Bioresource Technology 102 (2011) 5653–5658

Contents lists available at ScienceDirect

Bioresource Technology

journal homepage: www.elsevier.com/locate/biortech

Anaerobic hydrolysis and acidification of organic substrates: Determination of anaerobic hydrolytic potential

Rajinikanth Rajagopal, Fabrice Béline *

Cemagref, UR GERE, 17, Avenue de Cucillé, CS 64427, F-35044 Rennes, France
Université Européenne de Bretagne, France

ARTICLE INFO

Article history:
Received 28 November 2010
Received in revised form 15 February 2011
Accepted 17 February 2011
Available online 21 February 2011

Keywords:
Hydrolysis
Acidification
Fermentation time
Inoculum ratio
Volatile fatty acids (VFAs)

ABSTRACT

This study aimed to develop a biochemical-test mainly to evaluate the hydrolytic-potential of different substrates and to apply this test to characterize various organic substrates. Outcome of this study can be used for optimization of the WWTPs through enhancement of N/P removal or anaerobic digestion. Out of four series of batch experiments, the first two tests were conducted to determine the optimal operating conditions (test duration, inoculum-ratio etc.) for the hydrolytic-potential test using secondary and synthetically-prepared sludges. Based on the results (generation of CODs, pH and VFA), test duration was fixed between 6 and 7 d allowing to attain maximum hydrolysis and to avoid methanogenesis. Effect of inoculum-ratios on acid fermentation of sludge was not significantly noticed. Using this methodology, third and fourth tests were performed to characterize various organic substrates namely secondary, pre-treated sludge, pig and two different cattle slurries. VFA production was shown to be substantially dependent on substrates types.

1. Introduction

The biological nutrient removal (BNR) systems are capable of removing significant amount of total phosphorus (TP), total nitrogen (TN), BOD_5 and total suspended solids (TSS). The degree of removal depends on the characteristics of wastewater, operating temperature, many other design and operational features of the facility. BNR processes have been widely studied by several researchers and full-scale applications are present worldwide. The biodegradation characteristics of the available COD level are usually the critical parameter that defines the system efficiency. The magnitude of the N and P removal with biological processes is closely linked to the presence of easily biodegradable carbon source, which is often the limiting factor in the process (Ucisik and Henze, 2004; Chen et al., 2007). An external carbon source can be added in order to improve nutrients removal. Denitrification needs readily biodegradable COD as the carbon source, and volatile fatty acids (VFAs) are required by phosphorous accumulating organisms (PAO) for enhanced biological P removal (Chen et al., 2004). However, the addition of the soluble organic compounds increases both the operational cost and sludge production in the wastewater treatment plants (WWTPs). And hence, the use of a carbon source produced from WWTP by-products or from locally available organic wastes instead of commercial organic materials would decrease the operational cost.

On the other hand, anaerobic digestion can be used as a treatment option for sludge from WWTPs in order to transform organic matter into biogas and reduce the amount of sludge. Anaerobic digestion thus optimizes WWTP costs, its environmental footprint and is considered as a major and essential part of a modern WWTP (Appels et al., 2008). Also, anaerobic digestion is a widely used method for the treatment of sewage sludge (Dereix et al., 2006; Morgan-Sagastume et al., 2011). The optimization of anaerobic digestion process strongly depends on the increase of the hydrolysis efficiency, since the organic matter of sewage sludge exists in particulate form and hydrolysis is the rate-limiting step of the whole process (Miron et al., 2000; Appels et al., 2008; Lv et al., 2010). Therefore, attempts to improve the overall process have to focus on the hydrolysis/acidification reaction. One way for the optimization of such a process is the development of a two-phase digestion system that separates two limiting steps: hydrolysis and methanogenesis. The fundamental concept of two-phase digestion is to optimize the conditions for the hydrolytic–acidogenic group of bacteria as well as for the acetogenic–methanogenic group, leading to the production of the most suitable acid metabolites for the methanogens (Mahmoud et al., 2004; Appels et al., 2008) and subsequently it may allow a reduction in total reactor volume.

According to this context, the hydrolysis/acidification step of organic substrates could improve WWTP management by enhancing N/P removal or anaerobic digestion of sludge.

* Corresponding author at: Cemagref, UR GERE, 17, Avenue de Cucillé, CS 64427, F-35044 Rennes, France. Tel.: +33 (2) 23 48 21 21; fax: +33 (2) 23 48 21 15.
E-mail address: fabrice.beline@cemagref.fr (F. Béline).

0960-8524/$ - see front matter © 2011 Elsevier Ltd. All rights reserved.
doi:10.1016/j.biortech.2011.02.068

Some research works were conducted for a better understanding of the hydrolysis process, in particular, hydrolysis of waste activated sludge. When the particulate organic matters contained in activated sludge were not properly solubilized, only 30–50% of the total COD (CODt) or volatile solids (VS) in waste activated sludge were reportedly biodegraded within 30 d (Kim et al., 2007; Ucisik and Henze, 2008). Thus, to enhance the hydrolysis rate and maximize VFAs production, several efforts have been made to improve the efficiency of anaerobic digestion, such as by thermal (Morgan-Sagastume et al., 2011), thermo-chemical (Vlyssides and Karlis, 2004), mechanical (Dereix et al., 2006), ultrasonic (Liu et al., 2009) and microbial enzyme technology (Burgess and Pletschke, 2008). Operational parameters such as solids retention time (SRT), pH, temperature, waste-mixing ratio and sludge thickness were also investigated (Chen et al., 2007; Ucisik and Henze, 2008; Alkaya and Demirer, 2011). Chen et al. (2007) investigated the effects of pH from 4.0 to 11.0, on waste activated sludge hydrolysis and acidification. Mahmoud et al. (2004) investigated the effect of SRT and process temperature on the hydrolysis, acidification and methanogenesis of primary sludge in continuously stirred tank reactors (CSTRs). In addition, Miron et al. (2000) studied the effect of SRT between 3 and 15 days, on hydrolysis, acidification and methanogenesis of domestic sewage in CSTR. Except for the waste activated sludge, very limited studies were performed on the hydrolysis of organic substrates that could be used for the optimization of WWTP.

The aims of this work were (1) to develop a biochemical test to evaluate the hydrolytic potential of organic substrates and (2) to apply this test to characterize various organic wastes which can be used for the optimization of the WWTP. For that, firstly, the operating conditions for the hydrolysis process were examined (duration of the test, inoculum ratio, etc.) using secondary sludge and synthetically prepared substrate. Secondly, several substrates/sludges including secondary sludge from WWTPs, pre-treated sludge, pig slurry and cattle manures were characterized. The purpose of this study was therefore to examine the efficiency of hydrolysis and acidification with relatively short fermentation times on various sludge types as an internal carbon source production method for BNR processes or as an improvement of anaerobic digestion process.

2. Methods

2.1. Overview

At first, two series of batch experiments were conducted to determine a methodology in terms of optimal operating conditions for the hydrolytic potential test. In the first trial, secondary sludge was used as a substrate to determine the duration of the test. For this, inoculum was added to about 5% by working volume of the reactor, corresponding to about 30% in terms of carbon. In the second trial, secondary sludge and synthetically prepared substrate were used and the inoculum addition varied from 0% to 10% in terms of carbon.

Applying the optimal conditions, two additional trials were performed to characterize various organic substrates (*i.e.* secondary sludge, pre-treated sludge, pig slurry and cattle manures) in terms of hydrolytic potential.

2.2. Feed sources

Secondary sludge used in this study was obtained from the aeration tank of municipal WWTP located in Mordelles (France). Synthetic substrate was prepared by using cellulose (5 g COD/L) and casein (5 g COD/L). Secondary sludge after ultrasonic pretreatment was also obtained from municipal WWTP located in Mordelles. Pig slurry was collected from mechanically separated effluent using a screw-press auger in the treatment plant of a commercial pig breeding unit in Brittany (France). Cattle slurry was taken from two farms located in Brittany. Tap water was used for sludge dilutions to bring down the COD_t in each substrate to around 9.5 ± 1.2 g/L. Inoculum used in this study was collected from a methanogenic digester treating piggery wastewater. A summary of important characteristics of different substrates and inoculum used in this study are presented in Table 1.

2.3. Experimental set-up

Bench-scale experiments were conducted by using identical batch reactors, serum bottles of each 300 mL capacity with working liquid volume of 150 mL. Each reactor was mechanically stirred at 80 rpm (rotation per minute) and pH was not controlled in the sludge. These reactors were incubated at 37 ± 1 °C and in the beginning of each batch experiment, nitrogen gas was sparged to create anaerobic conditions in all the reactors. No other pretreatment or control techniques were followed to prevent methanogenesis during the hydrolysis and acidification process.

2.4. Analytic methods

Total solids (TS), total volatile solids (TVS), suspended solids (SS), volatile suspended solids (VSS), total and soluble COD, and total Kjeldahl nitrogen (TKN) were analyzed according to Standard Methods (APHA, 1992). Ammonium (NH_4^+) was analyzed on total wastewater by steam distillation using MgO followed by back titration of the boric acid distillates using sulfuric acid (0.1 M). Total carbon (TC) was determined by calcination at 1000 °C using a COT meter (Skalar® Technology). Volatile fatty acids (VFA) namely acetic (C2), propionic (C3), butyric (C4), iso-butyric (iC4), valeric (C5) and iso-valeric (iC5) were determined by high performance liquid chromatography (Waters® Technology) according to Peu et al. (2004). Gas compositions mainly methane and carbon dioxide was measured using gas chromatograph (Varian CP 4900, Chromatography Systems Middelburg, The Netherlands).

3. Results and discussions

3.1. Determination of methodology

Two series of batch experiments were conducted to develop a methodology for determining the fermentation potential of different substrates.

In the first trial, 12 identical batch reactors, serum bottles, were used as described in Section 2.3. All the reactors were fed with secondary sludge obtained from a WWTP, as substrate. Inoculum was added to about 5% by working volume of each reactor (30% in terms of carbon). The hydrolytic potential was determined in duplicates on day 0, 2, 4, 6, 8, 10 and 13. In this study, the hydrolytic potential of secondary sludge in terms of duration of the test (optimal fermentation time) and inoculum to substrate ratio was assessed.

3.1.1. Duration of the test

The primary goal of hydrolysis and acid fermentation is the solubilization of particulate organic fraction in the sludge during the treatment process. Sludge hydrolysis can be expressed by the changes of CODs concentrations (Chen et al., 2007). Fig. 1 gives the effect of fermentation time on CODs production and pH. It was observed that the CODs concentrations increased up to day 4 to about 360% compared to the initial value and then decreased gradually with time (Fig. 1). On the fourth day of fermentation, CODs concentration was 2490 mg/L, while it was 690 mg/L on