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Kinetic study of dry anaerobic co-digestion of food waste and cardboard for methane production

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ABSTRACT

Dry anaerobic digestion is a promising option for food waste treatment and valorization. However, accumulation of ammonia and volatile fatty acids often occurs, leading to inefficient processes and digestion failure. Co-digestion with cardboard may be a solution to overcome this problem. The effect of the initial substrate to inoculum ratio (0.25 to 1 gVS·g VS^{-1}) and the initial total solids contents (20–30%) on the kinetics and performance of dry food waste mono-digestion and co-digestion with cardboard was investigated in batch tests. All the conditions produced methane efficiently (71–93% of the biochemical methane potential). However, due to lack of methanogenic activity, volatile fatty acids accumulated at the beginning of the digestion and lag phases in the methane production were observed. At increasing substrate to inoculum ratios, the initial acid accumulation was more pronounced and lower cumulative methane yields were obtained. Higher amounts of soluble organic matter remained undegraded at higher substrate loads. Although causing slightly longer lag phases, high initial total solids contents did not jeopardize the methane yields. Cardboard addition reduced acid accumulation and the decline in the yields at increasing substrate loads. However, cardboard addition also caused higher concentrations of propionic acid, which appeared as the most last acid to be degraded. Nevertheless, dry co-digestion of food waste and cardboard in urban areas is demonstrated as an interesting feasible valorization option.

1. Introduction

The treatment and valorization of food waste (FW) is currently a global issue that needs to be addressed urgently. While traditional methods for FW treatment (*i.e.* landfilling and incineration) are associated with several environmental issues and increasing costs, anaerobic digestion (AD) appears as an effective environmental-friendly industrial process that allows at the same time valorization of the waste into biogas and digestate. From an industrial point of view, AD at high total solid (TS) contents and high loadings is particularly interesting due to the higher associated volumetric biogas production rates (Karthikeyan and Visvanathan, 2013). However, when digesting highly biodegradable substrates rich in nitrogen such as FW, accumulation of volatile fatty acids (VFAs) and free ammonia nitrogen (FAN) usually occurs (Banks et al., 2012, 2008; Capson-Tojo et al., 2016; Zhang et al., 2012a), limiting the loading capacity of the system. This excessive acidification of the digesters may eventually cause a drop of the pH, leading to failure of the digestion process with low methane yields and high chemical oxygen demand (COD) concentrations in the digestates (Capson-Tojo et al., 2016).

Different alternatives have been developed recently to avoid VFA accumulation when digesting FW (Capson-Tojo et al., 2016), such as supplementation of trace elements (Zhang et al., 2012b), addition of zero-valent iron (Kong et al., 2016) or co-digestion (Mata-Alvarez et al., 2011). Between those, co-digestion (*i.e.* simultaneous digestion of two or more substrates) appears as an efficient low-cost option that can be used to avoid accumulation of

Abbreviations: 3D-EEM, 3 Dimension Excitation Emission Matrix Fluorescence Spectroscopy; AD, anaerobic digestion; BMP, Biomethane Chemical Potential; CB, cardboard; COD, chemical oxygen demand; EPS, extra polymeric substances; FAN, free ammonia nitrogen; FW, food waste; IC, inorganic carbon; L, lag phase; M_{max}, final methane yield; OTU, operational taxonomic unit; PCR, Polymerase Chain Reaction; qPCR, Quantitative Polymerase Chain Reaction; R, maximum methane production rate; rRNA, Ribosomal Ribonucleic Acid; S/X, substrate to inoculum ratio; sCOD, soluble chemical oxygen demand; SMPs, soluble metabolic products; TC, total carbon; TKN, total Kjeldahl nitrogen; TOC, total organic carbon; TS, total solids; VFAs, volatile fatty acids; VS, volatile solids.

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VFAs. Co-digestion may improve the process by diluting inhibitory compounds, by balancing the C/N ratio and the concentrations of nutrients, by adjusting the moisture content or by increasing the buffering capacity (Mata-Alvarez et al., 2011). Several co-substrates, such as landfill leachate (Liao et al., 2014), paper waste (Kim and Oh, 2011), sewage sludge (Dai et al., 2013), piggery wastewater (Zhang et al., 2011), rice husks (Haider et al., 2015) or green waste (Kumar et al., 2010), have been effectively applied for stabilization of FW AD. Among these options, paper/cardboard waste (CB) can be a suitable co-substrate for FW dry AD, since it has a high C/N ratio, a high TS content and because of its low biodegradability. Furthermore, FW and CB are the two main organic solid waste streams in urban areas (*i.e.,* CB representing up to 35% of the municipal waste), which facilitates their centralized co-digestion (Hogg et al., 2002; Kim and Oh, 2011; Zhang et al., 2012a).

Besides the potential of this alternative, few studies have been carried out to optimize FW and CB dry co-digestion. At high TS contents (30–50%) Kim and Oh (2011) used paper waste to adjust the C/N ratio of FW, with a co-digestion ratio of 7:1 g TS FW:g TS CB. They achieved stable methane production (with yields up to 250 ml CH_4·g COD^{-1}) without significant VFA accumulation at OLRs up to 10 g TS·l^{-1} d^{-1}. Moreover, Asato et al. (2016) co-digested FW and CB under wet conditions (TS in the inoculum lower than 10%) at different co-digestion proportions and substrate loadings. Their results showed that mixtures with $\geq$75% of CB avoided failure of methanogenesis (occurring at concentrations of FW $\geq$18.75 g COD l^{-1}), suggesting that CB addition helped the process operation. In a recent paper at TS contents between 20 and 35%, Capson-Tojo et al. (2017) concluded that the substrate to inoculum ratio (S/X) and the structure of the microbial community in the inoculum were crucial for an efficient AD process. With an S/X of 0.25 g VS·g VS^{-1} methane yields ranging from 307 to 409 ml CH_4·g VS^{-1} were obtained, depending on the FW concentration and the co-digestion ratio. However, to our knowledge there is no study aiming at understanding the influence of the substrate loading and/or the TS content on the dynamics of VFA production/consumption and the methane yields during dry anaerobic batch co-digestion of FW and CB. As both parameters are critical to assess the feasibility of the AD process and to optimize its performance, their study is essential. Moreover, studying the AD kinetics at dry conditions may potentially lead to a deeper understanding of the process.

Accordingly, the objective of this study was to evaluate the influence of the initial organic load (*i.e.* S/X ratio in batch systems) and the initial TS content on the performance of dry FW mono-digestion and FW co-digestion with CB in batch systems. At the same time, the effect of CB addition itself was also assessed. For the first time under dry conditions using batch reactors, particular attention was paid to the dynamics of VFA production/consumption and methane generation. In addition, the influence of the aforementioned parameters on the final methane yields was assessed. Aiming to elucidate the fate of the organic matter not being transformed into methane, the characteristics of the residual soluble organic matter remaining in the digestates were also studied, as well as the structure of the final microbial communities.

2. Materials and methods

2.1. Substrate and inoculum

A model FW was synthetized according to the VALORGAS report (VALORGAS, 2010) as in Capson-Tojo et al. (2017). Compact cardboard (branded "Cartonnages Michel"; shredded to 1 mm) with a density of 1.42 kg·m^{-3} was used as co-substrate. The characteristics of these substrates are shown in Table 1.

Table 1
Main characteristics of the substrates (Capson-Tojo et al., 2017).

Parameter/element	Unit	Food waste	Cardboard
TS	% (w. b.)	21.6 ± 0.7	92.7 ± 3.7
VS	% TS	96.2 ± 0.1	77.5 ± 0.2
pH	Unit pH	5.60	7.10
COD	g COD·g TS^{-1}	1.37 ± 0.05	1.19 ± 0.05
BMP	ml CH_4·g VS^{-1}	498 ± 42	250 ± 3
NH_4	g·kg TS^{-1}	0.051	0.002
TKN	g·kg TS^{-1}	27.08 ± 1.64	2.00 ± 0.02
TC	g·kg TS^{-1}	442 ± 7	366 ± 6
C/N	g·g^{-1}	16.3	183
Carbohydrates	g·kg TS^{-1}	687 ± 15	958 ± 5
Proteins	g·kg TS^{-1}	169 ± 10	0
Lipids	g·kg TS^{-1}	72.3 ± 1.5	0

*TS stands for total solids; VS for volatile solids; COD for chemical oxygen demand; BMP for biochemical methane potential; TKN for total Kjeldahl nitrogen; TC for total carbon.

The inoculum was collected from an industrial plant treating a mixture of different organic streams. As the concentrations of TAN in the sludge were elevated (5.04 g TAN·l^{-1}; pH 8.1; 336 mg FAN·l^{-1}), it was assumed that the microbial population were already adapted to high TAN/FAN concentrations (like those found during FW AD). The sludge had a TS content of 5.81 ± 0.02%, with 59.13 ± 0.08% corresponding to volatile solids (VS).

2.2. Dry batch anaerobic co-digestion

When compared to continuous systems, batch reactors facilitate testing different conditions simultaneously much more easily and therefore they are particularly convenient for AD assays at different TS contents and inoculation ratios. To evaluate the influence of the S/X (*i.e.*, substrate loading), the initial TS content and the substrate composition, eight different conditions were defined (Table 2).

The first three reactors (FW-20-0.25, FW-20-0.50, FW-20-100) consisted in mono-digestion batch reactors fed with FW at a given TS content (20%) and different S/X (0.25, 0.50, 1.0 g VS·g VS^{-1}, respectively). To evaluate the effect of co-digestion, the same conditions were applied in reactors (FW+CB)-20-0.25 to (FW+CB)-20-1.00, but feeding a mixture of FW and CB. The co-digestion ratio was fixed at 7.48 g FW·g CB^{-1} (raw weights), obtaining a substrate with an initial TS content of 30%. Finally, two other conditions, FW-20–0.25 and (FW + CB)-30–0.25, were applied to test the influence of the initial TS content: an S/X of 0.25 g VS·g VS^{-1} was applied, with an initial TS content of 30%. To adjust the initial TS content in the reactors, dried stabilized compost was added into all the vessels. To correct the endogenous contribution to the biogas from the inoculum and the compost, four different blanks (one per S/X and TS content to consider the influence of the added compost) were carried out.

All reactors had a total volume of 2.5 l and were incubated at 35 °C. In order to have similar operating volumes in the reactors (0.6–0.7 l), different initial amounts of FW were added into the vessels. Afterwards, the respective amounts of CB, inoculum and compost (according to Table 2) were supplemented and the mixture was thoroughly homogenized. The headspace volume was determined by measuring the difference in pressure after addition of a known volume of gas and applying the ideal gas law. The reactors were sealed and flushed with nitrogen to ensure anaerobic conditions. The reactors used were specifically designed to allow sampling of the dry digesting medium during the AD process without disturbing the gas in the head space (Motte et al., 2015). These reactors were equipped with a "ball" valve on their tops, which allowed introducing a metallic sampler. During regular operation,

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