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Conductive heating and microwave hydrolysis under identical heating profiles for advanced anaerobic digestion of municipal sludge



Seyedeh Neda Mehdizadeh, Cigdem Eskicioglu*, Jake Bobowski, Thomas Johnson

School of Engineering, University of British Columbia, Okanagan Campus, 1137 Alumni Ave., Kelowna, BC V1V 1V7, Canada

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ABSTRACT

Microwave (2.45 GHz, 1200 W) and conventional heating (custom pressure vessel) pretreatments were applied to dewatered municipal waste sludge (18% total solids) using identical heating profiles that span a wide range of temperatures (80–160 °C). Fourteen labscale semi-continuous digesters were set up to optimize the energy (methane) output and sludge retention time (SRT) requirements of untreated (control) and thermally pretreated anaerobic digesters operated under mesophilic and thermophilic temperatures. Both pretreatment methods indicated that in the pretreatment range of 80-160 °C, temperature was a statistically significant factor (*p*-value < 0.05) for increasing solubilization of chemical oxygen demand and biopolymers (proteins, sugars, humic acids) of the waste sludge. However, the type of pretreatment method, i.e. microwave versus conventional heating, had no statistically significant effect (p-value >0.05) on sludge solubilization. With the exception of the control digesters at a 5-d SRT, all control and pretreated digesters achieved steady state at all three SRTs, corresponding to volumetric organic loading rates of 1.74-6.96 g chemical oxygen demand/L/d. At an SRT of 5 d, both mesophilic and thermophilic controls stopped producing biogas after 20 d of operation with total volatile fatty acids concentrations exceeding 1818 mg/L at pH <5.64 for mesophilic and 2853 mg/L at pH <7.02 for thermophilic controls, while the pretreated digesters continued producing biogas. Furthermore, relative (to control) organic removal efficiencies dramatically increased as SRT was shortened from 20 to 10 and then 5 d, indicating that the control digesters were challenged as the organic loading rate was increased. Energy analysis showed that, at an elevated temperature of 160 $^\circ$ C, the amount of methane recovered was not enough to compensate for the energy input. Among the digesters with positive net energy productions, control and pretreated digesters at 80 °C were more favorable at an SRT of 10 d. © 2013 Elsevier Ltd. All rights reserved.

1. Introduction

The treatment of wastewater produces semi-solid residuals, commonly called "sludge", which must be disposed to the

environment. The high quantities of waste sludge are produced in our modern society. According to Okuno (2007), annual production of sludge in Canada is at least 0.4 million dry metric tonnes and United States produced more than 7.5

^{*} Corresponding author. Tel.: +1 250 807 8544.

E-mail addresses: cigdem.eskicioglu@ubc.ca, cigdem.eskicioglu@gmail.com (C. Eskicioglu). 0043-1354/\$ – see front matter © 2013 Elsevier Ltd. All rights reserved. http://dx.doi.org/10.1016/j.watres.2013.05.055

million dry metric tonnes of sludge in 2010 (USEPA, 1999; Sanin et al., 2011).

Anaerobic digestion has become a major part of modern wastewater treatment plants (WWTPs) due to the potential of using the biogas generated from the waste as an energy source. In digestion, hydrolysis is generally the rate limiting step and results in longer sludge retention time and therefore digester volume requirements (Tiehm et al., 2001; Vavilin et al., 2003). Application of physical, biological and chemical pretreatments, prior to digestion, can release intracellular material into the water phase and accelerate the hydrolysis (Appels et al., 2008; Mottet et al., 2009; Zheng et al., 2009). In addition to aforementioned pretreatments, combinations of different methods have also been used for their synergistic effects (Eskicioglu et al., 2008; Yin et al., 2007; Chang et al., 2011; Tyagi and Lo, 2012). In most of the pretreatments, an accelerated hydrolysis/degradation rate results in reduced volume requirement of the digester and lower capital costs for a given organic load (Carrère et al., 2010).

Among various methods, thermal pretreatments are known to result in greater enhancements to the biogas production. Heat can disrupt the gel-like structure of the flocs releasing intracellular/extracellular biopolymers and chemical oxygen demand (COD) into soluble phase (Eskicioglu et al., 2006), but the solids, which have lower affinity to water, coagulate (Tchobanoglous et al., 2003). These alterations are independent of reaction time, but depend heavily on the treatment temperature (Valo et al., 2004). Furthermore, sludge sanitization and enhanced dewaterability are other advantages of thermal pretreatment. The effect on methane production depends on sludge type (Carrère et al., 2008). In some studies, although pretreatment increased solubilization at temperatures above 150 °C, it did not result in enhanced methane production (Dwyer et al., 2008). These results can be explained by Maillard reactions that occur at excessively high temperatures (170–190 °C). During these reactions, carbohydrates and amino acids transform to melanoidins which are difficult to degrade (Bougrier et al., 2008). After thermal pretreatment, the amount of energy produced via biogas is usually in excess of the treatment plant energy requirements (Carrère et al., 2010).

Two common methods for thermal pretreatment are conventional heating (CH) and microwave irradiation (MW). In CH, heat flow initiates from the surface of the material and the rate of heating depends on the material thermal properties and temperature differential. On the other hand, MW heating is a volumetric heating that gives rapid energy transfer to the material (Bogdal and Prociak, 2007). At elevated temperatures (170-190 °C) in a Zipperclave system, the viscosity of sludge decreased and it behaved more like a Newtonian fluid (Bougrier et al., 2006a). Valo et al. (2004) reported that thermal treatment of waste activated sludge (WAS), in an autoclave at temperatures of 130 °C, 150 °C and 170 °C, led to high solubilization of organic matters, but it did not affect the mineral content of sludge. In addition to direct temperature effects, at high temperatures, pressure difference can cause cell destruction. This leads to sludge sterilization making the digested product potentially useful for land application (Bougrier et al., 2006b).

Many studies reported that optimal operating conditions for sludge pretreatment are heating at 160-180 °C for

30–60 min (Bougrier et al., 2008; Tanaka et al., 1997). The pressures associated with these temperatures are in the range of 600–2500 kPa (Pinnekamp, 1989). The pretreatment time has little effect on biodegradation in this high-temperature range (Neyens and Baeyens, 2003; Dohanyos et al., 2004). In general, increases of the solubilization of sludge COD has a linear correlation with methane production (Carrère et al., 2008); however this may not be the case for pretreatments involving chemical addition due to the generation of refractory compounds (Eskicioglu et al., 2008; Saha et al., 2011).

Although MW treatments have been used widely in industrial, domestic and medical applications, MW applications as remediation tool for treatment of soils, sludge and wastewater are relatively new, but have been steadily growing (Nüchter et al., 2004; Pino-Jelcic et al., 2006; Wu, 2008). In the study by Wong et al. (2006), it was demonstrated that at MW temperatures above 120 °C, complete cell lysis occurred and heavy metals and nutrients are released into the supernatant along with COD solubilization. Other studies reported that pretreatment temperatures in the range of 37-96 °C increased the COD and biopolymer solubilization (Pino-Jelcic et al., 2006; Kennedy et al., 2007; Eskicioglu et al., 2007a); however the sludge concentration (1-5% w/v) and MW intensity did not have a significant effect on particulate COD solubilization quantified by soluble to total COD ratios (Kennedy et al., 2007). Similar to other pretreatments, the level of improvements in solubilization and biodegradation depended on sludge type.

Despite extensive development in thermal pretreatment methods for waste sludge, a comprehensive comparison between MW and CH over a wide range of temperatures (below and above the boiling points) with identical heating rates has not been done at the lab- or pilot-scale due to lack of instrumentation. Previous studies (Eskicioglu et al., 2007b; Hong et al., 2004; Bose et al., 1991) intended to compare the CH and MW heating for enhanced biogas production, as well as to investigate athermal effects. Athermal effects have been attributed to the polarization of macromolecules, and their alignment with the electromagnetic field poles that may cause breakage of hydrogen bonds (Loupy, 2002; Eskicioglu et al., 2008). However, these studies could not be performed under identical heating rates over a wide temperature range. Chang et al. (2011) compared the effects of CH (520 W, 80 °C, thermostatic bath) versus MW (600 W, 85 °C, 2450 MHz) on the sludge disintegration/aerobic digestion. The heating rates of methods were not similar as samples took 2 min and 12 min to reach 85 °C and 80 °C by MW and CH systems, respectively. Sludge solubilizations by MW and CH were 8.5% and 7%, respectively. Energy input evaluation indicated that MW irradiation consumed 5 folds less energy than the CH system due to shorter exposure time and minimized heat loss. Eskicioglu et al. (2007c) pretreated WAS, under boiling point temperatures (50-96 °C), by MW and CH under identical heating profiles (°C/min) to study the athermal effects of MW irradiation. In this study, both MW and CH WAS samples resulted in similar particulate COD and biopolymer (proteins and polysaccharides) solubilization. Furthermore, there were no noticeable MW athermal effects on the COD solubilization of WAS. However, improved biogas production (13%) for MW samples over CH samples was observed in the same study. It was concluded that the MW athermal effects had a positive

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