



# Anaerobic digestion of activated sludge after pressure-assisted ozonation



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## HIGHLIGHTS

- Ozonation of activated sludge (AS) improved solids reduction and biogas yield during digestion.
- Ozonation with 10 mg O<sub>3</sub> g<sup>-1</sup> TSS via pressure cycles improved COD solubilization of AS to 18%.
- Ozonation via pressure cycles improved VSS reduction by 1.6 folds and biogas yield by 8 folds.

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## ABSTRACT

This study was undertaken to examine the benefits of pressure-assisted ozonation (PAO) in enhancing solids reduction and biogas production in anaerobic digestion. The results showed significant improvements in both. With unacclimated inoculum at varied food-to-inoculum (*F/I*) ratios of 0.5–2, solids and COD reductions were improved by PAO, as well as by increased *F/I* ratios even without PAO, which would warrant further optimization of the *F/I* ratio for an unacclimated inoculum. With acclimated inocula at *F/I* ratio of 0.8, volatile suspended solids reduction and biogas production were improved by up to 60% and 800%, respectively, when the AS had been subjected to 20 cycles of PAO. In extended operation in plants where acclimated anaerobes are encountered, PAO pretreatment offers improved digestion of AS in terms of solids and COD removals and biogas production.

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## 1. Introduction

Excess wasted activated sludge (AS) is a significant issue worldwide prompting numerous recent studies aiming to alleviate the consequential environmental burden. Ozonation has shown promising results for improving anaerobic digestibility of AS from bench to full-scale wastewater treatment plants (Bougrier et al., 2007; Carrere et al., 2010; Elliott and Mahmood, 2007; Weemaes et al., 2000b; Yasui et al., 2005; Yeom et al., 2002). Cell solubilization as manifested in flocs disintegration, solids reduction, and soluble organics increases is generally achieved when 50 or more mg O<sub>3</sub> g<sup>-1</sup> dry solids is applied to AS (Park et al., 2003; Zhang et al., 2009). Studies have shown increased anaerobic biogas production with increased sludge solubilization by increased ozone dosage (Bougrier et al., 2006; Carrere et al., 2010; Chu et al., 2009b; Weemaes et al., 2000a). For example, a 155% increase in biogas production was observed with a dose of 150 mg O<sub>3</sub> g<sup>-1</sup> solids, and only 31% increase with 15 mg O<sub>3</sub> g<sup>-1</sup> solids (Bougrier et al., 2007). However, life cycle assessment of anaerobic digestion has shown that

ozonation may not be beneficial for solubilization of biological wastes (e.g., kitchen wastes and sewage sludge), meaning that the expended energy may result in a greater environmental burden than benefit (Carballa et al., 2011). However, few studies examined low dosage for sludge ozonation or demonstrated good solubilization efficiencies at low dosage (Chu et al., 2009a).

Pressure gradient ( $\Delta P > 30$  bar) was also tried for sludge treatment to enhance flocs disintegration, cell rupture, leading to increased volatile solids (VS) removal and biogas production (Carrere et al., 2010; Elliott and Mahmood, 2007; Park and Clark, 2002; Rai and Rao, 2009). VS removal was increased from 35% to 50% when the AS was treated by the plate collision method ( $\Delta P = 30$  bar); biogas production was increased by 18% when the AS was treated by a homogenizer at 600 bars of pressure gradient (Barjenbruch and Kopplow, 2003; Carrere et al., 2010; Choi et al., 1997). Solubilization efficiency was more effective with high pressure gradient than with low ones. With pressure gradient of 10 bar with CO<sub>2</sub>, COD solubilization was merely 5% with little improvement in VS removal (Ma et al., 2011). Pressure-assisted ozonation (PAO) was used for disrupting aggregated materials, including AS, sediment, and soil (Cheng et al., 2012; Hong and Nakra, 2009; Hong et al., 2008). A previous study showed when filamentous

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AS flocs were subjected to PAO, they became disrupted showing viscous patches with little discernible AS remaining (Cheng et al., 2012). PAO was effective that it delivered dissolved air and ozone into cells under hyperbaric pressure which upon pressure release produced expanding gas bubbles from within the cells leading to the cells' rupture and release of intracellular materials (Cheng et al., 2012). Relative to ozonation with and without the use of microbubble, PAO demonstrated advantages in COD and solids solubilization with low ozone dose and short contact time (Cheng et al., 2012; Chu et al., 2008, 2009a; Dogruel et al., 2007; Yeom et al., 2002). The advantages were attributed to microbubbles being created ubiquitously throughout the reactor volume and not dependent on the location of the gas injector (Chu et al., 2008, 2009a; Cha et al., 2010).

In assessing biogas production and biodegradability, feed-to-inoculum ratio ( $F/I$ ) is an important factor (Braguglia et al., 2006; Jensen et al., 2011; Tomei et al., 2008). In some cases, the  $F/I$  ratio became a critical factor (Neves et al., 2004). For example, solids reduction and sludge hydrolysis were increased in sonicated AS as well as in untreated AS when the  $F/I$  ratio was increased from 0.1 to 4 (Tomei et al., 2008). Although ozonation pretreatment of AS had been extensively studied, results are not available on batch anaerobic digestion or on its effectiveness as influenced by  $F/I$  of the fed AS having been subjected to low ozone dose via PAO (<50 mg O<sub>3</sub> g<sup>-1</sup> dry solids). While the previous study showed advantages of PAO in AS solubilization, the present study has investigated the biodegradability of PAO-treated sludge by batch anaerobic digestion and the kinetics of solid reduction and biogas production of the PAO-treated sludge at different  $F/I$  ratios. An ultimate goal is to reduce excess sludge volume and enhance biogas production during conventional anaerobic digestion.

## 2. Methods

### 2.1. Sludge and inoculum sources

Weekly samples of returned AS were taken from Central Valley Water Reclamation Facility (CVWRF), Salt Lake City, Utah. The AS samples were kept in a 4-L bottle in the refrigerator (4 °C) until use in 48 h. Two inocula were used for the study, the fresh inoculum from CVWRF (FI) and the laboratory incubated inoculum (LI). The original inoculum was obtained at the anaerobic digester of the CVWRF and used on the same day for the digestion tests as FI, whereas LI from the same source had been incubated in two 2-L jacketed beakers at 35 °C for more than 150 d before digestion tests. A digestion period of 21 d was used in order to compare with related studies (Bougrier et al., 2007; Carrere et al., 2010; Ma et al., 2011; Rani et al., 2012; Weemaes et al., 2000b). During incubation, LI was fed with AS from CVWRF with both solids retention time (SRT) and hydraulic retention time (HRT) of 21 d. The COD and the volatile solids to total solids ratio (VS/TS) of AS was  $10 \pm 1$  g L<sup>-1</sup> and 0.8, respectively. The volatile suspended solids (VSS) concentration of FI and LI was 12–14 and 3–5 g VS L<sup>-1</sup>, respectively, and VSS to total suspended solids ratio (VSS/TSS) of FI and LI was 0.7 and 0.8, respectively.

### 2.2. Sludge pretreatment

In PAO treatment, 1.2 L of AS was placed in a 1.5-L closed reactor and subjected to 10 or 20 cycles of compression (with an O<sub>3</sub>/air stream) and decompression. PAO treatment and supplied ozone dose determination were previously detailed (Cheng et al., 2012). The ozone generator (Model T-816, Polymetrics) was fed with dry, filtered oxygen or compressed air at 105 V at the

flow rate of 2 L min<sup>-1</sup>. Each pressure cycle began by compressing an ozone-air mixture (0.06% O<sub>3</sub> v/v) into the reactor to reach 1040 kPa by an air compressor (RIDGID model 45150); once reaching the target pressure, it was held there for 30 s for equilibration and then quickly vented to the ambient pressure in 3–5 s. For comparison, the same volume of AS was placed in the same reactor and contacted with an ozone-air mixture (0.9% O<sub>3</sub> v/v) bubbling through the suspension at 2 L min<sup>-1</sup> for 15 min under ambient pressure. In all ozonation treatments, any ejected foam and light particulate matters were collected for analysis and mass balance calculation. Solids concentration (TS, VS, TSS and VSS), pH, and COD of sludge were determined before and after treatment per Standard Methods (APHA et al., 2005); soluble COD (sCOD) of sludge was determined for the filtrate having passed through a 1.5- $\mu$ m glass filter (Whatman 934-AH) and closed reflux colorimetric method per Standard Methods (APHA et al., 2005).

### 2.3. Batch anaerobic digestion with LI

Batch anaerobic digestion test with LI was carried out for 20 d in 125-mL Erlenmeyer flasks. Prior to placement of the treated (20 PAO and conventional ozonation) and untreated AS samples, the flasks were purged with nitrogen and each flask contained 30 mL of LI. To maintain a constant  $F/I$  ratio (g VS of AS/g VS of inoculum) of 0.8 in experiments with 30 mL of LI, the volume of AS (with varying VS concentrations) to be incubated was varied. For example, 20 mL of untreated AS inoculated with 30 mL of LI will have the same  $F/I$  ratio as 22 mL of PAO-treated AS (of lower VS concentration) inoculated with 30 mL of LI. All flasks were sealed by rubber-sleeved stoppers and were incubated in a water-bath shaker agitated at 100 rpm at 35 °C (New Brunswick G76). Each flask was connected to a DI water-filled gas collection tube, which measured biogas production via liquid displacement. The COD, solids, and pH of the digested AS/inoculum mixtures were measured in triplicate or more at day 0 and 20 per Standard Methods (APHA et al., 2005). Biogas production was measured at regular time intervals within the 20-d digestion period.

Separate digestion test was performed at 35 °C for nearly 150 d in two jacketed beakers each of 1.5-L working volume approximating complete-mix anaerobic reactors. The reactor was mixed continuously by a magnetic stirrer at 200 rpm. The original inoculum in both reactors was FI, fed with AS for a SRT of 21 d over 10 months prior to experimentation. At day 0, one digester was fed with untreated AS and the other with treated AS by 20 cycles of PAO. The  $F/I$  ratio was  $0.8 \pm 0.1$  and the SRT was 21 d for both digesters. TS, TSS, VS, VSS, COD, sCOD, and pH were measured weekly per standard methods (APHA et al., 2005). Methane content in the biogas and the biogas production rate were determined from day 63 onward. Biogas was collected and measured weekly by water displacement and its methane content measured periodically by GC-TCD (HP 7890) equipped with a CARBONPLOT column.

### 2.4. Batch anaerobic digestion with FI

Anaerobic digestion was carried out over 14 d in batch using FI. The 125-mL Erlenmeyer flasks were likewise purged by nitrogen and mixed the AS with inoculum in different amounts to arrive at 49–68 mL of working volume, resulting in different  $F/I$  ratios at 0.5, 1, and 2 g VS/g VS. The COD, sCOD, solids, and pH of the digested sludge along with the biogas production rate were measured in duplicate at intervals within the 14-d digestion period per Standard Methods (APHA et al., 2005).

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