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# Evaluation of the improvement of sonication pre-treatment in the anaerobic digestion of sewage sludge



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#### ABSTRACT

Sewage sludge is a polluting and hazardous waste generated in wastewater treatment plants with severe management problems. The high content in heavy metal, pathogens and micropolluting compounds limit the implementation of the available management methods. Anaerobic digestion could be an interesting treatment method, but must be improved since the biomethanisation of sewage sludge entails low biodegradability and low methane production. A sonication pre-treatment at lab scale is proposed to increase the organic matter solubilisation of sewage sludge and enhance the biomethanisation yield. Sonication time was optimised by analysing the physicochemical characteristics of sewage sludge (both total and soluble fraction) at different pre-treatment times. The pre-treatment time was fixed at 45 min under the study conditions given that the solubilisation of organic matter did not increase significantly at lower sonication times, whereas the concentration of total nitrogen increased markedly at higher times. The volatile fatty acids generation rate was also evaluated for the pre-treatment conditions. The anaerobic digestion of untreated and pre-treated sewage sludge was subsequently compared and promising results were obtained for loads of 1.0 g VS/L (VS, total volatile solids). The methane yield coefficient increased from 88 to 172 mL<sub>STP</sub>/g VS (STP, 0 °C, 1 atm) after the pre-treatment, while biodegradability was found to be around 81% (in VS). Moreover, the allowed organic loading rate and methane production rate observed for the sewage sludge reached values of up to 4.1 kg VS/m<sup>3</sup>·d and 1270 L<sub>STP</sub>/m<sup>3</sup>·d, respectively.

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## 1. Introduction

The activated sludge process is one of the most commonly used treatment technologies in municipal and industrial wastewater treatment plants (WWTPs). Although this biological method is highly efficient in the removal of organic matter and nutrients, a large amount of waste sludge is produced. Sewage sludge contains heavy metals, organic micropollutants and pathogens, which have led to stringent legislation for sewage sludge applications (Hendrickx, 2009). Sewage sludge should therefore be adequately treated to prevent environmental pollution and human health risks. In this sense, the direct application in agriculture and composting of sewage sludge are losing focus due to the increasingly stringent restrictions on heavy metal concentration as established under European legislation (Working Document on

As a result, anaerobic digestion has been proposed as an efficient and sustainable technology for sewage sludge treatment (Appels et al., 2008). The benefits associated with anaerobic technology include mass reduction, odour removal, pathogen reduction, and, more significantly, energy recovery in the form of methane (Pilli et al., 2011). Nevertheless, the anaerobic digestion of sewage sludge has several disadvantages such as low methane production and biodegradability, which lead to high retention times and mixing costs (Bolzonella et al., 2005).

Different pre-treatments have been proposed to improve the biomethanisation of sewage sludge. Specifically, efforts have

sludge and biowaste, proposal to revise Directive 86/278/EEC on the agricultural use of sewage sludge and sewage sludge management, 2010). Landfill disposal cannot be considered an adequate management method due to the serious impact it has on the environment, such as odorous emissions, the generation of polluting lixiviate and landfill saturation. Moreover, the European Union established the target of decreasing landfilling as a sewage sludge management method by 50% in 2050 compared to 2000 (Lundin et al., 2004).

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#### Nomenclature

Alk alkalinity (mg CaCO<sub>3</sub>/L)

COD chemical oxygen demand (g O<sub>2</sub>/kg)

D.L. detection limit

FS total fixed solids (mg/kg)

GAL glucose, sodium acetate and lactic acid solution

N-NH<sub>4</sub><sup>+</sup> ammoniacal nitrogen (mg/L; mg/kg) OLR organic loading rate (kg VS/m<sup>3</sup>·d)

P<sub>total</sub> total phosphorus (mg/kg)

STP standard temperature and pressure conditions

(0 °C, 1 atm)

time required to reach 95% of the total methane

production (h)

TN soluble total nitrogen (mg/L)

TS total solids (mg/kg)

VFA volatile fatty acid (mg acetic acid/L)
VS total volatile solids (g/L; mg/kg; g/kg)
Y<sub>CH4/S</sub> methane yield coefficient (mL<sub>STP</sub> CH<sub>4</sub>/g VS)

been made to facilitate the hydrolysis phase, which is the rate limiting step when degrading solid organic waste with a high content of complex compounds (Ortega et al., 2008; Gabriel et al., 2011). Different pre-treatments, such as physical, biochemical, acidic or alkaline, heat-shock, freezing and thawing processes, have been proposed prior to the biomethanisation of organic solid wastes (Carrère et al., 2010; Cesaro and Belgiorno, 2014). Physical pre-treatments by ultrasound lead to the formation of cavitation bubbles in the liquid phase. These bubbles grow and then violently collapse causing high shearing forces in the surrounding liquid phase of the waste and the formation of radicals (Braguglia et al., 2012). The disintegration of the sludge entails breaking the sludge flocs, destroying the bacteria cell wall, and releasing the organic matter located inside the cell into the bulk liquid (Chang et al., 2011; Tyagi et al., 2014). Moreover, non-soluble organic matter is transformed into soluble forms, with the consequent positive effect on the subsequent biomethanisation process (Gronroos et al., 2005; Zhang et al., 2010).

Although several authors have described the use of ultrasound pre-treatments before the anaerobic digestion of sewage sludge, consensus has not been reached with regard to the optimisation of the pre-treatment parameters, such as ultrasound frequency, power input, intensity, and time, among others (Pilli et al., 2011; Tyagi et al., 2014). For example, the proposed sonication time values vary in the range of 1-150 min, while the power values range from 0.2 to 9.0 kW (Tiehm et al., 2001; Braguglia et al., 2011). Moreover, the space-time variability in the physicochemical characteristics of sewage sludge lead to important differences in the solubilisation of COD, TOC or nitrogen as observed in different studies (Feng et al., 2009a; Castro and Capote, 2007). Furthermore, most of the previous studies in the literature focus on improving the methane yield, but do not provide substantial information about the kinetics or enhancement of the treatment capacity of the process.

The present research work responds to the demand for optimisation a sonication technology by sewage sludge managers who must innovate to comply with European legislation. The main purpose of this study is to optimise the sonication pretreatment and evaluate its effects on sewage sludge biomethanisation with a view to preventing problems traditionally associated with the direct biomethanisation of this sludge.

#### 2. Materials and methods

#### 2.1. Chemical analyses

#### 2.1.1. Sonication pre-treatment

The following parameters were determined in the solid fraction of sewage sludge before and after treatment: total chemical oxygen demand (COD, g O<sub>2</sub>/kg), total solids (TS, g/kg), total fixed solids (FS, g/kg), total volatile solids (VS, g/kg), total phosphorus (P<sub>total</sub>, mg/kg) and ammoniacal nitrogen (N-NH4, mg/kg). Volatile fatty acidity (VFA, mg acetic acid/L), pH, and alkalinity (Alk, mg CaCO<sub>3</sub>/L were also analysed to characterise the soluble fraction of the substrate. All analyses were carried out in accordance with the test methods for the examination of composting and compost developed by the US Department of Agriculture and the US Composting Council (Thompson et al., 2001). Additionally, total soluble organic carbon (TOC; mg/L) and total soluble nitrogen (TN, mg/L) were determined using a Rosemount analytical Dohrmann DC-190 carbon analyser. The TOC analyser was calibrated with a standard solution of potassium phthalate prior to the TOC analyses. Separate volatile fatty acids (acetic, propionic, butyric, isobutyric, valeric, isovaleric, and caproic acids) were also determined in the sewage sludge after extraction with distilled water (Thompson et al., 2001). The determination was carried out using a Hewlett-Packard HP-5890 gas chromatograph equipped with a 15 m  $\times$  0.53 mm (i.d.) Nukol-silica semicapillary column and a flame ionisation detector. The oven temperature was gradually increased from 100 to 150 °C at a rate of 4 °C/min. Helium (28.6 kPa) was used as carrier gas at a flow rate of 50 mL/min. Hydrogen (14.3 kPa) and air (28.6 kPa) were used together to ignite the flame of the FID.

### 2.1.2. Biomethanisation

The following parameters were determined in the effluents of the reactors: pH, total chemical oxygen demand (COD, g  $O_2/kg$ ), total solids (TS, g/kg), total fixed solids (FS, g/kg), total volatile solids (VS, g/kg), volatile fatty acidity (VFA, mg acetic acid/L), and alkalinity (Alk, mg  $CaCO_3/L$ ). All analyses were carried out in accordance with the Standard Methods of the APHA (APHA, 1989).

#### 2.2. Substrate

The raw material used as substrate was sewage sludge collected from the urban wastewater treatment plant of Puente Genil (Cordoba, Spain). The wastewater treatment plant generates sewage sludge at a flow rate of 68.44 tonnes per year, on dry basis. The sewage sludge was composed of primary and secondary sludge and dehydrated in the plant by centrifugation after the addition of coagulant and flocculant. The main analytical characteristics of the substrate are shown in Table 1.

#### 2.3. Experimental set-up

#### 2.3.1. Sonication

The sonication system employed for the pre-treatment of sewage sludge consisted of a 6.0-L volume Selecta P. 3000513 ultrasonic cleaning bath under the following conditions: 25 °C, atmospheric pressure and a power generator of 150 W. The ultrasound system was equipped with a time programming system that allows the pre-treatment time to be controlled. The experimental pre-treatment times were fixed at 0, 15, 30, 45 and 60 min. The time range was selected in accordance with the wide time interval reported in the literature, which varies from 0.5 to 150 min (Pilli et al., 2011; Tiehm et al., 2001) depending on the power of the experimental set-up. Aliquots of 30 g of sewage sludge, which were contained in closed 0.25-L NORMAX bottles, were inserted in the basin of the device

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