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## Effects of ultrasonic pretreatment on the solubilization and kinetic study of biogas production from anaerobic digestion of waste activated sludge



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### ABSTRACT

Waste activated sludge (WAS) is a polluting waste with severe management problems that must be treated to prevent pollution and human health risks. Anaerobic digestion (AD) is the most used process to stabilize sludge; however, it must be improved because the biomethanation of sludge entails low biodegradability, pathogen inactivation, and biogas production. This study investigated the effects of ultrasonic pretreatment (USp) of WAS as a strategy to improve AD. Macromolecule solubilization, heavy metal behavior, pathogen inactivation and biogas production were evaluated. USp was applied at a range of 5000–35000 kJ/kg TS (total solids). The maximum solubilization degree of soluble chemical oxygen demand was 26%, and 22.9% of proteins at 35000 kJ/kg TS. The highest USp reduced only 2 log units of pathogens; nevertheless, a high inactivation was obtained when TS were reduced to 2% and continuous stirring was applied. AD of raw and sonicated WAS were compared with biochemical methane potential tests, and a biogas overproduction of 31.43% (219.5 mL/g VS) was achieved at highest USp. A modified Gompertz model was used for kinetic study of biogas production. The kinetic parameters at highest energy were: biogas production potential,  $G_0 = 634.2$  mL; maximum biogas production rate,  $R_{max} = 57.23$  mL/day.

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### 1. Introduction

Waste activated sludge (WAS) is a polluting and hazardous waste generated in wastewater treatment plants (WWTP) and has serious management problems. The concentration of heavy metals and pathogens limit the implementation of standard disposal methods (Martín et al., 2015). Therefore, WAS must be adequately treated to prevent environmental pollution and human health risks. Direct applications of WAS in agriculture, landfill disposal, and composting are losing popularity due to increasingly stringent restrictions (CEC, 1986; U.S. EPA, 1994).

Anaerobic digestion (AD) is a biological process whereby organic matter from the substrate is degraded by microorganisms in the absence of oxygen and is the most widely used method to stabilize sewage sludge (Liao et al., 2016). AD has the advantages of

(1) reducing the sludge volume, (2) generating energy-rich biogas, and (3) yielding a nutrient-containing final biosolid (Kim et al., 2010). It is also the preferred stabilization method of WAS because of its low cost and low energy footprint (Kim et al., 2015). The anaerobic process involves four stages: hydrolysis, acidogenesis, acetogenesis and methanogenesis. During hydrolysis, both solubilization of particulate matter and biological decomposition occurs via exoenzymes. This happens slowly, making it the rate-limiting step of the process (Alagöz et al., 2015; Lin et al., 2016; Zhen et al., 2017). However, methane production during the last stage may be adversely affected if the conditions are not adequate, such as pH, the type of inhibitors present (ammonia, sulfide, heavy metals, and organics), and overproduction and accumulation of volatile fatty acids during hydrolysis and acidogenesis (Liu et al., 2014; Lin et al., 2014). In the AD of WAS, the hydrolysis is limited due to the low biodegradability of the cell walls and extracellular polymeric substances of microorganisms; however, it can be enhanced by a pretreatment step. For this reason, many

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pretreatments have been studied to improve WAS biodegradability and biogas production, including thermal, mechanical, chemical or biological pretreatments (Appels et al., 2008; Carrère et al., 2010).

Ultrasonic pretreatment (USp) is the most effective sludge disintegration method and has been studied in the laboratory as well as in pilot and full-scale reactors (Pilli et al., 2011). USp has been examined because it generates a high level of solubilization, is a simple technology, increases the production of biogas and methane content, improves biosolid quality, does not require the addition of chemicals and can easily be coupled to other pretreatments (Houtmeyers et al., 2014; Naran et al., 2016). The disintegration of sludge by USp entails breaking sludge flocs, destroying the bacteria cell wall, and releasing soluble organic matter. Enhancing the solubilization of WAS promotes the rate of sludge hydrolysis, biodegradation and biogas yield during AD (Sahinkaya and Sevimli, 2013; Pilli et al., 2016).

The control parameters that have been studied in the USp of sludge include frequency, the energy applied, power input, time, type of sludge, and the concentration of solids. The energy applied to sludge can be expressed in several ways, including as the specific energy applied (SE) (kJ/kg TS), ultrasound dose (J/L), ultrasound density (W/L) or ultrasound intensity (W/cm<sup>2</sup>). Thus, it is not possible to compare the results in the literature because not all studies quantify the energy applied per content of solids (Table 1) (Tyagi et al., 2014; Le et al., 2015). It has been found that the solids content is a limiting factor for USp effectiveness; however, some studies do not consider this parameter. The optimal concentration of solids for a suitable USp are between 2.1 and 3.2% (Carrère et al., 2010; Appels et al., 2012).

Although several authors have studied the use of USp before the AD of sludge, a consensus has not been reached with regard to the optimization of the pretreatment parameters. As demonstrated in Table 1, the available literature shows that a wide range of specific energy have been studied, from 96 kJ/kg TS to 214400 kJ/kg TS, with very contrasting results about the increase on biogas production. The most commonly investigated specific energy range is between 1000 and 16000 kJ/kg TS. Nevertheless, it is difficult to standardize the effectiveness of USp of sludge based on available studies, due to the different treatment conditions applied in each study (Carrère et al., 2010; Pilli et al., 2011; Le et al., 2015; Zhen et al., 2017). Another important factor in USp is the equipment used. Ultrasonic cleaning baths and simple ultrasonic processors are not suitable for USp studies because they do not allow the amount of energy applied to sludge to be measured, assuming that the power delivered is constant during pretreatment. This fact increases the overall uncertainty. Likewise, most USp studies only assess their effect on

chemical oxygen demand (COD) solubilization or biogas production; few studies have focused on evaluating the effect of USp on the release of macromolecules, inactivation of pathogens and concentration of metals simultaneously in the same study, which are key factors that justify the use of a pretreatment to comply with current regulations.

It is clear that very large discrepancies exist between research studies with respect to the energy applied, energy expressions, equipment used, solids content, among other factors. Therefore, the main objective of this study was to apply and evaluate the effects of USp over a wide range of specific energies (5000–35000 kJ/kg TS) before the AD of WAS. Special attention was paid to macromolecule solubilization (carbohydrates and protein), the behavior of heavy metals and volatile fatty acids, pathogen inactivation, and the kinetic study of biogas production, providing a full perspective of the effects of USp on WAS solubilization and its subsequent AD. Finally, a modified Gompertz model was used for the kinetic study of biogas production to obtain the kinetic parameters of each USp; this equation has been identified as a good empirical non-linear regression model and is commonly used in the simulation of biogas accumulation (Deepanraj et al., 2015; Young et al., 2016; Kafle and Chen, 2016).

## 2. Materials and methods

The experimental study was carried out in two parts. In the first part, the effects of sonication on the sludge properties were investigated. The solubilization of WAS, based on the increase in the soluble chemical oxygen demand (sCOD), carbohydrates, proteins, volatile fatty acids (VFA) and heavy metals, was evaluated. In the second part, the influence of sonication on biogas production and the anaerobic biodegradability of sludge were studied by biochemical methane potential (BMP) assays in batch anaerobic reactors.

### 2.1. Waste activated sludge

WAS samples were collected from a municipal WWTP in Merida, Yucatan, Mexico. Sludge total solids (TS) were concentrated by gravity settling and mechanical filtration to obtain concentrated sludge with 2–3% TS, which was stored at 4 °C until further pretreatment and analysis. WAS contained 2.58% TS, 1.77% volatile solids (VS), 46433 mg/L total COD (τCOD), and 888 mg/L sCOD.

**Table 1**  
Effects of ultrasonic pretreatment on solubilization and anaerobic digestion of sludge.

Type of sludge (solids content)	Ultrasonic conditions/ Equipment	Energies applied	Anaerobic digestion conditions	Results	Reference
WAS (1.78% TS)	20 kHz, 60 W, 50 mL. Ultrasonic homogenizer	3600 - 108000 kJ/kg TS	Batch, 50 days, 37 °C	Increase biogas production of 84% at 108000 kJ/kg TS	Salsabil et al. (2009)
Activated Sludge (3.5% TS)	20 kHz, 100 W, 900 mL. Ultrasonic homogenizer	27900 - 214400 kJ/kg TS	N.A.	Increase the ratio of soluble organic matter of 32% (sCOD/τCOD) at 214400 kJ/kg TS	Laurent et al. (2009)
WAS (2.1% TS)	25 kHz, 0–1000 W. Tubular ultrasound reactor	0–1968 kJ/kg TS	Batch, 23 days, 37 °C	Increase methane production of 20% at 326 kJ/kg TS	Appels et al. (2012)
WAS (3.93% TS)	20 kHz, 200 W, 100 mL. Ultrasonic Homogenizer	0.5–1.5 W/mL, 1 min	Batch, 78 days, 37 °C	Increased biogas production of 6.3% at 1 W/mL, 1 min	Sahinkaya and Sevimli (2013)
Thickened sludge (4.36% TS)	100 W, 500 g of sludge. Horn type generator	96 kJ/kg sludge	Semi-continuous, 37 °C, 67 days	Increase biogas production of 27%	Houtmeyers et al. (2014)
Sewage sludge (13.2% TS)	150 W, 30 g of sludge. Ultrasonic cleaning bath	13500 kJ/kg sludge	Batch, 35 °C	Increase methane yield of 97%	Martín et al. (2015)
WAS (0.75% TSS)	30 min, 360 kJ/L. Ultrasonic homogenizer	360 kJ/L	Batch, 35 °C, 20 days	Increased methane production of 31%	Naran et al. (2016)

N.A.: Not applicable.

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