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Effect of ultrasonic pretreatment on the semicontinuous anaerobic digestion of waste activated sludge with increasing loading rates

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ABSTRACT

Anaerobic digestion (AD) is the most widely used method to stabilize and recover energy from waste activated sludge (WAS). However, AD of sludge results in a low biogas yield. Ultrasonic pretreatment (USp) is the most effective sludge disintegration technology and has elicited interest to increase the biodegradability and improve the efficiency of the AD. This study investigated the influence of USp on the semicontinuous AD of WAS as a strategy to increase its performance. During an experiment lasting 90 days, 0.8-L digesters were semi-continuously fed under different conditions: raw sludge and sonicated at 15000, 25000 and 35000 kJ/kg TS (total solids); and an increase of the organic loading rate (OLR) from 1 to 3 kgVS/m^3·d, for 30 days each. The maximum biogas rate (0.218 L/d) was reached at 25000 kJ/kg TS and 3 kgVS/m^3·d. The highest VS removal efficiency (23.7%) was observed at the highest USp and the lowest OLR. The reactors fed with raw sludge had a lower performance compared to the sonicated WAS in terms of VS reduction and biogas production. In contrast, the USp allowed the bioreactors to be operated at a high OLR (3 kgVS/m^3·d) without inhibition. Finally, a kinetic analysis of accumulated biogas was performed.

1. Introduction

Anaerobic digestion (AD) is the most common technology applied to treat waste activated sludge (WAS) (Campo et al., 2017). This biological waste contains organic matter, heavy metals, pathogens and emerging contaminants (pharmaceuticals or drugs) that are not currently covered by existing environmental and water quality regulations (Boix et al., 2016; Zhen et al., 2017). Inadequate treatment, use and disposal of WAS causes severe environmental impacts and risks to human health. The water and wastewater industry are facing unprecedented economic and environmental constraints because of increasingly stringent regulations and the large amounts of WAS produced. Proper management of waste sludge is a big challenge to wastewater treatment industries, because sludge handling and disposal account for up to 60% of the total operating costs of wastewater treatment plants (Uma Rani et al., 2013; Xie et al., 2016).

In light of the challenges described above, AD is an excellent alternative for the successful treatment of WAS to reduce negative environmental and health impacts. However, due to its low biodegradability and complexity, as well as the presence of micro-contaminants and inhibitors of the anaerobic process, the resulting biogas and methane yields and the reduction in organic matter and pathogens is limited. The low biogas yields are due to the complexity of the cell walls of the bacteria that make up the sludge flocs (Zhang et al., 2017). However, mesophilic AD can be improved by adding a pretreatment step (Carrère et al., 2010; Carlsson et al., 2012).

Ultrasonic pretreatment (USp) is the most effective sludge disintegration technology and has been applied in lab, pilot and full-scale systems (Pilli et al., 2011; Appels et al., 2012; Tyagi et al., 2014). USp has been successful because it generates a high level of solubilization, is an easy and simple pretreatment, increases the biogas and methane yields, improves digestate quality, and can be used together with other pretreatments (Gong et al., 2015; Zhao et al., 2015). However, there are strong disagreements regarding the effects of ultrasonic pretreatment studies, especially with respect to the energy applied, their energy expression, the equipment used, the total solids concentration, and the effects on solubilization and anaerobic biodegradability (Tyagi et al., 2014; Le et al., 2015). Most of the research on the effects of pretreatments of the AD of sludge only investigated the process using single stage batch digestion experiments (BMP-tests) (Ruiz-Hernando et al., 2014; Kim et al., 2015; Pilli et al., 2016). Therefore, there is still a lack of information on the behavior of the process in semicontinuous

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systems, particularly with respect to the optimum ultrasonic specific energy (SE), the maximum production of biogas and the maximum organic loading rate (OLR) at which the reactor can be operated.

In the first part of this work, USp was performed with a wide range of specific energies (5000–35000 kJ/kg TS) to evaluate its effects on solubilization, sanitization, and anaerobic biodegradability. The most effective energies were evaluated by BMP tests, with solubilization as a discriminating variable (Lizama et al., 2017). Nevertheless, is necessary to carry out semicontinuous studies to assess the performance under continuous disturbances to the reactor to evaluate the stability over long periods and with various organic load increments. The information provided by investigating this operational mode is more useful and representative of real-life scenarios, because the majority of high-scale anaerobic plants operate under continuous or semicontinuous conditions.

Therefore, the objective of this study was to evaluate the influence of USp at three SE levels on the semicontinuous AD of WAS with increasing organic loads during long duration experiments. Special attention was given to the performance and stability of the digesters, biogas production, organic matter reduction, volatile fatty acid production and composition, and the kinetic evaluation of biogas production, providing a full perspective of the influence of USp on the semicontinuous anaerobic digestion of WAS.

2. Materials and methods

2.1. Waste activated sludge samples

Waste activated sludge samples were collected from the municipal “Pensiones II” WWTP, which operates using an extended aeration system. This plant has the capacity to treat 12 L/s and is one of the 26 plants serving the city of Merida, Yucatan, Mexico. Sludge total solids (TS) were concentrated by mechanical filtration to obtain concentrated sludge with approximately 3% TS, which was stored at 4–6 °C prior to pretreatment and analysis. On average, the concentrated raw WAS samples used in this study had a pH of 6.68 and contained 2.89% TS, 1.9% volatile solids (VS), 1041 mg/L soluble COD ($_S$COD), and 38401 mg/L total COD ($_T$COD).

2.2. Sonication of waste activated sludge

The raw sludge used to semi-continuously feed the anaerobic reactors was treated at different SEs (15000, 25000, and 35000 kJ/kg TS). Raw sludge samples with 2.89% TS were sonicated according to the methodology described by Lizama et al. (2017) using a VCX 750 ultrasonic processor (Sonics & Materials, Inc. Newtown, CT, USA). A solid titanium high gain probe with a diameter of 25 mm was used. For each sonication batch pretreatment, a 400-mL sludge sample was placed in a 500-mL beaker. The total energy input (kJ) applied was calculated according to the specific energy formula (Equation (1)).

$$SE = \frac{P*t}{V*TS} \tag{1}$$

where SE is the specific energy input (kJ/kg TS), P is the power input (kW), t is the sonication time (s), V is the sludge volume (L), and TS is the total solids concentration (kg/L).

The increase in sludge temperature was not controlled during any USp, and continuous stirring was applied to minimize the occurrence of gradients inside the sample and increase the effectiveness of the sonication. The energy applied (J) was measured with the wattmeter of the ultrasonic processor. The sonication frequency used was according to the feed and the organic load requirements.

2.3. Semicontinuous anaerobic digesters

To observe the differences in the anaerobic biodegradability of raw and sonicated WAS, eight completely stirred anaerobic digesters with a 1-L volume (0.8 L liquid volume and 0.2 L headspace volume) were used. The reactors used were 1000-mL media bottles (KimCote™ GL 45 Media Bottles, KC14395-1000) and were processed under semicontinuous mesophilic conditions (36 ± 1 °C).

2.4. Digester start-up and operation

To initiate the experiments, the reactors were loaded with 400 mL of substrate (sonicated or raw WAS) and 400 mL of inoculum. The digesters had a final VS concentration of 21.13 ± 1.1 g/L. According to the solubilization and BMP tests results reported previously (Lizama et al., 2017), 15000, 25000, and 35000 kJ/kg TS and raw WAS (the control) were evaluated in this study. The inoculum was collected from the excess biomass of a mesophilic lab scale (6 L) anaerobic reactor treating WAS, which had been in continuous operation for more than two years and contained 19.4 g VS/L. The reactors were sealed with stoppers and screw lids and were purged for five minutes with nitrogen gas to remove oxygen from the systems. The anaerobic assays were carried out in duplicate reactors in an incubator at 36 ± 1 °C with shaking at 125 rpm, performed for an initial 20 days in batch mode. During this period, the only parameter measured was the biogas production, which was used as an acclimation stage for the corresponding substrates. The biogas produced was collected in 1000-mL graduated test tubes, and its volume was measured based on the water displacement method. After the acclimation stage, the feedstock was added to the reactors semicontinuously every 24 h at an organic rate of 1 kgVS/m^3·d. The OLR increased every 30 days (1, 2 and 3 kgVS/m^3·d). Raw and sonicated feed sludge, as well as digested samples, were stored at 4 °C and analyzed as soon as was possible.

To evaluate the influence of sonication and increases in OLR on the digesters performance, samples of the effluent digestion sludge, as well as the biogas, were collected and analyzed daily during the study. Digested sludge samples were analyzed for their operational parameters, including TS, VS, pH, alkalinity, and volatile fatty acids (VFAs). The biogas produced was measured and the methane content was quantified.

2.5. Analytical analysis procedures

The concentrations of TS and VS, pH, and alkalinity were determined according to the Standard Methods (APHA, 2005). The pH was measured using a pH meter (Thermo™ Orion, model 410). Soluble chemical oxygen demand (sCOD) and VFAs were analyzed in the soluble fractions of digested sludge samples. The soluble fractions were obtained after centrifugation at 13000 rpm for 15 min, and the resulting supernatants were subsequently filtered with a 0.45-μm membranes. The total chemical oxygen demand ($_T$COD) and sCOD were analyzed according to the colorimetric method (5220D) of the Standard Methods (APHA, 2005) using a spectrophotometer (Thermo Scientific™, Genesys 20). The individual VFAs (acetic, propionic, butyric, valeric, hexanoic and heptanoic acid) and their sum (TVFA, total volatile fatty acids) were analyzed by gas chromatography using an Agilent™ gas chromatograph (7890A GC) equipped with a flame ionization detector (FID) and a Nukol™ capillary column (15 m × 0.53 mm). A multi-component calibration standard (SUPELCO™, CRM46975) was used as the standard solution. The operating conditions were the same those reported by Lizama et al. (2017). The methane content was determined by gas chromatography using an Agilent™ 7890A GC, equipped with an FID detector and a TG-Bond Q column (Thermo Scientific™, C-22 Trace PLOT, 15 m × 0.32 mm × 10 μm). Pure methane (> 99.97%) was used as a calibration standard (PRAXAIR™). Cumulative biogas samples were collected in Tedlar™ gas sampling bags (30289-U, SUPELCO™). The conditions used for analysis were as follows: injector temperature: 250 °C; detector temperature: 300 °C; oven temperature: isothermal at 145 °C for 2.5 min. The samples (10 μL) were injected manually with a