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Robust assessment of both biochemical methane potential and degradation kinetics of solid residues in successive batches

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ABSTRACT

The well-known batch assay test is used worldwide to determine the biochemical methane potential (BMP) of solid substrates in a single batch but its use to estimate the degradation kinetics may lead to underestimations. To overcome this problem, a different approach was carried out to characterize simultaneously both BMP of solid substrates and their degradation kinetics in successive batches, i.e. after an acclimation period. In a second step, a simple model was developed based on the methane production curve in batch mode for dividing the organic matter of the substrate into three sub-fractions according to their degradation rates (rapid, moderate and slow). The protocol developed was applied to 50 different substrates and a database was built. This database includes: the overall BMP (mL CH_4/g VS) and the degradation kinetics for each substrate, i.e. the global specific organic degradation rate (g VS/g VSS.d) along with the 3 sub-fractions and their specific degradation rates. The comparison with the BMP from the literature did not highlight significant difference with the BMP measured in this study. Furthermore, the degradation rates seem to be specific characteristics for each substrate and no clear correlation was found between the degradation kinetics and the kind of substrates. The information available in the database will be useful for the design and operation of anaerobic digesters: Optimization of the mix of co-substrates, choice of the applied OLR, simulation of methane production and of the rate of substrate degradation.

1. Introduction

Organic solids are present in very large quantities in by-products or waste originating from agriculture, the agri-food industry, fruit and vegetable markets, etc. (Forster-Carneiro et al., 2008; Lastella et al., 2002). According to Charles et al. (2009), the production of organic solid waste is rising continuously and is predicted to reach 3 billion tons per year worldwide in 2025. Consequently, implementing better management practices has become indispensable to prevent uncontrolled emissions, minimize risk to human health, reduce burdens on the environment and maintain an overall balance in ecosystems (Khalid et al., 2011).

A possible option for treating various organic wastewater and solid waste is anaerobic digestion (AD). AD is a biochemical process used for the treatment of organic substrates both liquid, such as sewage and industrial effluents, and solid (Bouallagui et al., 2009) including animal manure, energy crops, agricultural residues and food waste (Raposo et al., 2012). AD has proven to be a reliable technology in full-scale operations which can be economically feasible and is considered to be environmentally friendly thanks to net energy recovery, low sludge production and low discharge of volatile compounds into the atmosphere (Carucci et al., 2005). As a result, the application of AD technology is increasing worldwide (Angelidaki et al., 2009).

The AD process can be carried out in batch, fed-batch or continuous mode. Batch tests are usually used at laboratory scale to

Abbreviations: AD, anaerobic digestion; AnSBR, anaerobic sequencing batch reactor; BMP, biochemical methane potential; BPR, Biogas Production Rates; COD, Chemical Oxygen Demand; FVW, fruit and vegetable wastes; ISR, Inoculum to Substrate Ratio; IWA, the International Water Association; MPR, methane production rates; OLR, Organic Loading Rate; TA, Total Alkalinity; TS, total solid; VFA, Volatile Fatty Acid; VS, volatile solid; VSS, volatile suspended solids; WAS, Waste Activated Sludge.

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assess anaerobic biodegradability of organic matter, biochemical methane potential (BMP), inoculum activity and inhibition (Raposo et al., 2012). Several protocols have been developed to measure the BMP of organic substrates in batch mode (Adani et al., 2001; Angelidaki et al., 2009; Angelidaki and Sanders, 2004; Chynoweth et al., 1993; Eleazer et al., 1997; Hansen et al., 2004; Owen et al., 1979). Basically, a batch operation consists in incubating a small amount of waste with an anaerobic inoculum and then measuring methane production (Hansen et al., 2004). The main differences between the existing protocols are due, firstly, to the variety of equipment used, which varies as to headspace pressure, liquid and headspace volumes, pH, and the volume measurement system; and, secondly, to different environmental conditions, including the source of inoculum and the substrate to inoculum ratio, which can differ from one test to another, often making the comparison of results quite difficult. Generally, these protocols are only used to assess BMPs but some authors have also used batch tests to evaluate degradation kinetics (Vavilin et al., 2008). Vavilin et al. (2006) reported that ideally the waste should be divided into two fractions with different kinetic rates -readily degradable and recalcitrant- for modelling the degradation of municipal solid waste in landfill conditions. Girault et al. (2012) developed an approach using experimental degradation kinetics, especially from batch experiments, to divide the organic matter of the substrate into different fractions with different degradation kinetics. The same "anaerobic respirometry" method was used by Yasui et al. (2008) to determine the kinetics of different substrates. The approach was to identify various COD fractions of the substrate and the degradation kinetics by using the interpretation of the evolution of the methane production rates (MPR) overtime. Compared to a fractionation based on physical-chemical characteristics, the main advantage of the MPR method is that the input variables obtained take into account the rate-limiting step, i.e. hydrolysis, which drive the substrate degradation kinetics (Girault et al., 2012). In the study of Mottet et al. (2013), batch assays were used to develop the ADM1 model and calibrate the kinetic parameters and biomass concentrations using methane production curves. Waste activated sludge particulate COD was divided into rapidly- and slowly- biodegradable fractions. The fractionation gave a strong calibration of ADM1 and was then used for modelling continuous reactor at full scale. In an interlaboratory study (Raposo et al., 2011), first-order degradation constants were determined using the methane production values from BMP curves found in the experiments carried out. In this work, the influence of several experimental conditions (flask volume, temperature, stirring, mineral medium additions, inoculum concentration, etc...) on degradation rates was investigated and the results showed that the rates differed significantly depending on the experimental conditions.

However, the kinetics were generally assessed from a single batch which, as shown by Martinez-Sosa et al. (2009), might have led to underestimation. This author studied the treatment of fatty solid waste from the meat industry using an anaerobic sequencing batch reactor (AnSBR) seeded with a high sludge concentration (15.6 g VSS of sludge/L) taken from a reactor treating distillery vinasse. The fed-batch mode was employed during the start-up period; 7 batches were processed in 25 days. The longest batch was the first and its biogas production profile was totally different from the subsequent batches. These results clearly show that a rapid acclimation occurred during the start-up period and that the kinetics in the first batch were very different from these of the following batches. Using only the results of the first batch to assess the degradation kinetics might, without any replication, lead to a marked underestimation.

To avoid the problems met during solid waste characterization as pointed out in the previous section, a new protocol including an acclimation phase was developed at laboratory scale. It was developed for the purpose of evaluating both the BMP and the degradation kinetics of solid waste. The tests were performed in 6 L anaerobic reactors operated with successive batches in order to promote an acclimation phase between the sludge and the substrate studied. 50 particulate substrates were characterized using the newly-developed protocol. A further fractionation of the solid substrates, into 3 sub-fractions with different kinetics parameters (rapidly-, moderately- and slowly-biodegradable sub-fractions), was also proposed using the methane production curves resulting from batch experiments. The aim was to construct a database for the different substrates studied which would include the BMP values, the overall kinetics (specific organic degradation rate) and the new fractionation (3 sub-fractions with 3 different degradation kinetics). This information can be used for the design, the optimization of the operation and the modelling of anaerobic co-digesters.

2. Materials and methods

2.1. Substrates

50 different solid substrates, divided into 9 main categories, were characterized: (i) Fruit and vegetables: peach, grape, apple, orange, mango, banana, pineapple, green cabbage, potato, carrot, lettuce, tomato (2 varieties), cauliflower, zucchini, chayote; (ii) Other plant products: grass cuttings, napier grass, wheat straw; (iii) Vegetable by-products from agri-food processes: grape marc, coconut meal, sunflower meal, rape meal, beet pulp; (iv) Cooked cereal products: pasta, rice, French bread; (v) Animal products: ground beef, coalfish, pork fat; (vi) Animal manure: cattle (3 batches), chicken, pig; (vii) Products and by-products from the refining of vegetable oil: used winterization earth, 2 tank sediments from the storage of rape and sunflower oils, 2 soapstocks from the refining of sunflower and rape seed oils, 2 deodorizing condensates from sunflower and palm oils, 2 skimmings of aeroflotation of the effluents, 1 gum from physical refining, 1 pure sunflower oil; (viii) Sludge from domestic wastewater treatment: one from an aerobic lagoon, one from a WTP operated at a medium organic loading rate (mix of primary and secondary sludge); (ix) Miscellaneous products: dry food pellets for guinea pigs and micro-algae.

Each solid residue was characterized by measuring its concentration of total solids (TS) and volatile solids (VS). Before use, all the substrates were crushed, mixed and stored at −20 °C.

2.2. Reactors

The experiments were carried out in double-walled glass reactors of 6 L effective volume, maintained at 35 °C by a regulated water bath. Mixing in the reactor was done by magnetic stirring. Biogas production was measured on-line using a MGC-1 V3.1 PMMA Milligascounter flow meter (Ritter) fitted with digital output. The "Odin-Silex" software developed by INRIA and INRA was used to log gas output.

2.3. Inoculum

The reactors were seeded, at a volatile suspended solids concentration (VSS) in the range 12–14 g VSS/L, with anaerobic granules taken from an industrial-scale anaerobic UASB reactor treating the effluents from a sugar refinery. 800 g of drained granules were added to each reactor. Tap water with 2.5 g/L of $NaHCO_3$ was added to 6 L. The reactor was maintained under agitation for 48 h to break up the granules. After seeding but prior to the addition of any

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