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Seeking to enhance the bioenergy of municipal sludge: Effect of alkali pre-treatment and soluble organic matter supplementation

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ABSTRACT

The aim of this research is to enhance the mesophilic anaerobic digestion of municipal sludge from Cadiz-San Fernando (Spain) wastewater treatment plant at 20 days hydraulic retention time (HRT). Two different strategies were tested to improve the process: co-digestion with the addition of soluble organic matter (1% v/v); and alkali sludge pre-treatment (NaOH) prior to co-digestion with glycerine (1% v/v). Methane production (MP) was substantially enhanced (from 0.36 ± 0.09 L CH_4 l/d to 0.85 ± 0.16 L CH_4 l/d), as was specific methane production (SMP) (from 0.20 ± 0.05 L CH_4/g VS to 0.49 ± 0.09 L CH_4/g VS) when glycerine was added. The addition of glycerine does not seem to affect sludge stability, the quality of the effluent in terms of pH and organic matter content, i.e. volatile fatty acids (VFA), soluble organic matter and total volatile solid, or process stability (VFA/Alkalinity ratio < 0.4). Alkali pre-treatment prior to co-digestion resulted in a high increase in soluble organic loading rates (more than 20%) and acidification yield (more than 50%). At 20 days HRT, however, it led to overload of the system and total destabilization of the mesophilic anaerobic co-digestion of sewage sludge and glycerine.

1. Introduction

Sludge treatment accounts for over 50% of the operating costs of wastewater treatment plants (WWTP) (Razaviarani and Buchanan, 2015; Rivero et al., 2014; Zahedi et al., 2017a). Anaerobic digestion (AD) is an attractive treatment strategy for municipal sludge and is of great benefit from an environmental point of view as this technology allow the production of bioenergy and fertilizer (Appels et al., 2011; Bolzonella et al., 2005; Di Maria et al., 2016, 2014; Forster-Carneiro et al., 2010; Gómez et al., 2006; Liao et al., 2016; Peces et al., 2016; Sosnowski et al., 2003; Wu et al., 2016; Zahedi et al., 2016a). It has been well demonstrated that mesophilic AD of municipal sludge from Cadiz allows the obtaining of Class B biosolids, i.e. an effluent with a density of faecal coliforms below 2×10^6 colonies g/1 total solids (Forster-Carneiro et al., 2010). Unlike Class A biosolids, which are essentially pathogen free and authorized for all uses, Class B biosolids may contain some pathogens and can be employed with a number of restrictions, such as crop harvesting, animal grazing, and public access for a certain period of time. Obtaining Class A biosolids requires an increase in temperature (thermophilic conditions, around 50 °C) (Riau et al., 2010). Numerous research studies have sought to optimize the AD of sludge, including the interesting options of the co-digestion process or sludge pre-treatments (Mata-Alvarez et al., 2011; Wang et al., 2013; Zahedi et al., 2016a), which increase the load of biodegradable organic matter and produce a higher biogas yield. The integrated management of sludge and fruit and vegetable waste by co-digestion and composting has recently been investigated from a life cycle perspective by Di Maria et al. (2016). Their results show that co-digestion enhances methane production. Recent studies have been also demonstrated the efficacy of anaerobic co-digestion of municipal sludge or solid waste together with readily biodegradable organic substances, such as glycerol, a major by-product of biodiesel production (Fountoulakis et al., 2010; Fountoulakis and Manios, 2009; Razaviarani et al., 2013; Razaviarani and Buchanan, 2015; Rivero et al., 2014; Zahedi et al., 2017c, 2016b). Studies on co-digestion have shown the optimal glycerine supplementation in the co-digestion of municipal sludge to be 1% (v/v) at 20 days hydraulic retention time (HRT) (Fountoulakis et al., 2010; Razaviarani et al., 2013; Razaviarani and Buchanan, 2015). Due to slow sludge fermentation rates (hydrolysis and acidification) and the advantages of the anaerobic digestion (AD) process, extensive research has been carried out on the optimization of pre-treated sludge to improve hydrolysis, the generation of volatile fatty acids (VFA) and biogas production (Carrère et al., 2010; Ennouri et al., 2016;

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Lee et al., 2014; Li et al., 2016b; Liu et al., 2012; Raynal et al., 1998). These pre-treatments seek to destroy cells and/or extracellular polymeric substances (EPS), with the subsequent release of intracellular and/or extracellular constituents to the aqueous phase (Carrère et al., 2010; Gianico et al., 2013; Wang et al., 2013). These released constituents are more easily biodegraded during anaerobic digestion, thereby enhancing methane production.

Most novel studies focus on combined methods, i.e. pre-treatment of substrates using different methods such as mechanical, chemical, thermal and/or others to increase their availability to microbial bioconversion (Dahunsi et al., 2016a).

One the most efficient, simple pre-treatments for municipal sludge is the alkali (NaOH) pre-treatment (Dahunsi et al., 2016a; Li et al., 2016a; Zhang et al., 2015). For example, Li et al. (2016a) reported that methane production in AD increased by 18% after microwave-ultrasonic pre-treatment or by 42% after pre-treating activated sludge at 175 °C for 60 min or up to 71% after pre-treating activated sludge at 120 °C with the addition of 20 mg NaOH.

Taking into account the above, glycerine supplementation (1% v/v) and alkali pre-treatment in sludge was applied in the present research to improve the methane yield, achieving enhancements of between 71% and 125%. The experimental protocol was designed to examine the effect of two strategies for enhancing AD of the municipal sludge from Cadiz-San Fernando (Spain) WWTP. One was co-digestion of municipal solid sludge with glycerine (1% v/v), while the other was alkali sludge pre-treatment (NaOH) prior to co-digestion of municipal solid sludge with glycerine. It should be noted that this study was carried out at the most widely-employed hydraulic retention time (HRT) in the mesophilic AD of municipal sludge at most WWTP. Hence, the results of this paper provide useful information to obtain in-depth knowledge of strategies to enhance bioenergy production at WWTP.

To assess whether these strategies might be an interesting option in an actual municipal WWTP, different parameters such as the increase in SCOD (%), acidification yield (%), process stability, quality of the digested sludge and biogas production were studied.

2. Materials and methods

2.1. Substrates, alkali pre-treatment and inoculum

Experimental work was carried out using sewage sludge samples (mixed primary sludge (30%) and activated sludge (70%)) from Cadiz-San Fernando WWTP. This plant is located in Cadiz-Spain and handles over 50,000 m^3 of wastewater daily. All the sludge samples were characterized on reception at the laboratory and kept under refrigeration at 4 °C before being used for the experiments so as to prevent biodegradation. The pH, volatile solids (VS) and soluble chemical oxygen demand (SCOD) concentrations in the municipal sludge were 6.8 ± 0.1, 35 ± 2 g VS/kg and 10 ± 1 mg SCOD/l, respectively.

For the co-digestion studies (with or without alkali pre-treatment), this sludge was mixed with 1% v/v glycerol supplied by the Panreac company, which constituted the reactor feed. According to Fountoulakis et al. (2010), the most appropriate concentration of glycerol in co-digestion with sewage sludge in anaerobic processes is 1%.

For the alkali pre-treatment, the pH of the sludge sample was adjusted to 12.0 ± 0.1, followed by stabilization for 5 min under stirring with 6 mol/L sodium hydroxide in line with Xiao and Liu (2009).

Regarding the inoculum, this was collected from the mesophilic anaerobic digester (hydraulic retention time (HRT)=20 d) located at the same WWTP. The pH, total solids (TS) and volatile solids (VS) were 7.5 ± 0.2; 32.0 ± 2.0 g TS/kg and 18.0 ± 0.2 g VS/kg, respectively. The inoculum to substrate ratio (ISR) in this reactor (g VS/g VS) was around 10.

2.2. Experimental equipment and operating conditions

Three laboratory-scale reactors operating in a laboratory-scale semi-continuous stirred tank reactor (CSTR) at the laboratory scale were employed in these studies. The reactors had a working volume of 5 L, without biomass recycling, and operated at the same HRT and Solids Retention Time (SRT), (20 days). Mesophilic conditions (35 °C) were maintained by circulating water through the jacket from thermostatic water baths. PRECISTERM 6000142/6000389 (SELECTA S.A.) baths, with a maximum capacity of 7 L water, were used for this purpose, Mixing was maintained constant in the three reactors using mechanical stirrers (23 rpm) and each reactor was equipped with a biogas outlet and a feed inlet. The gas volume produced in the reactor was emptied into Tedlar gas bags (40 L).

The three reactors employed were:

CR: fed with sewage sludge.

GR: fed with sewage sludge and glycerine (1%).AGR: fed with alkali pre-treated sewage sludge and glycerine (1%).

All the reactors operated at 20 days HRT (ISR around 10) and were fed once a day (semi-continuous) without the addition of nutrients or pH correction. The volatile solids organic loading rates (OLR) was 1.75 g VS/l/d.

The overall duration of the experiment for each reactor was 60 d, except in the AGR, where the overall duration was 7 days because destabilization was observed.

2.3. Analytical methods

The following variables were analysed to characterise and monitor the process effluents: pH, alkalinity, volatile fatty acids (VFA), soluble chemical oxygen demand (SCOD) and volatile solids (VS). These analyses were conducted in accordance with standard methods (APHA, 1995) and Zahedi et al. (2017c). The gas volume produced in the reactors was measured directly using a high-precision flow gas meter: Ritter TG-01 drum-type gas meter – (wet-type).

VFA were determined by gas chromatography using a gas chromatograph (Shimadzu GC-2010) equipped with a flame ionization detector (FID) and a capillary column filled with Nukol. The gas volume produced in the reactor was measured directly using a high-precision flow gas meter: Ritter TG-01 drum-type gas meter (wet-type). The composition of the biogas was determined by gas chromatography separation (SHIMADZU GC- 2010). H_2, CH_4, CO_2, O_2 and N_2 were analysed by means of a thermal conductivity detector (TCD) using a Supelco Carboxen 1010 Plot column. Commercial mixtures of H_2, CH_4, CO_2, O_2, N_2 and H_2S (Abelló Linde S. A.) were used to calibrate the system.

Gas volume and composition were measured daily, as was the pH of the effluent. VS, COD, alkalinity and VFA were analysed approximately two/three times a week.

2.4. Parameters used to determine the effect of the different strategies on the reactor feeds

Changes in the soluble OLR (SOLR) and acidification yield were the parameters used to analyse the effect of the different strategies on the feed.

Acidification yield was calculated via the soluble COD of VFA (S_{TVFA}) through the following equation (De La Rubia et al., 2009; Zahedi et al., 2014, 2013:

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