu-

lum and oligo-elements was prepared to reach six TS contents from "wet" to "dry" anaerobic conditions: TS = 10%, 15%, 20%, 25%, 30%, 35%. A substrate to biomass ratio S/X of 20 (w/w) was used to limit the influence of the inoculum composition on the degradation, S and X representing the initial TS contents of the substrate and the biomass, respectively. The inoculum corresponded to a leachate of pressed MSW digestate sampled in an industrial plant treating MSW. One mL of an oligo-element solution was added to the mixture. This oligo-element solution was composed of: FeCl₂, 4H₂O (2 g L⁻¹), CoCl₂, 6H₂O (0.5 g L⁻¹), MnCl₂, 4H₂O (0.1 g L⁻¹), NiCl₂, 6H₂O (0.1 g L⁻¹), CuCl₂, 2H₂O (0.04 g L⁻¹), Na₂SeO₃ (0.05 g L⁻¹), CuCl₂, 2H₂O (0.04 g L⁻¹), Na₂MoO₄, 2H₂O (0.01 g L⁻¹). The experiments were run over 298 days under mesophilic conditions (35 °C) without mixing. Each TS condition was tested in four replicates.

2.3. Analytical methods

2.3.1. Biogas quantification and composition analysis

Biogas production and composition were determined daily during the first 2 months, and then once a week after the exponential phase of methane production.

The biogas production was measured by the water displacement method and then normalized according to the ambient temperature. The biogas composition was determined using a gas chromatograph (Varian μ GC-CP4900) by injecting a sample volume of 2 mL. This gas chromatograph was equipped with two columns: a Molsieve 5A PLOT column for O₂, N₂, CH₄ and CO and a HayeSep A column for CO₂ quantification. The calibration was carried out with a standard gas composed of 25% CO₂, 2% O₂, 10% N₂ and 63% CH₄. The temperatures were 30 °C for the oven and 100 °C for the injector and the thermal conductivity detector. The gas carrier of the flow was Helium.

2.3.2. VFA and pH analysis

Volatile fatty acids (VFA) were analyzed at the end of the batch test. After centrifugation (13,000 rpm, 15 min), VFA concentrations were measured by a gas chromatograph (Varian μ GC-CP3900) equipped with a flame ionization detector (FID). The column used was a semi-capillar Econocap FFAP (Alltech). The gas carrier of the flow was nitrogen. The rest of the assay technique is described in Ganesh et al. (2010).pH was measured directly on the digestate using a pH meter Eutech Instruments pH510 with Mettler Toledo InLab[®] Expert Pt1000 pH electrodes.

2.4. Mathematical model

The Anaerobic Digestion Model No. 1 (ADM1) (Batstone et al., 2002, 2006) was used to describe the experimental data. The characterization of the shredded cardboard with respect to ADM1 state variables, in particular carbohydrates and particulate inerts, was based on the results of the Van Soest fractionation and the degradable fraction of fibers (hemicellulose and cellulose). The degradable fraction of hemicellulose and cellulose (major degradable constituents of cardboard) was calibrated with the gas curve at 10% TS. The estimated value of 40% is of the same order of magnitude as the degradable fraction of fibers reported by Koch et al. (2010) for grass silage. In the model, the state variable for carbohydrates, X_{ch}, was considered to be composed of the biodegradable fraction of hemicellulose and cellulose, and the soluble organic fraction. The non-degradable fraction of hemicellulose and cellulose, as well as the lignin fraction were considered as particulate inerts (X_I). Overall, 0.33 gCOD gTS⁻¹ were attributed to X_{ch} and 0.85 gCOD gTS⁻¹ to X_I. Simulations were run in Matlab/Simulink (Version 7.3.0).

The model was calibrated to the experimental data of the batch experiments at 10% TS using a trial and error approach. Compared

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