 

# Influence of the operating conditions of the intermediate thermal hydrolysis on the energetic efficiency of the sludge treatment process.

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## Abstract

The application of steam explosion between two stages of anaerobic digestion may improve energy recovery from sludge while increasing organic matter removal. The influence of the operating conditions of the thermal process: temperature (130 - 210 $^{o}$C), retention time (5 – 45 min) and TS concentration (5.4 – 10.8%), on the efficiency of VS removal, the biochemical methane potential of hydrolysed sludge and the kinetic constant of the degradation were evaluated using a Taguchi design. Increasing temperature and time increased the removal of VS and the potential of methane production but the kinetic constant was higher at lower temperatures. An optimal operating scheme was found at 170 $^{o}$C (6 barg), 25 min at the greatest TS concentration in the feeding. Under such conditions, the thermal energy obtained from biogas combustion in a CHP covered the requirements for vapour generation and a profit of 3.54 € $m^{-3}$ of sludge was estimated.

**Keywords**

High solids digestion, intermediate thermal hydrolysis, sludge treatment, techno-economic analysis, wastewater treatment plant.

## 1. Introduction

In wastewater treatment plants (WWTP), the most widely used technique is the activated sludge process, where aerobic microorganisms decompose organic matter and large amount of sludge is generated, accumulating most of the eliminated contamination (~60% of inlet COD) (Metcalf et al., 2002). The high amount of organic matter in the sludge favours its treatment through anaerobic digestion (AD) which represents an economically attractive and environmentally friendly process for the valorisation of an organic waste into electrical and thermal energy (Zhen et al., 2017).

During AD of sludge, the microbial decomposition of complex organic matter results in the production of biogas, mainly composed of methane, carbon dioxide and several pollutants in a lower extent, while destroying pathogens and eliminating odours. In spite of all the proven environmental and economic advantages of the process (Speece, 2008; Mirmasoumi et al., 2018), the digestion of solid wastes such as sludge has efficiency limitations in the removal of organic matter, with a volatile solids (VS) removal rate of around 45-50%. In order to enhance the hydrolysis of organic matter and to increase its subsequent degradation to biogas and obtain a more favourable energetic balance, several pre-treatment techniques have been applied (Gonzalez et al., 2018; Carrére et al., 2010; Pérez-Elvira et al., 2006). The efficiency, convenience and technology readiness level (TRL) of the different alternatives can be found elsewhere (Wu et al., 2020), and the choice of thermal hydrolysis (TH) as the most promising option for sludge pre-treatment prior to anaerobic digestion is justified, based on low energy requirements, a positive overall energy balance in comparison with the conventional AD (Cano et al., 2015; Carrère et al., 2010), and availability at commercial scale (Barber, 2016).

In the TH process the sludge is heated to 130-180 °C for about 20-40 minutes at the corresponding vapor pressure of water. While most of the publications evaluate and agree on these ranges of operation conditions, very few papers extrapolate pressure-temperature-time values to energetic-economic results, and just analyse sludge solubilisation, biogas production and organic solids removal. From this last point of view, it has been shown that the process is capable of partly solubilizing complex organic matter and disintegrating the biological cells, thus facilitating its subsequent digestion and reducing the viscosity (Chen et al., 2019; Kepp et al., 2000). Regarding operation conditions optimisation, the greatest effect on digestibility occurs at a temperature of approximately 175 °C and that the AD of thermally pre-treated sludge at that temperature results in a 60% to 70% increase in methane production compared to non-pre-treated sludge (Barber, 2016; Pilli et al., 2015), generally accepting that the sludge does not need to be treated for more than 30 min (Mirmasoumi et al., 2018).

Recently, the application of TH after a stage of anaerobic digestion and prior to a second digestion step gained attention as an alternative to the pre-treatment (Fernández-Polanco and Tatsumi, 2016), named intermediate thermal hydrolysis (ITH) . The main motivation was the reported increase in the global methane production of the sludge treatment process (90 ºC and NaOH addition) between two AD stages (Campo et al., 2018) and it has been identified that the thickening stages play a key role to achieve the thermal self-sufficiency of the process (Rus et al., 2017). Also regarding self-sufficiency, total solids (TS) concentration has shown to be a key parameter in order to reduce the sensible heat necessary to maintain the temperature of the digestion process; at 70% thermal exchange efficiency a minimum of 6% TS was required and increased to 8-9% when a efficiency of 50% was considered (Ruffino et al., 2019). For the ITH scheme, several aspects have been reported (sludge nature and operation conditions influence), while others are scarcely reported (optimization and energetic-economic implications). Nevertheless, it was recently found that an attractive payback can be expected with the implementation of thermal pre- or inter-treatments in large WWTP (Fernández-Polanco et al., 2021).

Regarding the nature of the sludge feeding in the TH-AD scheme, for waste activated sludge higher overall solids removal and biogas productivity have been observed when the intermediate treatment is purely thermal compared to the application of hydrolysis as a pre-treatment (Bjerg-Nielsen et al., 2018; Ortega-Martinez et al., 2016). On the contrary, the application of ITH hydrolysis exclusively to the primary sludge exhibited no significant differences in terms of methane production with respect to single digestion or TH pre-treatment (Yuan et al., 2019), as well as no significant increase in methane yield was observed when ITH was applied to sludge composed mainly of primary sludge (76%) (Liu et al., 2021), indicating that the intermediate treatment particularly favours the degradation of the secondary sludge. In fact, the most favourable scenarios for the energy optimization of the process were those in which the intermediate hydrolysis process was applied only to secondary sludge (Ruffino et al., 2019).

Thermal operation requires higher temperatures (> 130 $^{o}$C) than alkaline hydrolysis to obtain significant improvements in solids removal efficiency and biogas productivity but allows avoiding the use of chemical reagents. The intermediate process at temperatures of 170 $^{o}$C during 20-30 min (as well as hydrolysis as a pre-treatment) has shown a better performance compared to the pre-treatment (Díaz et al., 2020) at the same operating conditions. In contrast, similar methane yields were found in pre- and inter-treatments, with the only benefit of a greater VS removal efficiency (Zhang et al., 2021). The influence of temperature and retention time of ITH, particularly of the latter (Bjerg-Nielsen et al., 2018), is ambiguous along with the uncertainty of the efficiency required in the thickening stage, showing the lack of global optimization of the operation. Most of the reports on parameters assessment or optimization point to a higher influence of temperature on sludge solubilisation and methane production, being time less significant (Zhou et al., 2011). A temperature limit is generally identified due to the formation of recalcitrant or inhibitory compounds when TH is applied as a pre-treatment for mixed sludge (Lu et al., 2018; Neyens and Baeyens,

2003) but this effect is not reported for digested sludge. Methane production rates are also assessed for mixed sludge pre-treatment, but not for digested sludge (Razavi et al., 2019; Zhou et al., 2021). Finally, reports on the energetic and economic implications of variations on the operation temperature-time conditions are void, just extrapolated from TH pre-treatment schemes that depending on the efficiency of energy recovery in combined heat and power systems, it is essential to reach a solids concentration (TS) above 8% in order to reduce the sensible heat necessary to maintain the temperature of the digestion process, although the implication of the concentration of sludge on process performance is scarcely reported.

Then, this study is aimed at optimizing the operating conditions of Intermediate Thermal Hydrolysis (temperature, time and TS concentration) in the sludge line of a WWTP to maximize process efficiency and to assess the energetic and economic implications of different hydrolysis operation conditions when being carried out in the assessed two-stages AD scheme.

## 2. Materials and methods

The implementation of ITH in the sludge line was assessed according to the following scenario (Figure 1): Fresh sludge (the mixture of primary and secondary sludge) is firstly thickened and anaerobically digested. Secondly, digested sludge is thickened again before it is thermally treated in ITH unit by steam explosion. Finally, hydrolysed sludge is treated in a second digestion stage before it is, finally, dewatered producing the biosolid.

## 2.1. Experimental design

Taguchi's experimental design method was employed and implemented in Statgraphics Centurion 18 software. A combined array was selected as a model of design in order to include controllable variables as well as noise variables and their interactions, obtaining, only, the average and the variance of the controllable ones, with $\sigma^2 = 0$ as the objective (Montgomery, 2006).

Target responses were, the maximum methane productivity ($mLCH_4\ gVS^{-1}$) from biochemical methane potential (BMP) tests, VS removal efficiency (%) during BMP, VS losses in vapours released during steam explosion and the kinetic constant ($k_H$) from BMP data adjustment to a first order kinetics (equation 1) as reported elsewhere (Díaz et al., 2011),

$$P(t) = P_\infty \cdot [1- \exp(k_H \cdot t)] \qquad \text{(equation 1)}$$

where P(t) is the production of methane (cumulative) ($mLCH_4\ gVS^{-1}$), $P_\infty$ the biochemical methane potential ($mLCH_4\ gVS^{-1}$), $k_H$ the kinetic constant ($d^{-1}$) and t the elapsed time (d).

Three influencing factors were selected. Two controllable factors of the process: temperature (T) and time (t); and an uncontrollable or noise factor: VS (%w.), because mixed thickened sludge received from the water treatment plant varied and it was not possible to apply the same control as in the case of temperature and time during the

TH process. Three levels were selected for each variable, covering the range from 130 to 210 $^{o}$C in temperature, from 5 to 45 min in time and from 3.1 to 6.5% in VS, which corresponded to 5.4 - 10.8% in TS.

An orthogonal Taguchi's array (OA) $L_9$ ($3^4$) type was used. Two replicates were added per set of levels as three BMP test were made from each hydrolysis batch. In this way, 9 data blocks were extracted, giving rise to 27 runs (Table 1). An analysis of variance (ANOVA) was carried out, providing the necessary information to evaluate the effect of experimental factors individually, as well as how their interactions affected the response variables of the process.

## 2.2. Sludge sampling and Inoculum

The anaerobic sludge used for BMP in this study was collected from the Municipal Wastewater Treatment Plant (WWTP) of Valladolid (Spain). The main characteristics of the anaerobic sludge were: TS and VS of 2.20 % (w.) and 1.28% respectively, total Kjeldahl nitrogen (TKN) of 2.2 g $L^{-1}$, N-$NH_4^+$ = 1.0 g $L^{-1}$, pH = 7.46, and a soluble COD of 0.9 g $L^{-1}$. Samples were stored at 4°C prior to use for a maximum of 48h and employed as inoculum for BMP determination. Besides that, the anaerobic sludge was also concentrated in a Thermo Sorvall Legend RT+ Refrigerated Benchtop Centrifuge (Madrid, Spain) to attain the desired TS values in the feed of the thermal process (Table 1).

## 2.3. Experimental set-up

### 2.3.1. Thermal hydrolysis procedure

The concentrated sludge was thermally pre-treated at the TH process lab-scale plant. The plant employed for steam explosion is described elsewhere (Díaz et al. 2020). The sludge was manually fed to the reactor and a saturated steam was supplied to maintain the pre-set temperature during a given operating time. Finally, the hydrolysed sludge was suddenly decompressed (steam explosion effect) when it reached the pre-set time. The TH process was operated at different temperatures in a range of 130 –210°C and reaction times between 5 and 45 min according to Table 1 and a sludge sample of 500 mg was added in each experiment.

### 2.3.2. Biochemical Methane Potential Tests

The effect of ITH on the biodegradability of substrate and its ultimate biogas yield was assessed through BMP. Tests were performed in triplicate using 160 mL serum bottles with a working volume of 100 mL filled with 40 mL of inoculum, the corresponding amount of substrate corresponding to a substrate to inoculum ratio of 0.5 gVS $gVS^{-1}$ and a nutritive media. Micronutrients, macronutrients (Angelidaki et al., 2009) and buffer ($NaHCO_3$) were added to the nutritive media to ensure adequate conditions for anaerobic microorganisms. The inoculum was degassed during 72h at 35°C as recommended elsewhere (Angelidaki et al., 2009; Sapkaite et al., 2017). The initial VS in the 27 flasks was 0.766 % (w.) after inoculum, substrate and media addition. The bottles were incubated in a rotatory shaker at 35 °C and at agitation speed of 150 rpm.

Methane production was measured periodically through a manometric method and the composition of the biogas was analysed by GC_TCD. Tests were stopped when the daily methane production was lower than 1% of the cumulative methane production for three consecutive days (Holliger et al., 2016). Additional information on the methodology and preparation of BMP followed can be found in (Donoso-Bravo et al., 2011).

## 2.4. Analytical methods

The pH and the concentrations of TS, VS, COD, Total Kjeldahl Nitrogen (TKN) and N-$NH_4^+$ were determined according to standard methods (APHA, 2012). The methane concentration in the biogas produced during BMP tests was analysed by sampling 100 µL and subsequent injection in GC-TCD following the methodology reported in (Díaz et al., 2015).

## 2.5. Calculations

### 2.5.1. Regression models and coefficients

The regression coefficients of a 2-factors interaction model were calculated to fit equation 2 in order to estimate the different parameters (P), namely: VS removal during tests (%), BMP (mL$CH_4$ $gVS^{-1}$), $k_H$ ($d^{-1}$) and VS losses in vapours of steam explosion at the operating conditions tested.

$$P = k + A \cdot T + B \cdot t + C \cdot TS + AB \cdot T \cdot t + AC \cdot T \cdot TS + BC \cdot t \cdot TS \qquad \text{(Equation 2)}$$

where P is the estimated parameter and k the constant, A the coefficient for temperature, B the coefficient for time, C the coefficient for initial TS, AB the coefficient for temperature-time interaction, AC the coefficient for temperature - TS interaction and BC the coefficient for time - TS interaction. The independent variables are temperature (T, ºC), time (t, min) and TS (%w.). An analysis of variance (ANOVA) was carried out to determine those regression coefficients with a statistically significant effect (p-value < 0.05) on every parameter.

**2.5.2. Mass and energy balances to the thermal hydrolysis unit**

The values of VS removal during tests, BMP and VS losses were used to perform mass and energy balances to the TH unit and the second digestion stage, according to Díaz et al. (2020), to analyse the feasibility of the ITH process in the different pre-treatment temperature scenarios, as both the energy demand and energy recovery (biogas production) vary with the operation temperature.

Thermodynamic properties and mass balances were applied to calculate the fresh steam demand, translated into an energy value with the corresponding vapour enthalpy. The energy recovery was calculated considering a typical combined heat and power system for heat and electricity generation (38%, 48% and 19% electric, thermal and exhaust gases efficiency respectively). From the biogas production results experimentally obtained (expressed in $m^3$ biogas/ $m^3$ sludge), electric power and thermal recovery ($W/m^3$ sludge) were calculated for each operation scheme (130, 170, 210ºC). The thermal energy recovered from exhaust gases was compared in each case to the thermal requirement to generate the steam needed in order to assess thermal self-sustainability or, in case of non-sufficiency, to calculate the fraction of biogas to scarify in a boiler to cover the thermal demand.

The results of energy demand and energy recovery were expressed per unit of fresh sludge processed. For the economic analysis, the mass balances for sludge generation and associated calculations were performed according to Pérez-Elvira & Fdz-Polanco (2019). For the translation of energy recovery income from biogas and biosolids disposal cost, market prices were considered: 0.10 € $kWh^{-1}$ for power price and 15 € $ton^{-1}$ for sludge management cost.

# 3. Results and discussion

## 3.1. The effect of ITH on the efficiency of solids removal in BMP tests.

Organic matter (VS) removal during BMP tests increased as more severe operating conditions (higher heating temperature and longer operation time) were applied. Across the entire applied range of time and temperature conditions, higher VS removal efficiency was observed as temperature and operating time increased (Figure 3a). Under the less severe conditions (130 °C, 5 min) a minimum value of VS removal was found (21.7%), while this efficiency increased gradually as temperature and time increased until an observed maximum of 32.3% (210 °C, 45 min).

Analysing the effect of each of the parameters on VS removal, it was observed that temperature ($p < 0.001$) and time ($p = 0.007$) presented a significant impact ($p < 0.05$)

while the effect of the initial TS concentration ($p = 0.144$) and the interactions among effects are below the significance level (Figure 4a). By increasing both the temperature and the time, a positive effect on the elimination of VS was observed with a similar standardized effect of both parameters in contrast to Zhou et al. (2021), where increasing the severity of the TH to the maximum (60 min and 180ºC) of waste activated sludge led to a lower methane yield and, consequently, lower VS removal during the AD stage. Considering the treatment scheme of Figure 2 and the results of VS removal during the tests, the application of ITH and two stages of AD would result in an increase in the overall removal of VS between 55.8% and 61.1% in comparison to VS removal during the first AD stage (45%, reported in the WWTP). In this regard, Liu et al., (2021) also found an increase from 39.2% VS removal in one stage AD to 57.7% when ITH was applied between two digestion stages. A reduction in VS content in final sludge can reduce the final biosolid generation after dewatering. Although final dewatering is poorly documented, an enhancement in dewaterability from 20% dry solids (DS) in conventional digestion up to 43% DS for post-AD thermal treatment has been reported (Svennevik et al., 2019; Svensson et al., 2018).

The statistical model used, considering only the individual effects of temperature and time, explained more than 79% of the variability observed in the removal efficiency (Table 2).

### 3.2. Influence of ITH on subsequent AD. Biochemical methane potential of the sludge after ITH.

As for VS removal, the methanogenic potential of the hydrolysed sludge increased with more severe operating conditions; the increase in temperature and hydrolysis time resulted in higher methane productivity (Figure 3b) throughout the range tested in contrast to previous studies where TH was applied as a pre-treatment, where the most severe conditions reduced the BMP (Zhou et al., 2021). The effect of the operating temperature of ITH was previously reported in Zhang et al. (2021), where methane yield increased with T (90 - 155 $^{o}$C) but the yield was similar or lower when T was increased at 185 $^{o}$C.

The observed potential increased from 175 $mLCH_4$ $gVS^{-1}$ at 130 $^{o}$C and 5 min (Figure 5a), to 223 $mLCH_4$ $gVS^{-1}$ at 170$^{o}$C and 25 min (Figure 5b) and to a maximum of 248 $mLCH_4$ $gVS^{-1}$ at 210 $^{o}$C and 45 min (Figure 5c). These values are slightly below those observed for TH as a pre-treatment before AD (Donoso-Bravo et al., 2011) (230 – 300 $mLCH_4$ $gVS^{-1}$). It should be noted that the most easily biodegradable organic matter was already consumed during first digestion stage in the case of this study, in which digested sludge was the substrate of the thermal process. Literature comparing TH pre-treatment and inter-treatment shows notable differences among studies; a reported increased in methane productivity of 29% was detected in the inter-treatment scheme of waste activated sludge in comparison to 9% attributed to the pre-treatment at 170 $^{o}$C (Nielsen et al., 2011). In contrast, no statistical significant difference was found in methane yield between a scheme of TH as a pre-treatment or an inter-treatment (Liu et al., 2021) when sludge submitted to TH was 76% primary sludge and 24% waste activated sludge. The greater the contribution of waste activated sludge the lower the biodegradability of sludge and, therefore, the higher the potential of TH to increase the availability of organic matter for methane production in AD.

Regarding the effect of each parameter on methane productivity, it was observed that there are no significant interactions ($\alpha > 0.05$) between the parameters and that only temperature ($p < 0.001$) and time ($p < 0.001$) have a relevant level of significance in the model (Figure 4b) while the effect of TS concentration presented a p-value of 0.215. The order of magnitude of the standardized effects is similar for time and temperature when increasing BMP as for VS removal in BMP tests.

The model, including only the individual effects of temperature and time, is capable of explaining more than 82% of the variability in BMP in the range studied (Table 2).

### 3.3. The kinetics. Effect on $k_H$

The kinetic constant, obtained by first order adjustment (Equation 1) of the methane production data over time during the BMP tests, showed its overall maximum value (0.315 $d^{-1}$) at low temperature and long times (130 ºC, 45 min). However, the next highest local maximum value (0.288 $d^{-1}$) was observed at 170 ºC and 25 min. Also, at 170 ºC, the constant increased at 25 min with respect to 5 min but decreased later when 45 min were applied. Interestingly, the constant always decreased when time increased at 210 ºC in agreement with Zhou et al. (2021) and Razavi et al. (2019), where a slower biodegradation was observed after a threshold temperature. Also in this direction, the kinetic constant of carbohydrates degradation increased when TH was applied, whether as pre-treatment or inter-treatment in comparison to conventional AD (Shana et al., 2013). The least favourable conditions appeared when low temperatures were applied at short operating times (130 $^{o}$C, 5 min), as would be expected due to inefficiency of the treatment, but also at elevated temperatures for a long time (210 $^{o}$C, 45 min), both with values of $k_H$ below 0.2 $d^{-1}$. Summarizing the results, when a higher temperature was used the maximum kinetic constant was found at shorter times. The maximum kinetic constant calculated at 130, 170, and 210 $^{o}$C was observed at 45, 25 and 5 min respectively (Figure 3c).

The analysis of the effect of each parameter on the apparent kinetic constant revealed that the TS concentration is the parameter with the highest standardized positive effect on $k_H$ with $p < 0.001$. The negative interaction between temperature - time ($p < 0.001$) and the positive interaction between time - TS ($p = 0.011$) were also relevant (Figure 4c). In this case, the effect of temperature ($p = 0.671$) and operating time ($p = 0.064$) were not significant compared to its interaction. The adjusted model, including the individual effect of VS concentration and the temperature - time and time - TS, explained in this case more than 89% of the variability in the values of the constant (Table 2). Therefore, the value of the kinetic constant behaved differently than VS removal during tests and BMP, which increased when applying higher temperatures and times. This fact leads to the necessity of finding a compromise solution with a view to optimize the process; severe conditions that increased VS removal and the methanogenic potential could result in slow kinetics, causing prohibitive capital expenses (reaction volume) to obtain a sufficient methane productivity that justifies the energy invested in ITH.

### 3.4. Quantification of VS losses in vapours of steam explosion

The batch process of thermal hydrolysis releases a stream of vapour (Figure 2) after steam explosion. The quantification of VS losses in vapours is scarce in literature but allows the calculation of mass balances to the continuous operation where released vapours are recirculated to thermal hydrolysis unit. The amount of VS lost in vapor was between 4.0% and 33.3% in the conditions studied and VS losses increased with the severity of the treatment (Figure 3d). The individual effects of temperature ($p < 0.001$), time ($p < 0.001$) and the interactions temperature - TS concentration ($p = 0.003$) and time - TS concentration ($p < 0.001$) were significant and positive indicating that VS losses increased with temperature and time and that this effect was higher when higher TS concentrations were tested. The standardized effect of temperature was more than double than that of time (Figure 4d); therefore, increasing the operating temperature showed a more powerful effect on VS losses than increasing the operating time.

The model considering the individual effects of temperature, time and the interactions temperature - TS concentration and time - TS concentration explained more than 97% of the variability observed in VS losses (Table 2).

### 3.5. Feasibility of ITH. Comparative study of the energy balances.

#### 3.5.1. Energy requirements for ITH implementation

The energy requirement of a TH unit is not a constant value and depends on several parameters. First, the technology is a key aspect, as the heating mechanism and the energy recovery possibilities makes a huge difference (Fernández-Polanco and Tatsumi, 2016). In this paper, the scheme considered is the one represented in Figure 2.

The ratio of live steam needed per unit of sludge processed was calculated in each of the three operation temperatures considered: 130 $^oC$, 170 $^oC$ and 220 $^oC$. Table 3 summarizes the input (operation pressure) and the main outputs (results) of the simulations performed for each scenario. The energy required was expressed per unit ($m^3$) of sludge treated, and the obtained values are consistent with those reported for full-scale installations (Theodolou et al., 2016).

It can be observed that the higher the operating temperature, the higher the fresh steam needed, although the relationship is not linear, as the vapours recirculation play a key role. In fact, while for lowest reactor temperature all the vapours are recirculated to the preheating tank (0% vapour losses), for high temperature operation (scenario C) more than half of the vapours coming from the flash correspond to losses, and therefore their calorific potential is wasted and the need of fresh steam nearly doubles with respect to scenarios A and B (4.42 W $m^{-3}$ sludge with respect to 2-2.3 W $m^{-3}$ sludge).

### 3.5.2. Biogas production, energy recovery from organic matter and overall energetic efficiency

The energetic feasibility of a thermal process such as TH relies on the feasibility of covering the energy requirements to run the pre-treatment with the thermal energy generated from biogas burning in a combined heat and power system that also generates electric profit. If the thermal energy recovered from exhaust gases is enough to generate the steam needed, the process will be energetically self-sufficient. Otherwise, a portion of the biogas should be sacrificed to cover the remaining demand. Table 4 presents the results obtained for heat and electricity generation in a typical combined heat and power system, for the three operation temperatures considered: 130 $^{o}$C, 170 $^{o}$C and 220 $^{o}$C. To simplify the discussion, only the results for 25 minutes operation were considered. The input corresponds to the results of biogas obtained in the experimental study, and the outputs summarize the most representative parameters of the balance, including thermal and electric energy. The results were again expressed per unit ($m^3$) of sludge treated, considering 10% TS concentration in the sludge feeding to the digester.

From Table 4 it is evident that while the highest TH is optimum from the point of view of biogas generation, it is however worst in the global energetic analysis, as nearly 23% of the biogas produced needs to be derived from the CHP to a boiler to produce the thermal energy needed for the pre-treatment. The pre-treatment at 130 $^{o}$C and 170 $^{o}$C (scenarios A and B) present a positive global thermal balance, which means that all the energy demand to run the hydrolysis is covered with the energy recovered from the exhaust gases in the CHP, and therefore all the biogas is profitable for electric energy generation. The results show an optimum energy balance for 170 ºC thermal pre-treatment, energetically self-sufficiency and producing 4.5 watts of electricity per cubic meter of sludge processed.

### 3.6. Economic perspectives of the process including ITH

Mass balances were performed in parallel with the energetic efficiency analysis for the complete ITH scheme, from the feeding to biogas (both digesters) and digestate. The results from the mass and energy balances were translated into economic figures in order to quantify the economic significance of the analysis. The case study considered was a municipal WWTP treating the sludge produced by half a million population equivalent city (24 ton $h^{-1}$ sludge, 1 ton TS $h^{-1}$), considering the implementation of a TH unit to hydrolyse the sludge coming from a first digestion step (11.1 $m^3$ biogas $m^{-3}$ sludge) (Díaz et al., 2020) and prior to the second digestion step studied in this paper. Two economic items were considered as most significant: the benefit of selling electricity, and the cost of biosolids disposal. Table 5 summarizes the results, expressed per unit of sludge processed as annual cost or benefit.

In short, a lower income was obtained for the highest operating temperature, and the highest benefit for 170 ºC hydrolysis. The cost of biosolids management is consistent with the biosolids removal results obtained: as the temperature increases, the solids removal decreases, and therefore the final biosolids volume and disposal cost decreases. Considering both contributions, and as expected from the higher weigh of the electric energy, the resulting values for total benefit show and optimum for scenario B: 170 °C thermal pre-treatment, with an annual income of over 740 k€. From capex market prices and considering a payback period of 10 years for the thermal unit, the annual benefit can still be estimated in around 560 k€, which is an attractive economic perspective. While the is no published report linking parameters optimization with economic profit for ITH schemes, Fernández-Polanco et al. (2021) also reported the inter-treatment scheme as an energy self-sufficient configuration which yields the best operating numbers, estimating annual savings over 800k€ for a 1 million population calculation basis.

## 4. Conclusions

Digestion efficiency increased with increasing treatment severity (operating temperature and time, the first being more influential) while the digestion kinetics tended to decrease. With this compromise, an optimum was identified at 170 °C and 25 min thermal hydrolysis fed at the highest solids concentration tested (11% TS). At those conditions, an optimum energy balance for 170 °C was found, achieving self-sufficiency of the process at 10% TS concentration in the feeding to the TH. The translation of electric energy income and reduction of biosolids disposal cost into economic profit was estimated at 3.54 € $m^{-3}$ of sludge for large WWTP.


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**Figure captions**

Figure 1: Block diagram of the sludge treatment process including ITH.

Figure 2: Thermal hydrolysis simulation scheme.

Figure 3. Surface plots of VS removal during BMP tests (a), biochemical methane potential (b), kinetic constant (c), and VS loss during TH (d) at initial TS concentration of 10%w. as a function of temperature and time.

Figure 4. Pareto diagrams of standardized effects of the independent variables on VS removal during tests (a), BMP (b), kinetic constant (c) and VS loss during TH (d) for $\alpha = 0.05$ (red line).

Figure 5. Evolution of experimental methane production during BMP tests (dots) and first order fitting (lines) at 130ºC (a), 170ºC (b) and 210 ºC (c).

**Table captions**

Table 1. Design matrix of sequential conditions for the TH process and data obtained for every run of the experimental design.

Table 2. Coefficients of the adjusted model (Equation 2). N.S.: Not significant ($\alpha < 0.05$).

Table 3: Values considered for TH energy demand calculations and results obtained.

Table 4: Values considered for energy calculations and results obtained.

Table 5: Economic analysis.

Figure 1 Click here to access/download;Figure;Figure 1.docx

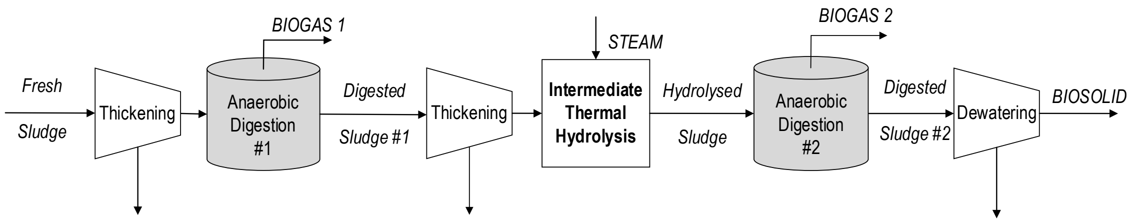


Figure 1: Block diagram of the sludge treatment process including ITH.

Figure 2

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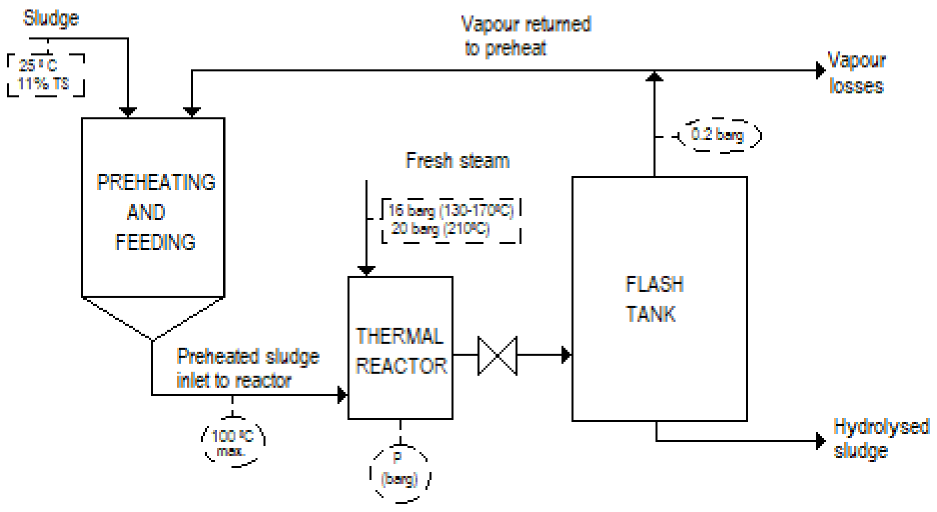
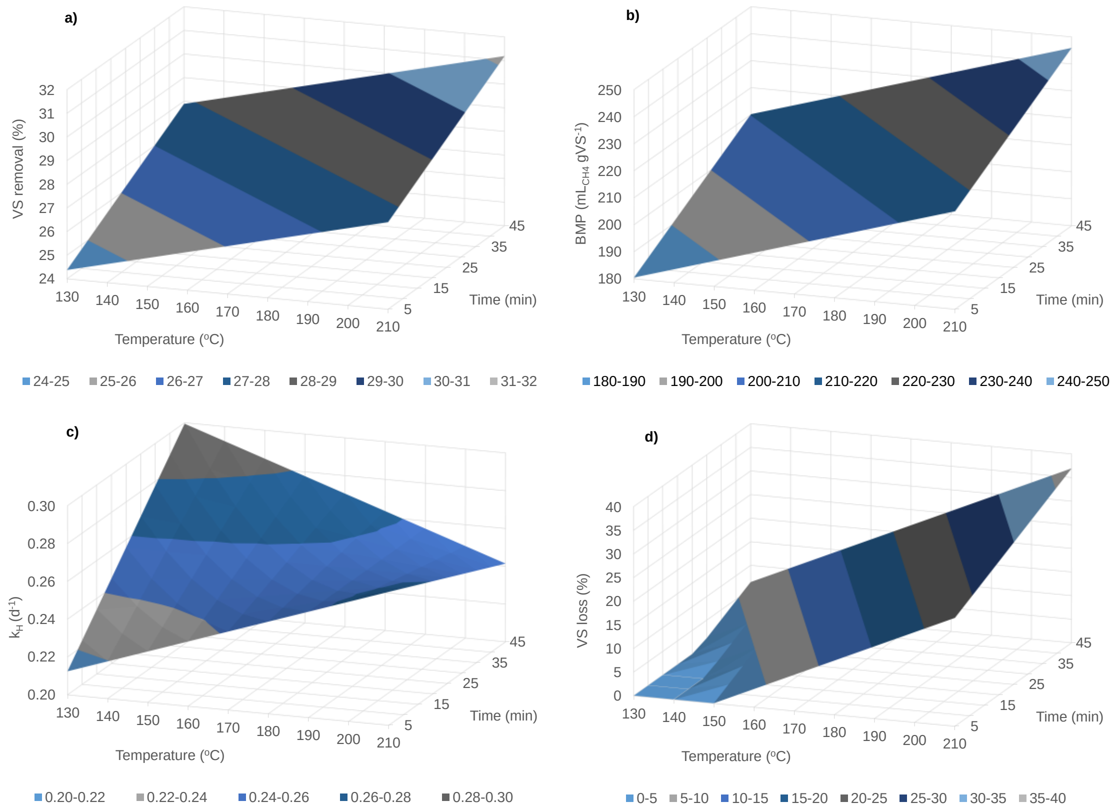



Figure 2: Thermal hydrolysis simulation scheme.

Figure 3 Click here to access/download;Figure;Figure 3r.docx

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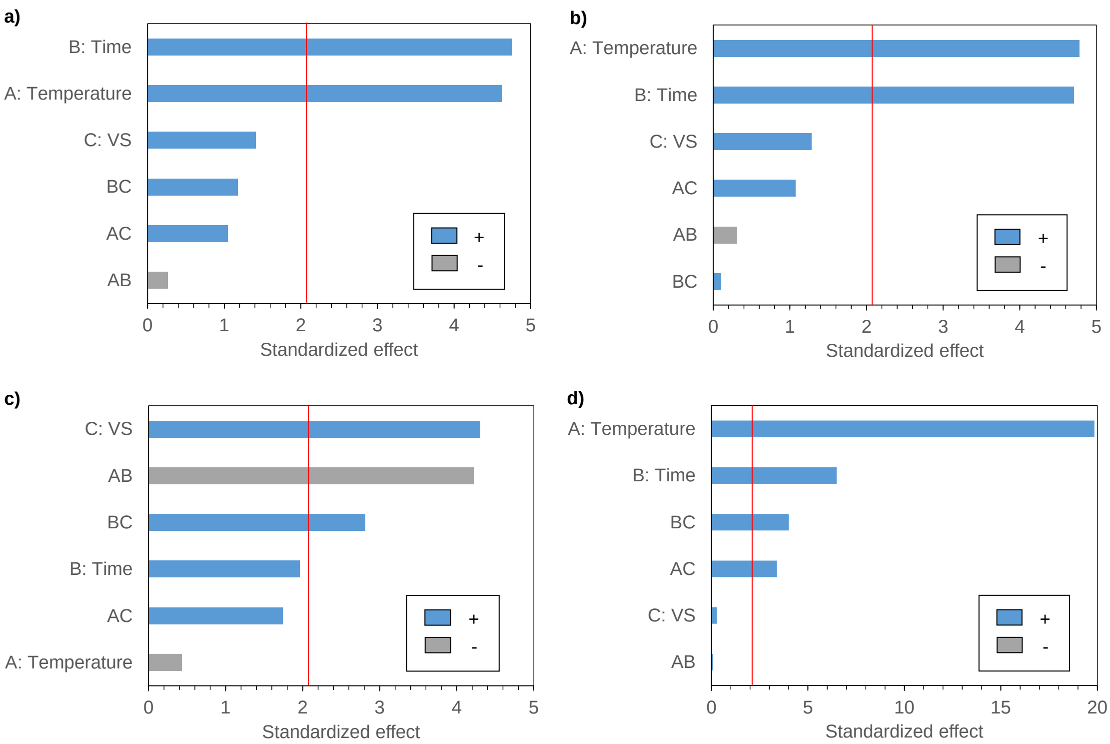


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Figure 5 

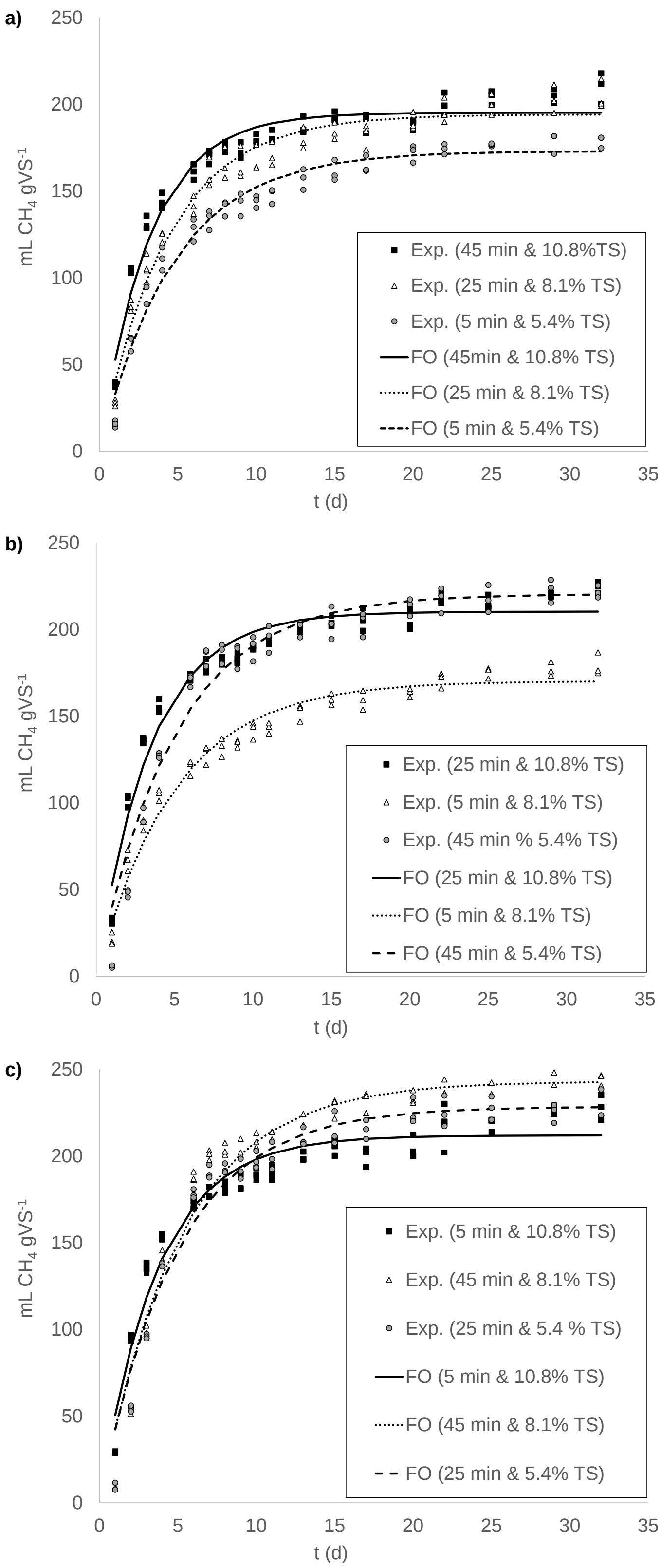


Figure 5. Evolution of experimental methane production during BMP tests (dots) and first order fitting (lines) at 130ºC (a), 170ºC (b) and 210 ºC (c).

| | Operating conditions | | Feed | | Hydrolyzed | | | BMP tests | |
|---|---|---|---|---|---|---|---|---|---|
| *Run #* | *T* | *t* | *TS* | *VS* | *VS* | *mass* | *Final VS* | *BMP* | *kH* |
| | *ºC* | *min* | *% (w.)* | *% (w.)* | *% (w.)* | *kg* | *% (w.)* | *mL $CH_4$ $gVS^{-1}$* | *$d^{-1}$* |
| 1 | 130 | 45 | 10.8 | 6.48 | 1.511 | 2.00 | 0.548 | 212 | 0.315 |
| 2 | 130 | 45 | 10.8 | 6.48 | 1.511 | 2.00 | 0.549 | 200 | 0.315 |
| 3 | 130 | 45 | 10.8 | 6.48 | 1.511 | 2.00 | 0.547 | 218 | 0.315 |
| 4 | 170 | 25 | 10.8 | 6.48 | 1.782 | 1.60 | 0.548 | 221 | 0.288 |
| 5 | 170 | 25 | 10.8 | 6.48 | 1.782 | 1.60 | 0.544 | 225 | 0.288 |
| 6 | 170 | 25 | 10.8 | 6.48 | 1.782 | 1.60 | 0.547 | 227 | 0.288 |
| 7 | 210 | 5 | 10.8 | 6.48 | 1.751 | 1.42 | 0.547 | 228 | 0.273 |
| 8 | 210 | 5 | 10.8 | 6.48 | 1.751 | 1.42 | 0.546 | 221 | 0.273 |
| 9 | 210 | 5 | 10.8 | 6.48 | 1.751 | 1.42 | 0.548 | 235 | 0.273 |
| 10 | 130 | 25 | 8.10 | 4.70 | 1.672 | 1.34 | 0.571 | 215 | 0.234 |
| 11 | 130 | 25 | 8.10 | 4.70 | 1.672 | 1.34 | 0.558 | 199 | 0.234 |
| 12 | 130 | 25 | 8.10 | 4.70 | 1.672 | 1.34 | 0.562 | 201 | 0.234 |
| 13 | 170 | 5 | 8.10 | 4.70 | 2.066 | 1.02 | 0.589 | 187 | 0.201 |
| 14 | 170 | 5 | 8.10 | 4.70 | 2.066 | 1.02 | 0.577 | 175 | 0.201 |
| 15 | 170 | 5 | 8.10 | 4.70 | 2.066 | 1.02 | 0.565 | 176 | 0.201 |
| 16 | 210 | 45 | 8.10 | 4.70 | 0.563 | 2.78 | 0.531 | 247 | 0.194 |
| 17 | 210 | 45 | 8.10 | 4.70 | 0.563 | 2.78 | 0.519 | 246 | 0.194 |
| 18 | 210 | 45 | 8.10 | 4.70 | 0.563 | 2.78 | 0.522 | 241 | 0.194 |
| 19 | 130 | 5 | 5.40 | 3.13 | 1.476 | 0.97 | 0.568 | 175 | 0.212 |
| 20 | 130 | 5 | 5.40 | 3.13 | 1.476 | 0.97 | 0.570 | 181 | 0.212 |
| 21 | 130 | 5 | 5.40 | 3.13 | 1.476 | 0.97 | 0.600 | 181 | 0.212 |
| 22 | 170 | 45 | 5.40 | 3.13 | 0.550 | 2.47 | 0.559 | 225 | 0.200 |
| 23 | 170 | 45 | 5.40 | 3.13 | 0.550 | 2.47 | 0.550 | 221 | 0.200 |
| 24 | 170 | 45 | 5.40 | 3.13 | 0.550 | 2.47 | 0.543 | 218 | 0.200 |
| 25 | 210 | 25 | 5.40 | 3.13 | 0.598 | 1.99 | 0.556 | 223 | 0.205 |
| 26 | 210 | 25 | 5.40 | 3.13 | 0.598 | 1.99 | 0.538 | 224 | 0.205 |
| 27 | 210 | 25 | 5.40 | 3.13 | 0.598 | 1.99 | 0.538 | 238 | 0.205 |

Table 1. Design matrix of sequential conditions for the TH process and data obtained for every run of the experimental design.

| | VS removal during tests (%) | BMP ($mLCH_4$ $gVS^{-1}$) | $k_H$ ($d^{-1}$) | VS loss in TH (%) |
|---|---|---|---|---|
| $R^2$ | 0.7919 | 0.8219 | 0.8909 | 0.9730 |
| k | $1.856 \cdot 10^{1}$ | $1.187 \cdot 10^{2}$ | $7.268 \cdot 10^{-2}$ | $1.229 \cdot 10^{1}$ |
| A: Temperature | $4.133 \cdot 10^{-2}$ | $4.470 \cdot 10^{-1}$ | $9.073 \cdot 10^{-4}$ | $7.024 \cdot 10^{-2}$ |
| B: Time | $8.774 \cdot 10^{-2}$ | $7.422 \cdot 10^{-1}$ | $3.083 \cdot 10^{-3}$ | $-3.822 \cdot 10^{-1}$ |
| C: TS | N.S. | N.S. | $1.093 \cdot 10^{-3}$ | -7.118 |
| AB | N.S. | N.S. | $-3.629 \cdot 10^{-5}$ | N.S. |
| AC | N.S. | N.S. | N.S. | $3.109 \cdot 10^{-2}$ |
| BC | N.S. | N.S. | $3.811 \cdot 10^{-4}$ | $7.331 \cdot 10^{-2}$ |

Table 2. Coefficients of the adjusted model (Equation 2). N.S.: Not significant ($\alpha < 0.05$).

 

| | INPUTS | | OUTPUTS | | |
|---|---|---|---|---|---|
| Scenario | Reactor pressure | Fresh steam pressure | Ratio of vapour losses (kg lost $kg^{-1}$ flashed) | Fresh steam needed (kg $kg^{-1}$ sludge) | Thermal energy required for TH (W $m^{-3}$ sludge) |
| A-130 °C | 2 barg | 16 barg | 0% | 0.142 | 2.03 |
| B-170 °C | 6 barg | 16 barg | 6% | 0.151 | 2.30 |
| C-210 °C | 19 barg | 20 barg | 53% | 0.294 | 4.42 |

Table 3: Values considered for TH energy demand calculations and results obtained.

 

| | INPUTS | OUTPUTS | | |
|---|---|---|---|---|
| Scenario | Biogas ($m^3$ $m^{-3}$ sludge) | Thermal energy balance (W $m^{-3}$ sludge) | % biogas destined to burning to cover thermal demand | Electric energy generated (W $m^{-3}$ sludge) |
| A-130 ºC | 4.4 | + 0.2 | 0.0% | 4.5 |
| B-170 ºC | 5.1 | + 0.1 | 0.0% | 4.8 |
| C-210 ºC | 5.2 | - 2.0 | 22.6% | 3.7 |

Table 4: Values considered for energy calculations and results obtained.

 

| | Electric energy income | | Biosolids disposal cost | | Total benefit | |
|---|---|---|---|---|---|---|
| Scenario | € $m^{-3}$ sludge | € $year^{-1}$ | € $m^{-3}$ sludge | € $year^{-1}$ | € $m^{-3}$ sludge | € $year^{-1}$ |
| A-130 °C | 3.98 | 835067 | 0.69 | 144922 | 3.29 | 690145 |
| B-170 °C | 4.18 | 877548 | 0.64 | 133623 | 3.54 | 743925 |
| C-210 °C | 3.25 | 683547 | 0.63 | 132274 | 2.62 | 551273 |

Table 5: Economic analysis.