



## Research article

## Optimization of a thermal hydrolysis process for sludge pre-treatment

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## ABSTRACT

At industrial scale, thermal hydrolysis is the most used process to enhance biodegradability of the sludge produced in wastewater treatment plants. Through statistically guided Box-Behnken experimental design, the present study analyses the effect of TH as pre-treatment applied to activated sludge. The selected process variables were temperature (130–180 °C), time (5–50 min) and decompression mode (slow or steam-explosion effect), and the parameters evaluated were sludge solubilisation and methane production by anaerobic digestion. A quadratic polynomial model was generated to compare the process performance for the 15 different combinations of operation conditions by modifying the process variables evaluated. The statistical analysis performed exhibited that methane production and solubility were significantly affected by pre-treatment time and temperature. During high intensity pre-treatment (high temperature and long times), the solubility increased sharply while the methane production exhibited the opposite behaviour, indicating the formation of some soluble but non-biodegradable materials. Therefore, solubilisation is not a reliable parameter to quantify the efficiency of a thermal hydrolysis pre-treatment, since it is not directly related to methane production. Based on the operational parameters optimization, the estimated optimal thermal hydrolysis conditions to enhance of sewage sludge digestion were: 140–170 °C heating temperature, 5–35min residence time, and one sudden decompression.

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## 1. Introduction

In a conventional wastewater treatment plant, up to 60% of chemical oxygen demand (COD) initially present in the influent is recovered as a mixture of primary and activated sludge (Garrido et al., 2013). A new perspective is being generated due to the increase of the amount of sewage sludge and its disposal limitation by reason of strict environmental regulations, effective methods of making full use of sludge's rich organic components are on continuing interest (Zhang et al., 2014). Anaerobic digestion still appears as the most suitable method to treat the sludge due to its limited environmental impact, high potential for energy recovery as biogas, and reduction in the amount of biosolids to be disposed (Ariunbaatar et al., 2014). It is well known that for solid wastes the hydrolysis (liquefaction or solubilisation) step is the main rate limiting factor for digestion. The introduction of a physical, chemical or biological pre-treatment step before digestion has

demonstrated to improve the global kinetics and performance of the process.

On an industrial scale, the thermal hydrolysis represents the most profitable and reliable alternative (Cano et al., 2015). The main drivers for the use of thermal hydrolysis is the better energy balance due to the increase on biogas production and on better quality of the biosolids produced after digestion, both factors positively affect the operational cost of the treatment plant. From a disposal point of view, it is important to note that the hydrolysed sludge is sterilised at elevated temperatures and complies with the Environmental Protection Agency (EPA) requirements for Class A sludge.

Although nowadays developed at industrial scale (Cambí<sup>®</sup>, Biothelys<sup>®</sup>, Exelys<sup>®</sup>, TPH<sup>®</sup>, Lysotherm<sup>®</sup>, Turbotec<sup>®</sup>), there is no general agreement on the optimum operation conditions.

Two different mechanisms can be applied to hydrolyse the sludge: i) thermal effect based on the single action of elevated temperatures and ii) steam explosion generated by a sudden decompression (Pérez-Elvira et al., 2008).

According to some researches, heating temperature and duration of the thermal pre-treatment depend on the nature of the

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sludge (Ariunbaatar et al., 2014; Appels et al., 2008). The generally accepted operation conditions vary between 150 and 230 °C temperature, with time ranging from 20 to 60 min. Regarding the steam explosion option, some commercial processes flash the sludge, while other commercial technologies do not use this steam explosion mechanism. There is no bibliographical reference evaluating if flashing or re-flashing (several sudden decompressions) can further enhance the solubilisation of organic matter thereby increasing the methane production. However, the recovery of steam when flashing the sludge can be a clear economic advantage by steam recovery.

The objective of this study was therefore to determine the optimum conditions for thermal hydrolysis of waste activated sludge after studying the combined effect of pre-treatment temperature, time and flash by Response Surface Method (RSM) using the Box-Behnken experimental design (BBD). An optimal combination of factors that maximize methane production is proposed and analyzed.

## 2. Materials and methods

### 2.1. Sludge sampling

The study was performed with a single sample of waste activated sludge (WAS), provided by the municipal waste water treatment plant of Valladolid (Spain). According to Pérez-Elvira et al. (2008), the sludge was thickened without polyelectrolyte to 14% TS (73% VS) to perform the study with concentrated sludge, which is the real operation in pre-treatment units.

The anaerobic inoculum for the biochemical methane potential (BMP) tests was sampled from the anaerobic digester in the WWTP treating mixed sludge, and pre-incubated for 2 days at 35 °C in a thermostated chamber prior to use to activate the microorganisms and to deplete most residual organic matter.

### 2.2. Thermal pre-treatment procedure

The thermal hydrolysis pilot plant operated (Fig. 1) consisted of a 20 L hydrolysis reactor heated with live steam (12 bars) from a boiler, and connected to an atmospheric flash vessel (100 L) by a decompression valve. The operation is batch, controlling heating temperature and time. The decompression is also controlled by an automatic decompression valve, that relieves the reactor pressure slowly (no flashing) or suddenly (in a steam-explosion effect). When the flash takes place, the hydrolysed solids are collected in the flash vessel at atmospheric pressure.

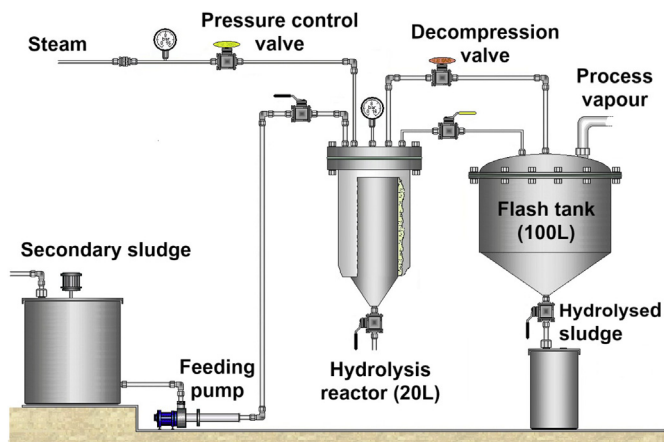


Fig. 1. Thermal pre-treatment system (Fernández-Polanco et al., 2008).

### 2.3. Anaerobic digestion tests

Biochemical methane potential (BMP) tests were performed in triplicate assays using 300 ml serum bottles, filled with 100 ml of a mixture of anaerobic inoculum and the corresponding substrate at a substrate to inoculum ratio (SIR) of 0.5 g/g (on volatile solids (VS) basis). In this test, micronutrients and macronutrients were added ensuring no nutritional limitation for optimal function of anaerobic microorganisms. Moreover, NaHCO<sub>3</sub> and Na<sub>2</sub>S were added to provide buffer capacity and avoid aerobic conditions respectively. The methodology used was based on the one suggested by Angelidaki et al. (2009).

The bottles were incubated in a thermostated chamber at 35 °C in an orbital shaker at 150 rpm/min. Methane production in the BMP tests was determined by periodic measurements of pressure and biogas composition.

### 2.4. Analytical methods and performance parameters

Total solids (TS) and volatile solids (VS) concentrations were determined according to Standard Methods (21st edition, 2005). The soluble phase for chemical oxygen demand (SCOD) was obtained by centrifugation at 5000 rpm for 10 min and filtration using a 47 mm hydrophilic Glass Fiber filter with a 0.7 µm pore size (AP40). The total COD was obtained by a direct COD analysis.

The pressure in the headspace of the BMP bottles was measured with a pressure sensor PN 5007 (IFM, Germany), and biogas composition was determined using a gas chromatograph coupled to a thermal conductivity detector (Varian CP-3800, USA).

Two performance parameters were calculated: solubilisation and methane production increase.

The solubilisation factor (SB) (Equation (1)) was calculated with respect to the particulate fraction of the chemical oxygen demand (TCOD-SCOD), in contrast to the most of the references (Jung et al., 2015; Kim et al., 2015; Appels et al., 2010) that express this parameter with respect to the total TCOD. This proposed expression is more accurate as the particulate matter is the potentially hydrolysable fraction during the pre-treatment.

$$\%SB = \frac{(SCOD/TCOD)_{TH} - (SCOD/TCOD)_0}{((TCOD - SCOD)/TCOD)_0} \times 100 \quad (1)$$

The specific methane production was evaluated according to Equation (2):

$$CH_4 = \frac{mLCH_4}{gVS_{fed}}, \text{ at standard conditions (0 °C, 1 atm)} \quad (2)$$

And the performance of the digestion was calculated by comparing the CH<sub>4</sub> values obtained for the treated samples with respect to the untreated WAS, calculating the increase in methane production (Equation (3)):

$$\%CH_4 = \frac{(CH_4)_{TH} - (CH_4)_0}{(CH_4)_0} \times 100 \quad (3)$$

### 2.5. Experimental design

In order to statistically and mathematically determine the optimal conditions of the key operational conditions and the effects of their interactions on the global efficiency of the process, RSM with Box-Behnken experimental Design BBD (Benito-Roman et al., 2013; Jung et al., 2015; Sarat Chandra et al., 2014) was used in this work.

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