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Dynamic effect of total solid content, low substrate/inoculum ratio and particle size on solid-state anaerobic digestion

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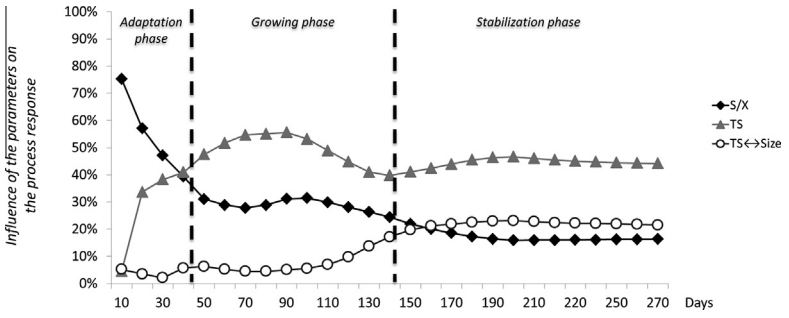
HIGHLIGHTS

- Total solid content is the main factor that drives solid-state anaerobic digestion.
- *S*/*X* ratio has an impact on the start-up phase of dry anaerobic digestion.
- The start-up phase is affected by the soluble fraction of fine particle.
- Both TS content and particle size modify the apparent water quantity.

GRAPHICAL ABSTRACT



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ABSTRACT

Among all the process parameters of solid-state anaerobic digestion (SS-AD), total solid content (TS), inoculation (*S*/*X* ratio) and size of the organic solid particles can be optimized to improve methane yield and process stability. To evaluate the effects of each parameter and their interactions on methane production, a three level Box-Behnken experimental design was implemented in SS-AD batch tests degrading wheat straw by adjusting: TS content from 15% to 25%, *S*/*X* ratio (in volatile solids) between 28 and 47 and particle size with a mean diameter ranging from 0.1 to 1.4 mm. A dynamic analysis of the methane production indicates that the *S*/*X* ratio has only an effect during the start-up phase of the SS-AD. During the growing phase, TS content becomes the main parameter governing the methane production and its strong interaction with the particle size suggests the important role of water compartmentation on SS-AD.

1. Introduction

Anaerobic digestion (AD) of solid wastes, such as agricultural residues or organic fraction of municipal solid waste, is a process in constant development at industrial scale because of its capacity to degrade organic matter simultaneously into a valuable biogas composed of methane (CH_4) and carbon dioxide (CO_2) and into a nutrient-rich digestate with agronomic qualities (Karthikeyan and Visvanathan, 2012). Total solid (TS) content of the medium within the anaerobic digester is used to define two technologies: wet AD for TS below 15% and Solid-State AD (SS-AD) for TS content higher than 15 % (Mata-Alvarez et al., 2000; De Baere and Mattheeuws, 2010). Operating in dry conditions allows the increase of the substrate concentration, which leads to the reduction of the digester volume for a given organic loading rate, the reduction of specific energy consumption for heating the digester and the simplification of the final step for the digestate dewatering (Karthikeyan and Visvanathan, 2012). These advantages resulted in an early development of the SS-AD systems at the industrial scale.

Because of a lack of scientific knowledge, SS-AD processes are not optimized and performances could therefore be improved. The methane yield and stability of industrial digesters are usually dependent of the empirical knowledge of the operators. Dry anaerobic digesters are mainly handled in batch mode or continuous plug-flow mode depending on the origin and quantity of the

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substrate. Several process parameters can be optimized to operate SS-AD: TS content, *S*/*X* ratio, physico-chemical pretreatment of the substrate, mixing mode, temperature, etc. (Karthikeyan and Visvanathan, 2012).

A main parameter that drives SS-AD is the water content (i.e., TS content) (Abbassi-Guendouz et al., 2012). SS-AD industrial plants are operated from 20% to 30%TS and up to 40%TS for some technologies (Karthikeyan and Visvanathan, 2012). It is mostly admitted that increasing TS content leads to lower process performances in terms of methane production and substrate conversion (Staley et al., 2011; Abbassi-Guendouz et al., 2012). Some authors observed that TS content affects the various steps of the AD process: hydrolysis, acidogenesis, acetogenesis and methanogenesis (Fernández et al., 2001; Valdez-Vazquez and Poggi-Varaldo, 2009; Le Hyaric et al., 2012). However, if the dynamics of dry anaerobic ecosystems depends on TS content, only one dynamic study has yet been published (Shi et al., 2013).

Inoculation phase is an important step affecting SS-AD, both in batch and continuous mode (digestate recycling). Inoculation is usually characterized by the substrate (*S*) to inoculum (*X*) ratio (*S*/*X*), for example on a volatile solid (VS) basis ($g_{VS\text{-}S}\ g_{VS\text{-}X}^{-1}$). At the industrial scale, SS-AD generally involves very low *S*/*X* ratios in order to maximize reaction kinetics and to avoid the risks of AD failure ($S/X \approx 0.3$–0.5 in VS basis) (Guendouz et al., 2010; Xu et al., 2013). At the laboratory scale, *S*/*X* ratios between 2 and 6 (in VS basis) are typically used, suggesting that *S*/*X* ratio actually applied in industry can be optimized (Brown et al., 2012; Liew et al., 2012; Lü et al., 2012; Xu et al., 2013). By considering only the first 30 days of operation, some authors demonstrated an inefficiency of AD digestion for *S*/*X* higher than 4 (Cui et al., 2011; Liew et al., 2012). However, experiments operated under higher *S*/*X* ratio were successfully realized (10 in VS basis) (Abbassi-Guendouz et al., 2012) but required an adaptation phase longer than 30 days and a duration of experiment higher than 250 days in order to achieve the AD reaction. The main advantage of operating under high *S*/*X* ratio is the observation of changes in the microbial communities, which are not detectable at low *S*/*X* ratios. Therefore, for an academic purpose, the main advantage of a low inoculation is the simplification of the microbial diversity of the dry anaerobic media. Moreover, this approach allows the study of the dry anaerobic process by an important deceleration of the process dynamics.

The reduction of the particle size represents an interesting pretreatment of solids for biological transformations since it is not necessary to add water or chemical substances, and the relative technical simplicity (Barakat et al., 2013). It allows higher biological processes kinetics through a release of dissolved organic matter (Müller, 2003), as well as an increase of solid particles surface area and pore volume, and a modification of the lignocellulosic structure such as the crystallinity of the cellulose or lignin distribution (Monlau et al., 2013). Several authors investigated the effect of particle size on the anaerobic digestion process, but only in wet AD. Comminution generally increases biogas yield and degradation kinetics (Sharma et al., 1988; Mshandete et al., 2006; Lindmark et al., 2012). However, contradictory results have been obtained probably because of the release of inhibitory compounds (Izumi et al., 2010) or the low effect of the pretreatment on the available surface area (Pommier et al., 2010). If an economical optimum of particle size reduction is around 1 mm (Barakat et al., 2013), a biological optimum for AD may also exist as suggested by Izumi et al. (2010) (0.6 mm for food waste) or Dumas et al. (2010) (0.2-0.3 mm for wheat straw).

The influence of operating parameters on AD is often studied separately and contradictory results found in the literature may be attributed to the interdependency (interaction) of these parameters. To our knowledge, the simultaneous effect of TS content, *S*/*X* ratio and particle size has not yet been investigated. However, due to the high sensitivity of dry media, their interaction may be critical for keeping adequate AD performances, as a strong interaction between TS content and *S*/*X* ratio (Forster-Carneiro et al., 2007, 2008). According to the literature, three selected factors have been investigated within the range of critical zone effects. The TS content was studied between 15% and 25%TS to investigate the limit between wet and dry AD (Forster-Carneiro et al., 2007). A *S*/*X* ratio between 47 and 28 (on a VS basis) was selected to identify the dynamic of the SS-AD (Lü et al., 2012). The size of particles was reduced from 0.1 to 1.4 mm (Dumas et al., 2010; Izumi et al., 2010). A Box–Behnken experimental design (Wang and Wan, 2009) has been applied in the purpose of analyzing the TS content, *S*/*X* ratio and particle size effects and their interaction on the SS-AD dynamics of lignocellulosic residues.

2. Methods

2.1. Substrate

Wheat straw (*Triticum aestivum*) was used as a model of the lignocellulosic substrate available for dry anaerobic digestion. The substrate used in this experiment was harvested in summer 2010 in an organic farm located in southern France (Hérault). Straw bales were homogenized and stored at ambient temperature. Three size fractions, coarse, medium and fine, were prepared by using a cutting miller. Average particle sizes were obtained by a gravimetric technique (coarse and medium fraction) or a laser granulometer (fine fraction) depending on the technical limitations. The coarse fraction was prepared by milling straw with a 10 mm grid and then sieving between grids of 4 and 10 mm (average particle size of 1.45 mm). The medium fraction was obtained by milling with a 1 mm grid, and sieving between grids of 1 and 4 mm (average particle size of 0.67 mm). The fine fraction was milled with a grid of 0.25 mm (average particle size of 0.11 mm).

2.2. Experimental device in dry conditions

The inoculum was sampled from a mesophilic SS-AD pilot treating organic fractions of municipal solid waste. A liquid phase was extracted by centrifugation (3000*g*, 15 min, 4 °C) and used to inoculate the batch tests. SS-AD batch tests were obtained by incorporating 100 g of medium into a 500 mL sealed flask. The composition of the medium was calculated to sustain the experiment conditions. First, about 15, 20 and 25 g of straw were added for TS content of 15%, 20% and 25% respectively, while taking into account the slight variations of TS content of the three particle sizes (0.11, 0.67 or 1.45 mm). Then, the quantity of inoculum was adjusted to respect the *S*/*X* ratio (28, 37.5 or 47 in VS). Finally, water was added, to sustain the final TS content. To maintain a constant *C*/*N* ratio of about 40, NH_4Cl was added, its quantity depending on the *S*/*X* ratio. In each flask, 5.2 g of bicarbonate buffer (H_2CO_3), corresponding to an initial pH of 8.4, were added as well as 1 mL of a trace element solution having the following composition: $FeCl_2{\cdot}4H_2O$ (20 mg L^{-1}), $CoCl_2{\cdot}6H_2O$ (5 mg L^{-1}), $MnCl_2{\cdot}4H_2O$ (1 mg L^{-1}), $NiCl_2{\cdot}6H_2O$ (1 mg L^{-1}), $ZnCl_2$ (0.5 mg L^{-1}), H_3BO_3 (0.5 mg L^{-1}), Na_2SeO_3 (0.5 mg L^{-1}), $CuCl_2{\cdot}2H_2O$ (0.4 mg L^{-1}), $Na_2MoO_4{\cdot}2H_20$ (0.1 mg L^{-1}). Once the reactors were sealed, nitrogen gas was flushed into the headspace to avoid traces of oxygen. Batch tests were incubated at 35 ± 1 °C during 273 days. Because biological tests are known to be very sensitive to low inoculation ratios (Abbassi-Guendouz et al., 2012), three replicates were carried out for each condition. In order to check the methanogenic activity of the inoculum, a control batch reactor was realized with ethanol as a substrate. Even if endogenous production could be neglected in high *S*/*X* ratio conditions, two blanks were carried