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# Thermal Hydrolysis of Municipal sludge: Finding the Temperature Sweet Spot - A Review

Perrine Devos<sup>a, 1</sup>, Mathieu Haddad<sup>a, 1</sup>, H el ene Carr ere<sup>b, \*</sup>

<sup>a</sup> Suez Treatment Infrastructure, 183 avenue du 18 juin 1940 ; 92500 Rueil Malmaison, France

<sup>b</sup> INRAE, Univ Montpellier, LBE, 102, Avenue des Etangs, 11100 Narbonne France

<sup>1</sup> Joint first authors

\* **Corresponding author:**

E-mail address: helene.carrere@inrae.fr

## Abstract

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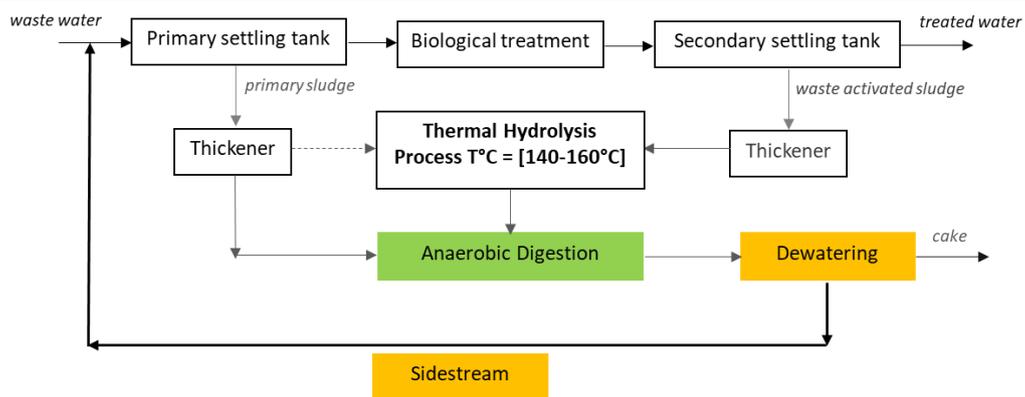
The increasing demand for a Thermal Hydrolysis Process (THP) to pretreat municipal sludge upstream Anaerobic Digestion (AD) opens the opportunity to further develop and optimise this technology. The optimal THP temperature remains unclear due to the production of refractory compounds at high temperature. A compilation of literature data was conducted to investigate the existence of a temperature sweet spot for the THP applied to municipal sludge. All related reports (n=43) were included. The THP temperature range impact was assessed in the range of 100°C – 200°C on 4 AD and dewatering performance indicators (CH<sub>4</sub> production, Volatile Solid Reduction (VSR), Dewaterability (DW) and filtrate quality). Other parameters potentially affecting the performance indicators were also considered. These parameters include the type of sewage sludge and operational conditions related to THP and AD. The impact of all parameters on performance indicators was evaluated with a Kruskal-Wallis statistical test. For THP temperature optimisation, a pairwise comparison, using a Wilcoxon test, was made. A temperature optimum in the [140-160]°C range was proposed. It seemed to minimize the production of refractory compounds, while maximising AD and dewatering performances. It is noteworthy that above 160°C, the concentration in refractory compounds and soluble COD increases sharply, thus leading to a potential deterioration of WWTP effluent quality.

## Keywords

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Sewage sludge; Anaerobic Digestion; Pretreatment; Thermal Hydrolysis Process; THP

# Graphical Abstract



**Optimal THP temperature range for an optimal outcome on both sludge and wastewater treatment lines**

## Statement of Novelty

Thermal Hydrolysis Process (THP) is a well-known process in the pretreatment of mesophilic Anaerobic Digestion (AD) used for enhancing biogas production and digested sludge dewaterability. However, although thermal hydrolysis parameters are usually optimised as a function of AD performance, (i.e. reduction in the amount of sludge and/or maximisation of biogas production), this type of pretreatment has other consequences, such as the increase in soluble COD in the anaerobic digestion effluent, that have rarely been taken into account. For the first time, the present review gathers and standardises all published quantitative data relative to the impact of THP on AD performance (biogas production and VS reduction), sludge dewaterability and on dewatered effluent quality. Firstly, the most impactful THP parameters, i.e. sludge type and temperature, are examined. Secondly, the optimal temperature range is determined considering AD performance, sludge dewaterability, as well as dewatered effluent quality. Despite the numerous articles that recommend 165-175°C as an optimal temperature range which is applied in full scale plants, this holistic approach reveals how THP operated within the 140-160°C temperature range favours the maintenance of good biogas production and volatile solid removal while minimizing the impact on rejected water quality. . These results should be useful in the current context where traditional wastewater treatment plants (WWTPs) are increasingly regarded as water resource recovery facilities (WRRFs). Indeed, the impact of a typical sidestream quality can be significant on water line carbon, nitrogen and phosphorus removal and recovery processes.

# List of Abbreviations

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- AD: Anaerobic Digestion
- BMP: Biochemical Methane Potential
- CH<sub>4</sub>: Methane
- DW: Dewatering
- COD: Chemical Oxygen Demand
- DON: Dissolved Organic Nitrogen
- DW: Dewatering
- HRT: Hydraulic Retention Time
- in : introduced
- N-NH<sub>4</sub>: Ammonium
- PI: Performance Indicators
- TAN: Total Ammonia Nitrogen
- TH: Thermal Hydrolysis
- THP: Thermal Hydrolysis Process
- TS: Total Solids
- VS in: Volatile Solids inlet
- VSR: Volatile Solids Reduction
- sCOD: soluble Chemical Oxygen Demand
- rDON: refractory Dissolved Organic Nitrogen
- TS: Total Solids
- UVA: Ultraviolet Absorbance
- WWTP: WasteWater Treatment Plant

## 1. Introduction

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The production of wastewater sludge is increasing worldwide due to the growing population. This increase is also the consequence of stricter nitrogen regulations that imply high sludge handling/disposal costs [1]. Anaerobic digestion (AD) is a key process to reduce sludge quantities and to recover energy. It is a mature biological process consisting of four steps: hydrolysis, acidogenesis, acetogenesis and methanogenesis. The first step of the process, hydrolysis, has been recognised as a rate-limiting step. It consists in the degradation of insoluble organic matter and macromolecular compounds into small and soluble compounds [2]. A slow and incomplete hydrolysis results in slow anaerobic degradation kinetics and high hydraulic retention times in the digestion process. This issue is found to be more pronounced for mesophilic digestion than for thermophilic systems. To overcome this bottleneck, extensive research has been carried out to test pretreatments upstream of the mesophilic digester [3]. The aim of sludge pretreatment is to destabilize the floc structure and solubilize intracellular compounds, thus allowing organic compounds to be more accessible to hydrolytic enzymes and microorganisms. The final expected result is an increase in both anaerobic digestion rates and its yield.

A number of articles have reviewed different sludge pretreatment technologies applied upstream to batch Biochemical Methane Potential (BMP) tests, lab-scale continuous or full-scale anaerobic digesters [2–6]. The different pretreatments found in the literature can be divided into four groups: thermal, chemical, mechanical and biological. Thermal Hydrolysis (TH) pretreatments include two subcategories: low and high temperature TH. Low temperature TH (<100°C) has most often been combined with

alkaline conditions [7]. Not requiring any chemical addition, it results in extensive retention times such as 72h [8], 24h [9], 10h [10] or 5h [7], which proves to be uneconomical at industrial scales. High temperature TH, operated at temperatures above 100°C, is commonly referred to as Thermal Hydrolysis Process (THP). Chemical pretreatment comprises acid or alkaline (thermal) hydrolysis and advanced oxidation methods [2]. In thermo-chemical hydrolysis methods, an acid or base is added to solubilise the sludge heated at moderate temperatures (<100°C). The most frequent studies on oxidative methods involve ozonation and peroxidation, based on the generation of hydroxyl (OH<sup>•</sup>) radicals which are extremely powerful oxidants [11]. Mechanical pretreatments include ultrasonification, microwave or electrokinetic disintegration, also known as pulse electric field and high pressure homogenisation. Biological pretreatments are essentially based on Temperature Phased Anaerobic Digestion (TPAD). TPAD includes a short (1 to 3 days) mesophilic (37-40°C) or thermophilic (50-70°C) pre-treatment stage applied prior to a conventional mesophilic anaerobic digestion [12]. Certain studies report other biological pretreatment opportunities, such as the addition of external enzymes [2] or microbial electrolysis cells [6], but these methods are still being investigated. Only a few of them have been transferred to full-scale WWTPs, including THP, ultrasounds, high pressure, electrokinetic and temperature-phased anaerobic digestion.

Thermal hydrolysis process is reported as the most widely developed technology [13]. Indeed, existing full-scale references have so far demonstrated its enhancement in sludge anaerobic digestion. It has proven to be energy efficient and economically sustainable [14, 15]. THP consists in heating the sludge to a temperature generally in the 140-180°C range with a treatment time span of 30 to 60 minutes, and under a pressure ranging between 6 and 11 bars. THP has been largely applied in municipal WWTP since the first full-scale plant in Hamar, Norway in 1995. Today, more than 82 THP full-scale references exist. These references are mainly divided amongst five technology suppliers: Cambi, Veolia, Sustec, Haarslev and Eliquo. Two configurations are possible for a THP: (1) partial lysis, when only the waste activated sludge is thermally pre-treated or (2) a full lysis, when the entire sludge stream undergoes thermal hydrolysis.

Table 1 presents the main THP technology suppliers in terms of operational characteristics and number of references at full scale. While most of these technologies use steam injection to heat the sludge, one technology uses indirect heating by steam circulation in heat exchangers. Most current full-scale plants operate in batch mode, although some technologies can be continuous or semi-continuous. They can also differ by the presence or absence of a flash system, which could account for a sudden release in pressure.

**Table 1: THP technology characteristics**

Technology	CambiTHP™	Biothelys™	Exelys™	HCHS	Turbotec®	Lysotherm®
<b>Supplier</b>	Cambi	Veolia	Veolia	Haarslev	Sustec	Eliquo
<b>Number of references</b>	61	7	7	3	2	2
<b>Process temperature (°C)</b>	160-180	165	165	150-170	140	140-170
<b>Flash System (quick pressure drop)</b>	Yes	Yes	No	Yes	No	No
<b>Operating mode</b>	Batch	Batch	Continuous	Semi continuous	Continuous	Continuous
<b>Pre-heating</b>	Steam recovery	Steam recovery	No pre-heating	Steam recovery	Direct mixing of fresh and hydrolysed sludge	Heat Exchanger
<b>Heating mode</b>	Direct heating (steam injection)					Indirect heating (Heat Exchanger)
<b>References</b>	[14]	[16]	[17]	[18]	[19]	[20]

The main impact of the thermal hydrolysis of sludge is the solubilisation of organic compounds, generally measured as the soluble Chemical Oxygen Demand (sCOD) [21]. However, the increase in methane potential or sludge biodegradability is limited to temperatures less than 200°C. In this temperature range, the increase in biodegradation is related to the transfer of particulate organic matter towards the soluble fraction which presents a higher rate of biodegradability than the solid fraction [5]. In this way, THP enhances anaerobic digestion as reported in [21–26] and entails a reduction of the Hydraulic Retention Time (HRT) in the digester [27]. The THP process does not favour an increase in the biodegradability of the remaining particulate fraction [21] but it results in the degradation of high molecular substances such as soluble microbial products and extracellular polymeric substances [28]. At high temperatures, THP leads to the formation of brown recalcitrant compounds in the soluble fraction [29]. They are generally attributed to Maillard reactions involving the conversion of carbohydrates and amino acids or proteins to melanoidins [30]. Their production prevents any further increase in sludge biodegradability even though the solubilisation yield is high. Thus, while increasing temperature increases sludge solubilisation [31], no increase in methane conversion is necessarily obtained [32]. A few recent studies

investigated the impact of THP temperature on soluble COD remaining in the digestate, either after Biochemical Methane Potential (BMP) tests [33] or after semi-continuous digestion [34]. They all led to the same result, namely an increase in residual soluble COD with an increase in temperature within the tested range (130-170°C). According to Zhang et al. [30], in addition to high temperature, the production of soluble organic nitrogen compounds, assimilated to melanoidins originating from Maillard reactions, is favoured by high pH values and is impacted by the presence of metallic ions. Lu et al. [35] provided a detailed characterisation of soluble COD after 172°C thermal hydrolysis and batch anaerobic digestion in BMP tests. The main high molecular weight compounds were humic substances while low molecular ones were aromatic and nitrogen compounds. High temperature TH also induces the production of colloids, which increase the turbidity of the final dewatered effluent [36].

In addition, it is noteworthy to recall how THP was first used to improve sludge dewatering. Neyens and Bayens (2003) [37] reported full scale processes during the 1960's, operating at 200-250°C, whereas Bougrier et al. (2008) [21] highlighted a minimal temperature of 150°C to increase sludge dewaterability. There is a consensus concerning the prominent role of Extracellular Polymeric Substances (EPS) in retaining water in sludge flocs and thus in dewaterability performance [37]. The positive impact of THP on sludge characteristics is thus related to EPS solubilisation and floc destabilisation. In addition, Zhang et al. [38] combined rheological characterisation with the analysis of a porous network structure to explain the mechanism of sludge dewaterability with or without THP. They concluded that THP breaks down the porous network structure of sludge which is further weakened during anaerobic digestion, thus enhancing sludge dewaterability. Furthermore, TH benefits also include a reduction in sludge viscosity, favouring an increase in Total Solid (TS) concentrations in the digester and an increase in the loading rate [34].

One acknowledged limitation of THP is the increase in Total Ammonia Nitrogen (TAN) and consequently in pH values, which shifts the equilibrium from ammonium into its free state, ammonia ( $\text{NH}_3$ ), thus leading to potentially inhibitory levels ( $>100\text{mg}_{\text{NH}_3}/\text{L}$ ) [39]. In the case of ammonia inhibition, a metabolic pathway over acetate oxidation to hydrogen can become dominant instead of classic acetoclastic methanogenesis. This adaptation process on a microbial community generates anaerobic digestion instability. To avoid long-term acclimation when commissioning THP projects, addition of acclimated seed with 1500mg/l ammonia is advised [27].

Sludge sanitation with the subsequent production of class A biosolids is also a considerable advantage of THP. Class A refers to sludge categories defined by the United States Environmental Protection Agency in the 40 CFR – Part 503 regulation. While part 503 rules also define quality regarding heavy metals, the focus of AD processes and digestion enhancements are focused on meeting the requirements concerning pathogen and vector attraction reductions. If biosolids can abide to regulations for Class A, restrictions on land applications could be significantly reduced. Concerning pathogen reductions, according to the Part 503 rule, six alternatives have been defined for meeting Class A Biosolid requirements. Alternative 1 involves the time-temperature requirement, which many of the other alternatives and processes are based on in order to further reduce pathogens. This time-temperature requirement, which implies a given residence time at a given temperature for a type of sludge, can be reached thanks to THP [40].

Recently, very few studies investigated the impact of THP on the fate of micropollutants such as endocrine disrupting compounds [41] or antibiotic gene resistance [42]; these results are too few to be reviewed but should be taken into account for future development or optimisation of the process.

In summary, THP has been studied extensively in order to improve AD performance, i.e. biogas production and VS reduction; industrial plant parameters are based on these results. A significant number of studies dealing with the THP impact on digested sludge dewaterability have been published. However, the main drawback of THP, consisting in the increase of remaining soluble organic nitrogen compounds in the digested sludge, has only recently been highlighted in a small number of studies.

The first objective of this work was to compile and standardise quantitative literature data according to a holistic approach in order to determine the most impactful parameters related to THP on AD performance, digested sludge dewaterability as well as residual colour and soluble COD in the digestate. The second objective was to determine an optimal THP temperature range for which the previously reported drawbacks could be minimized, while anaerobic digestion and dewatering performances would be maintained.

The scope of this study was focused on TH temperatures above 100°C. As discussed previously, TH at low temperatures generally involve either a too long treatment time or alkaline addition. Chemical addition entails other mechanisms, which are not addressed here. Furthermore, as the purpose of this paper is focused on both AD and dewatering performances, low temperature pretreatments are not relevant since they do not enhance dewaterability performances [5].

## 2. Method & Approach

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### 2.1 Data Selection

#### 2.1.1 Performance Indicators

Data used for assessing anaerobic digestion and dewatering performances downstream from thermal pretreatment were selected according to the following performance indicators (PI):

- CH<sub>4</sub> production (CH<sub>4</sub> prod) in Nm<sup>3</sup>/tVS added
- Volatile Solid Reduction (VSR) in %
- Dewaterability (DW) in %TS in dewatered sludge cake, which will be identified as cake dryness in this study.
- Soluble Chemical Oxygen Demand (sCOD), UV absorbance at 254 nm (UVA), ammonium (N-NH<sub>4</sub><sup>+</sup>), dissolved organic nitrogen (DON) concentration in digester effluent. These parameters allow for the quantification of the THP impact on AD dewatered sidestream quality.

In addition, a performance indicator increase was defined for methane production and VSR. For all other performance indicators (dewaterability and filtrate quality), little data on raw sludge were available for

this assessment. Percentages of increase are based on the results obtained with raw sludge in the reference article compared to hydrolysed sludge, according to the following equations:

$$CH_4 \text{ prod increase (\%)} = \frac{CH_4 \text{ prod hydrolysed} - CH_4 \text{ prod raw}}{CH_4 \text{ prod raw}} \quad (1)$$

$$VSR \text{ increase (\% point)} = VSR \text{ hydrolysed} - VSR \text{ raw} \quad (2)$$

### 2.1.2 Parameters Affecting Anaerobic Digestion and Dewatering Performances

The performance indicators presented above are usually associated with THP temperature, but AD and dewatering depend on different parameters. Among the extensive list of factors affecting anaerobic digestion, only the sludge type, the digester hydraulic retention time (HRT) and the AD operating mode (batch, semi-continuous, continuous) were considered. THP temperature, THP treatment time and the impact of a flash-based technology (quick pressure drop) were also examined.

The raw sludge was divided into three main categories: waste activated sludge (WAS), mixed sludge (partial and full lysis) and primary sludge, depending on literature specifications. This parameter was essential for the study, since, due to their different inherent characteristics, AD of primary sludge and WAS lead to different types of performance [22, 40]. Partial and full lysis refer to THP configurations. In a partial lysis configuration, the THP solely processes waste activated sludge. The hydrolysed waste activated sludge is then mixed in with raw primary sludge before being fed to the digester. On the other hand, in a full lysis configuration, the entire sludge stream is thermally treated upstream of the digester. Due to a limited dataset, data from both partial and full lysis configurations were categorised as mixed sludge. In this study, digester HRT ranges between 9 and 40 days with a median at 20 days, a first quartile at 15 days and a third quartile at 28 days. Zhang et al. [38] demonstrated how digestate dewaterability could improve when the duration of digestion was extended. This demonstrates the necessity to assess the impact of HRT in the present study. The selected data combine both industrial and laboratory references. As stated by Barber [14] it is difficult to compare references from lab and full-scale studies due to different operational conditions. Therefore, the data were classified according to the AD operating mode: batch (BMP assays used at lab scale), semi continuous (often used at pilot scale) or continuous mode (industrial references). THP temperatures, ranging between 100°C and 200°C, have been sorted into three categories: [100-140]°C, [140-160]°C and [160-200] °C. THP treatment times vary from 5 min to 180 min with a majority of data around 30 minutes (median, first and third quartile are equal to 30 minutes). As Donoso-Bravo et al. [43] demonstrated improved dewaterability with increasing hydrolysis treatment time from 5 to 30 minutes, this factor was also assessed here. Thermal hydrolysis technologies have been segregated according to whether a flash system was used or not, as no consensus has been found regarding its impact on methane production. Indeed, on one hand, Mottet et al. [44] found that the heating methods (steam injection vs. electric heating) of the TH reactor did not impact downstream digestion performances. On another hand,

Sapkaite et al. [45] and Abelleira-Pereira et al. [46] showed how flash-based THP technology increased biogas production, in contrast with the work of Ngwenya et al. [47].

The 43 selected articles [9, 10, 16, 23-25, 29, 31, 32, 34, 39, 43, 45, 46, 48–76], with their various criteria and performance indicators are presented in the supplementary materials section (Table S1).

## 2.2 Data Analysis

Prior to assessing the impact of temperature on dewatering and AD performances, the influence of (1) the sludge type, (2) THP temperature range, (3) THP duration, (4) flash system, (5) AD HRT and (6) the AD operating mode were explored for each performance indicator. The analysis was carried out with a Kruskal Wallis test. Indeed, a Shapiro test revealed that the set of data did not follow a normal distribution, thus implying that an ANOVA could not be performed. Consequently, the data were extracted and compiled against the above-mentioned performance indicators, box-plotted and statistically analysed to determine the impact of temperature on each indicator. To facilitate statistical analysis, the data were sorted into three THP temperature ranges instead of being compiled individually: temperatures from 100°C to 140°C ([100, 140[°C), from 140°C to 160°C ([140, 160] °C) and from 160°C to 200°C ([160, 200] °C). THP temperature optimisation was assessed through a pairwise comparison between each temperature range using a Wilcoxon test.

## 3. Results & Discussions

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### 3.1 AD and Dewatering Performances: Determination of Impactful Parameters

Prior to the examination of THP temperature optimisation on AD and dewatering performances, each parameter mentioned above (sludge type, digester HRT, AD operating mode, THP treatment time and flash-based technology) was considered using a Kruskal-Wallis Test. Results are summarised in Table 2. The entire dataset used for this study is available in the supplementary materials section.

Unsurprisingly, the sludge type (WAS, PS) and temperature range ([100-140[°C, [140-160] °C, ]160-200] °C) presented a statistically significant impact on all performance indicators ( $p$ -value < 0.05). Indeed, primary sludge contains more lipids than WAS. These are more easily biodegradable in comparison to proteins, the major component of WAS [77]. In addition, Carrère et al. [22] demonstrated the impact of sludge types by highlighting the positive linear regression between initial sludge biodegradability and the efficiency of the thermal treatment. The effect of different sludge types on dewaterability and filtrate quality (UVA and soluble COD) was not assessed because data were only

available for one sludge type. The THP temperature impact is explained for each performance indicator in the following sections 3.2 to 3.5.

The flash system was found to have a significant impact on both VSR and methane production. However, no impact was observed upon either VSR increase or methane production. This result appears contradictory ( $p$ -value  $> 0.05$ ) and does not allow for any conclusion to be drawn concerning the significance of its impact. Similar findings were made for AD operating methods. Consequently, these two parameters could not be accounted for during this study. THP treatment time only impacted soluble COD, because this result was most probably linked to a THP duration of 180 minutes (see supplementary materials section), which is significantly higher than the usual duration of 30 minutes. Nevertheless, in agreement with several published studies, although related data were maintained within the dataset, THP treatment time was not considered to be a discriminatory factor during the rest of this study. Indeed, Donoso-Bravo et al. [43], Sapkaite et al. [45] and Dohányos et al. [78], for instance, demonstrated that the retention time in the thermal hydrolysis process presented a minor impact on digestion performances.

The AD HRT was found to significantly affect methane production only. However, no increase in methane production was observed, because most of the data included a HRT  $> 15$  days, which allowed the methane production to approach its maximum value. Indeed, as demonstrated by Xue et al. [9] and Dwyer et al. [29], biogas production in digesters placed downstream of a THP reaches  $>85\%$  and  $>90\%$  of the measured BMP for a HRT of 10 and 15 days respectively and with a THP temperature ranging from 120 to 180°C. This result was also supported by Ngwenya et al. who reported, after THP, no significant difference in biogas production for a HRT ranging from 10 to 18 days [47]. In conclusion, HRT was not selected as an impactful parameter in the following.

The assessment of impactful parameters highlights the significant impact of sludge types and temperature compared to THP treatment time, flash-based systems or AD operating methods and HRT. The entire data set can therefore be grouped without segregation between THP technologies (flash-based system, THP duration), AD operating methods (batch, semi-continuous or continuous) and AD HRT. Consequently, data compilation and THP temperature optimisation were only based on the sludge type during the remainder of the study. Temperature ranges can subsequently be box-plotted against performance indicators for each sludge type and statistically analysed with Kruskal-Wallis tests and pairwise comparisons (Wilcoxon test).

**Table 2 : p-value of Kruskal Wallis test for the impact of AD HRT, THP duration, AD operating mode, flash system, THP temperature range and sludge type on each AD and dewatering performance indicator**

Parameters	Performance indicators						
	VSR	VSR increase	CH <sub>4</sub> prod	CH <sub>4</sub> prod increase	Cake dryness	Soluble COD	UVA
<b>AD HRT</b>	0.22	0.81	<b>0.0013</b>	0.072	0.52	0.092	-
<b>THP treatment time</b>	0.14	0.12	0.064	0.11	0.71	<b>0.027</b>	-
<b>AD operating mode</b>	<b>0.038</b>	0.77	<b>0.013</b>	0.12	0.92	0.25	-
<b>Flash system</b>	<b>0.013</b>	0.32	<b>0.026</b>	0.10	1	0.14	-

THP temperature range	0.0059	0.0017	0.0069	0.0044	0.033	0.0074	0.050
Sludge type	0.014	0.011	1.6E-08	7.4E-05	-	-	-

## 3.2 Volatile Solid Reduction (VSR)

All data used and associated literature references are available in the supplementary materials. VSR and increase in VSR were box-plotted against each temperature range for each sludge type (Figure 1). Primary sludge was not illustrated due to lack of data. Results from the Kruskal-Wallis test presented in Table 3 highlight the strong impact of the THP temperature range on both VSR and increase in VSR ( $p$  value  $< 1\%$ ) for waste activated sludge.

Pairwise comparisons using the Wilcoxon test revealed that VSR and increase in VSR values were significantly greater at the  $[160\text{--}200]^\circ\text{C}$  temperature range, compared to  $[100\text{--}140]^\circ\text{C}$ . This difference is also illustrated by the box plot in Figure 1. A significant difference was observed between ranges  $[100\text{--}140]^\circ\text{C}$  and  $[140\text{--}160]^\circ\text{C}$  for the VSR increase but not for the VSR indicator. No significant difference was found for both VSR and VSR increase indicators between  $[140\text{--}160]^\circ\text{C}$  and  $[160\text{--}200]^\circ\text{C}$ . THP therefore allows for volatile solid reduction to be enhanced by boosting the limiting hydrolysis step, even from  $100^\circ\text{C}$ . The impact can be observed specifically on waste activated sludge with an increase of  $5 \pm 3$  pt.%,  $14 \pm 5$  pt.%,  $19 \pm 7$  pt.% (percentage points) for the following temperature ranges,  $[100\text{--}140]^\circ\text{C}$ ,  $[140\text{--}160]^\circ\text{C}$ ,  $[160\text{--}200]^\circ\text{C}$  respectively. Indeed, a VSR of 26 – 35% is usually expected for further anaerobic digestion of WAS. VSR values for waste activated sludge of  $38\% \pm 6\%$ ,  $43.5\% \pm 8\%$ ,  $49.5\% \pm 10\%$  were observed for the above-mentioned temperature ranges (Figure 1).

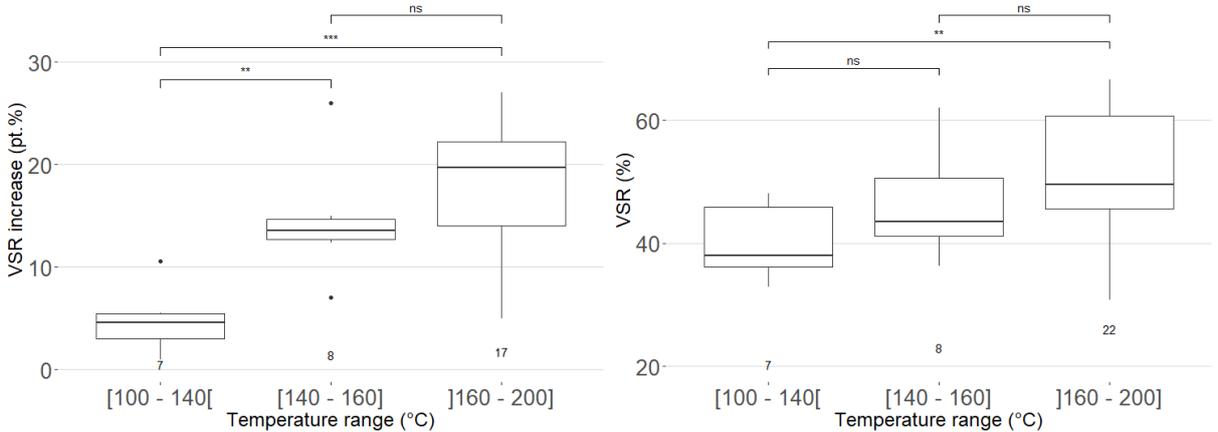
For mixed sludge, no statistically significant difference was observed at the different temperature ranges. This can be due to (1) the limited available data for mixed sludge for the first two temperature ranges and (2) the inherent biodegradability of primary sludge [3]. The latter dilutes the impact of THP operating temperature on VSR performances compared to performances obtained on waste activated sludge. However, an increase in VSR (pt.%) occurred across all temperature ranges for mixed sludge. In ascending order of temperature ranges, a VSR increase of  $2.7 \pm 0$  pt.%,  $9 \pm 2$  pt.%,  $8 \pm 4$  pt.% was achieved respectively (Figure 2), thus highlighting the usefulness of THP even for mixed sludge. The temperature range for optimum performances in volatile solid reduction remained within the  $[140\text{--}200]^\circ\text{C}$  range. No apparent decrease was observed at high temperatures, as observed by Li and Noike [24], Pinnekamp et al. [25] and Xue et al. [9]. Indeed, as most of the data used here belong to the  $[140\text{--}180]^\circ\text{C}$  temperature range, it is thus difficult to evidence a decrease in performances within the  $[160\text{--}200]^\circ\text{C}$  range.

Additionally, VSR calculation method may vary between articles as it was not always clearly specified (Van Kleeck method vs. mass balance). For example, the Van Kleeck equation assumes that fixed solids (mineral matter) are maintained during the digestion process [79], while this assumption does not apply for the mass balance equation.

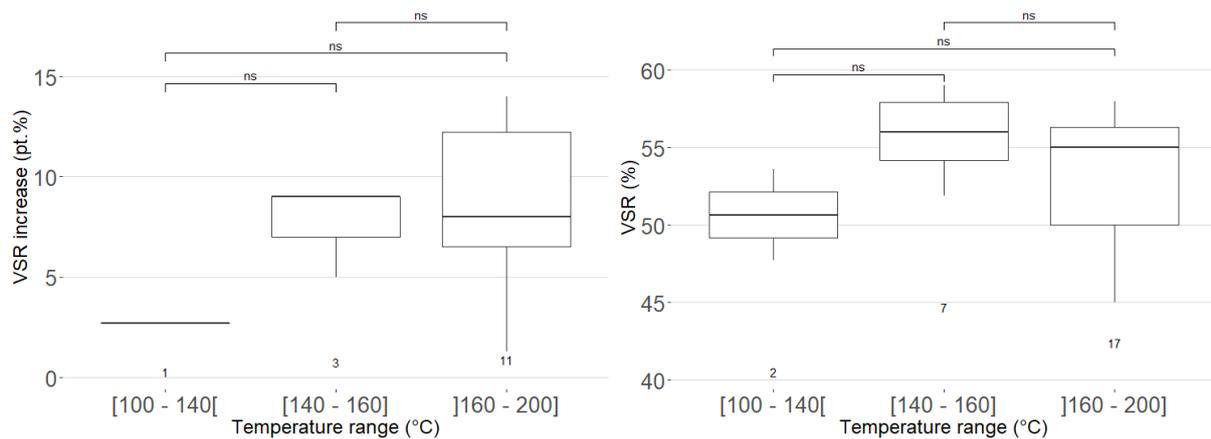
Regarding the VSR performance indicator, the present work suggests the THP operating temperature should be equal or greater than 140°C. For waste activated sludge, box-plot median values tend to increase above 160°C. This observation was not consistent with the mixed sludge performances, where the median rather tended to decrease with increasing temperatures. In addition, for both sludge types, box-plots at high temperatures were wider than those for the [140 – 160] °C range, thus suggesting higher performance variability at high temperatures.

**Table 3 : p-value of Kruskal Wallis test for the impact of THP temperature range on VSR performance indicator and p-value of the THP temperature range pairwise comparison using Wilcoxon test**

Statistical tests	Parameters	Performance indicators			
		WAS – VSR increase	WAS – VSR	Mixed – VSR increase	Mixed – VSR
<b>Kruskal Wallis</b>	<b>THP Temperature range</b>	0.0055*	0.0092*	0.21	0.37
<b>Pairwise comparison using Wilcoxon test</b>	[100 – 140[ °C vs. ]140 – 160] °C	0.0043*	0.189	0.32	0.73
	[100 – 140[ °C vs. ]160 – 200] °C	0.0018*	0.016*	0.56	0.73
	[140 – 160] °C vs. ]160 – 200] °C	0.23	0.182	0.56	0.88



**Figure 1 : VSR versus THP temperature range for WAS. Number of observations are indicated below each box-plot.**



**Figure 2 : VSR versus THP temperature range for mixed sludge. Number of observations are indicated below each box-plot.**

### 3.3 Methane Production

Methane production is a widely used performance indicator in most AD pre-treatment systems as it represents a revenue potential and/or a source of energy. Biogas can be used to generate heat (1 m<sup>3</sup> CH<sub>4</sub> is equivalent to 10 kWh heat), or both electricity and heat when burned in a CHP engine. It can also be upgraded to biomethane and injected into the natural gas grid or used as biofuel.

All data and associated references where methane production performances have been assessed according to THP operating temperature ranges are available in the supplementary materials.

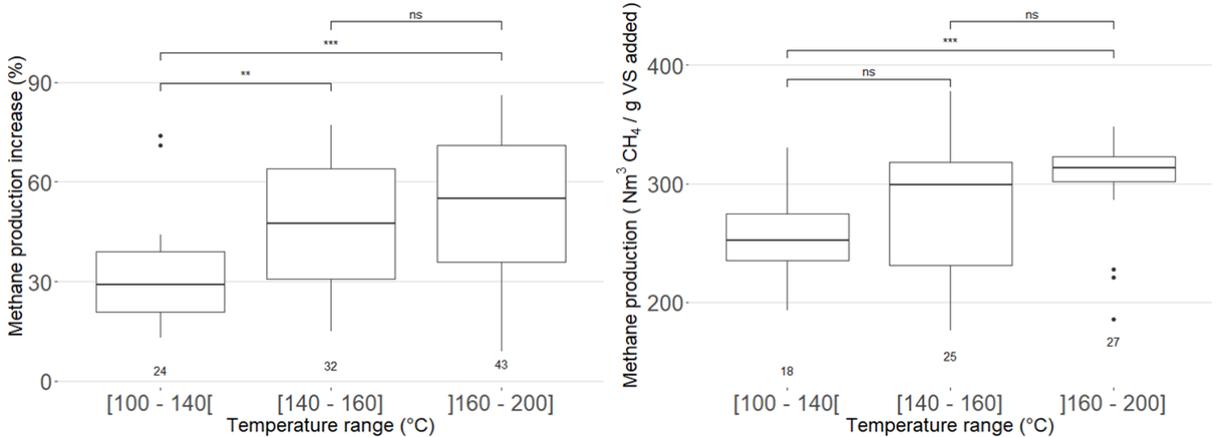
For waste activated sludge, the THP temperature range significantly affected methane production and methane production increase (Table 4 and **Erreur ! Source du renvoi introuvable.**). A pairwise comparison highlighted the differences between both performance indicators, i.e. methane production and methane production increase between [100-140] °C and ]160-200] °C, but not between [140-160] °C and ]160-200] °C. These results concur with the work of Dwyer et al. [29], Wilson et al. [32] and Batstone et al. [50] who did not report any difference in methane production increase between 140°C and 170°C. Being directly related to VSR performances, THP triggered a sharp increase in methane production from waste activated sludge: +29% ± 23%, +48% ± 19%, +57% ± 22% respectively in the order of temperature ranges (Figure 3). However, many publications [9, 23–25, 80] also demonstrated the negative impact of TH temperature above 175°C on biogas production. Indeed, at these temperatures, melanoidins or Maillard reaction compounds are known to be produced. This latter reaction is commonly associated to high temperatures in the food industry. In addition, Pinnekamp et al. [25] showed that the differences in gas yield increases at pretreatment temperatures between 120°C and 180°C were not significant. This is illustrated by the mixed sludge box-plot (Figure 3). Although no statistically significant differences were evidenced between the different THP temperature ranges (Table 4), median values from [100-140]°C to [140-160]°C displayed an increase in biogas production: +20% ± 0% and + 36.5% ± 22% respectively. Nevertheless, a slight decrease was detected from [140-160]°C to [160-180]°C: + 36.5% ± 22% and + 31% ± 10% respectively.

Primary sludge undergoing thermal treatment did not display any increase in methane production. Only Haug et al. [23] found a +1.3% increase. Articles on the thermal hydrolysis of primary sludge are scarce, due to its inherent biodegradability.

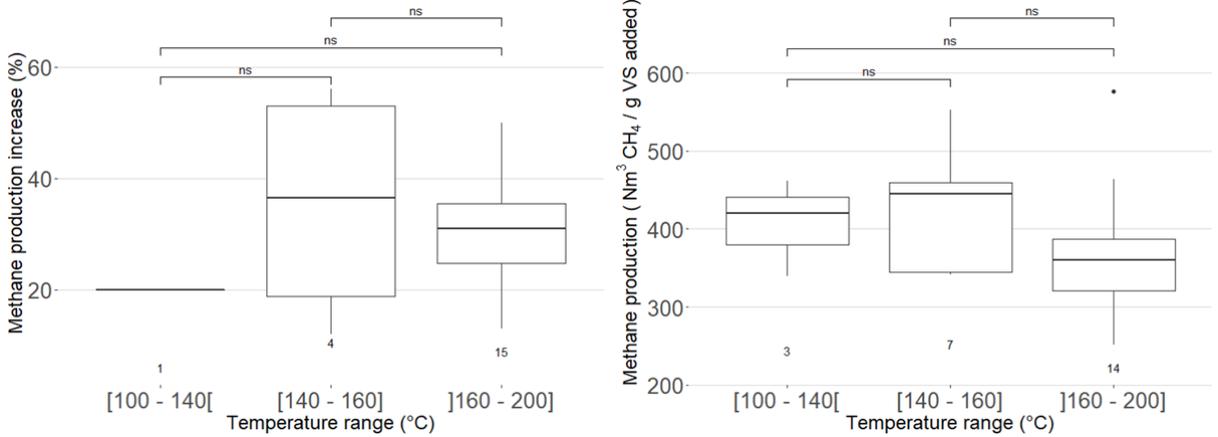
Results obtained from methane production are consistent with those from VSR. The minimum THP operating temperature was found to be 140°C. No maximum has been observed during this study, although individual literature sources highlighted a maximum around 175°C [21, 23–25].

**Table 4 : p-value of Kruskal Wallis test for the impact of THP temperature range on methane production performance indicator and p-value of the THP temperature range pairwise comparison using Wilcoxon test**

Statistical tests	Parameters	Performance indicators			
		WAS – methane production increase	WAS – methane production	Mixed – methane production increase	Mixed – methane production
<b>Kruskal Wallis</b>	<b>THP Temperature range</b>	0.00023 *	0.0033 *	0.56	0.43
<b>Pairwise comparison using Wilcoxon test</b>	[100 – 140[ °C vs. ]140 – 160] °C	0.00359 *	0.16	1	0.86
	[100 – 140[ °C vs. ]160 – 200] °C	0.00036*	0.0019 *	0.98	0.86
	[140 – 160] °C vs. ]160 – 200] °C	0.22	0.13	1	0.84



**Figure 3 : Methane production versus THP temperature range for WAS. Number of observations are indicated below each box plot.**



**Figure 3 : Methane production versus THP temperature range for mixed sludge. Number of observations are indicated below each box plot.**

### 3.4 Dewaterability

Data used for assessing the performance of dewaterability are available in supplementary materials section. Only mixed sludge data were available for this assessment.

The positive influence of thermal hydrolysis on sludge dewaterability is unanimously agreed upon in the literature [21, 37, 38, 81]. For example, TH was found to improve mesophilic digested sludge dewaterability by ca. 10% DS points, depending on the influent sludge composition [14]. Final cake dryness could therefore reach a DS content greater than 30%. On the contrary, without THP, a mean value of 22% can be expected for digested mixed sludge dewatered by centrifugation [40, 82]. In the present study, although all data originate from centrifuge dewatering, it is noteworthy that for THP sludges, dewatering by belt filter press produces equivalent cake solids and capture rates as for centrifuge dewatering [83].

The result from the impact of temperature ranges on dewaterability performances (Figure 4) reveals a significant difference ( $p$ -value < 0.05) on cake dryness performances with a value of  $30 \pm 3$  %DS for temperatures within the [140 – 160] °C range and a value of  $33 \pm 2$  %DS for the [160 – 200] °C range. These values indicate enhanced performances at high temperatures.

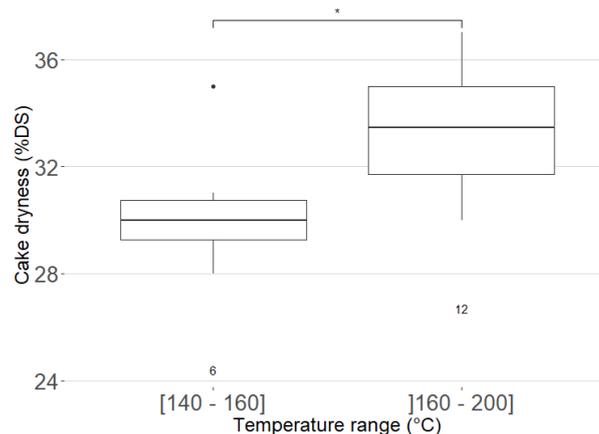
By reviewing individual articles in the literature, a general consensus can be found among the different authors for improved performance above 150°C. Based on laboratory capillary section tests (CST) to describe sludge filterability, Bougrier [31] demonstrated how filterability was enhanced when TH temperature rose above the 150°C threshold. Above this temperature Fdz-Polanco et al. [72] and Haug et al. [23] found an improvement in sludge filterability as the TH temperature increased. Furthermore, the impact was as significant on primary sludge as on activated sludge. Kepp et al. [68] also made similar observations: instead of affecting biodegradability, their thermal pretreatment of primary sludge increased dewaterability. Fishern and Swanwick [84] equally confirmed that dewaterability could improve at temperatures above 150°C over a wide range of sludge types. Finally, more recently, Higgins et al. [85] observed an increase in digested mixed sludge dewaterability from 30% DS at 150°C to 33% DS at 170°C.

Enhancement in cake dewaterability downstream from a THP is related to two main phenomena [81]: (1) the degradation of extracellular polymeric substances (EPS) (proteins and polysaccharides), which in turn reduces their water retention capacity, and (2) the stimulation of flocculation which reduces the amount of fine flocs.

EPS are charged polymers that represent up to 80% of the mass of activated sludge [81]. They occur as an extremely hydrated gel matrix (98% water) in which microbial cells can establish stable synergistic consortia [86, 87]. EPS are produced internally within microbial cells (loosely or tightly bound EPS), and then excreted into the cell environment (free or soluble EPS) where they can form larger microbial aggregates known as biofloc particles. Due to their strong water binding capacity, EPS are responsible for binding water to the floc surface and thus capturing water inside sludge flocs [88]. This water-binding capacity was demonstrated through the swelling/unswelling of the biofloc particles [88]. Recent works by Hasan et al. [89] confirmed that cake dryness correlated negatively with both the bound water and

the EPS content. Moreover, the authors demonstrated an improvement in cake TS with THP, thereby confirming the role of THP in releasing bound water from sludge. This corroborates the work of Neyens et al. [81] and Tian et al. [90], where the destruction of the structural integrity of the EPS matrix plays a crucial role in improving the potential of sludge dewatering [90].

It is noteworthy that the thermal hydrolysis process can favour an increase in polymer demand for dewatering. For example, 13% [71], 22% [62] and 24% [63] rises in polymer consumption have been reported in the literature. These values should be considered with caution as they depend on the sludge type and characteristics of the polymer.



**Figure 4 : Dewaterability versus THP temperature range on mixed sludge**

### 3.5 Filtrate Quality

A number of publications focus on THP for AD and dewatering performances. Yet, in-depth characterisation of the sidestream produced by the dewatering step of digested hydrolysed sludge is still lacking. This stream can also be referred to either as recycled centrate, THP return liquor or anaerobic digestion dewatering sidestream. It is usually rich in nutrients (nitrogen and phosphorus), which could be of potential interest for recovery and use as fertilizers [91, 92]. As it cannot be discharged into the natural environment, it is rather returned to the WWTP headworks. Although the recycled centrate only represents about 1% of the volumetric load, it can contain very high concentrations in carbon, nitrogen and phosphorus as well as refractory compounds and colloidal particles [93, 94] that may affect the quality of treated water. For instance, it can contain up to 15-30% of the nitrogen load of a wastewater treatment plant (WWTP) [95]. To overcome this issue, plants that are required to meet strict nutrient limitations; i.e.,  $TN < 3 \text{ mg/L}$  and  $TP < 0.18 \text{ mg/L}$ , employ a form of biological and/or chemical sidestream treatment to reduce nutrient loads before returning to the mainstream flow [96]. However, thermal hydrolysis can affect the quality of this AD dewatering sidestream as has been highlighted in literature with an increase in  $N-NH_4$ , COD, DON as well as the colour of the digester effluent [29, 97]. Furthermore, an increase in inert particles has also been observed [98]. The present work compiles and compares the nitrogen, carbon and phosphorus contents in the downstream dewatering effluent between a conventional mesophilic AD and a boosted AD. Due to the scarcity in data from AD dewatering sidestreams, the soluble fraction of digested sludge rather than the dewatering

effluent was considered. Consequently, UV absorption, soluble COD and dissolved organic nitrogen (DON) in digester effluent were selected as performance indicators to assess the filtrate quality.

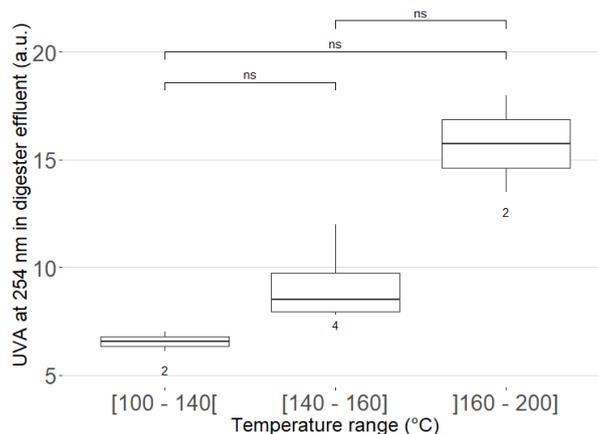
### 3.5.1 UV Absorbance

The characteristic brown colour of the THP return liquor results from the substantial amount of colour generated above 160°C [37]. The colour was found to result from the formation of recalcitrant material, namely lower and high molecular weight melanoidins produced by Maillard reactions. This reaction, also known as browning in the food processing industry, is a non-enzymatic browning reaction that occurs by reducing sugars and amino groups at an elevated temperature, thus forming dark-coloured, UV-quenching, recalcitrant polymers (e.g. Melanoidins) [99]. Melanoidins are nitrogen-containing, macromolecular, dissolved compounds which are used as representative compounds of refractory Dissolved Organic Nitrogen (rDON) species [100]. During THP, Maillard reactions are triggered by the fragmentation of proteins and the release of additional free amines in addition to organic matter solubilisation [13]. Wilson and Novak [77] showed that UV absorbance at 254 nm significantly correlates with the organic nitrogen concentration in post-THP samples.

The products of these reactions, characterised by high molecular organic polymers, are responsible for the poor degradability of sludge hydrolysate produced at high temperatures [101]. In addition, as anaerobic or aerobic digestion downstream of THP does not mitigate the colour associated with these dissolved compounds, they can severely affect downstream UV disinfection (tertiary treatment or portable treatment plant) [32]. Figure 5 illustrates the increase in UVA with THP temperature (p value of Kruskal-Wallis test < 0.05). However, due to insufficient data, a conclusion on the THP temperature range threshold cannot be inferred with pairwise comparison (see supplementary materials). Nonetheless, the overall tendency indicates that a reduction in the coloration without deteriorating AD and dewatering performances can be made possible by maintaining the THP operating temperature within the [140-160] °C range. This is in accordance with the work of Dwyer et al. [29] where a 70% effective reduction of colour formation in THP effluent was observed after decreasing the temperature from 165°C to 140°C. As both melanoidin treatment and mitigation are related to nitrogen removal, this topic is further discussed in the following section (§3.5.3).

**Table 5 : p-value of Kruskal Wallis test for the impact of THP temperature range on UVA performance indicator and p-value of the THP temperature range pairwise comparison using Wilcoxon test**

<i>Statistical tests</i>	Parameters	Performance indicator
		WAS – UVA
<i>Kruskal Wallis</i>	THP Temperature range	0.05 *
<i>Pairwise comparison using Wilcoxon test</i>	[100 – 140] °C vs. [140 – 160] °C	0.4
	[100 – 140] °C vs. [160 – 200] °C	0.4
	[140 – 160] °C vs. [160 – 200] °C	0.4



**Figure 5 : UVA at 254 nm versus THP temperature range on waste activated sludge digester effluent**

### 3.5.2 Soluble COD in Digester Effluent

The increase in COD solubilisation with increasing THP temperature has been highlighted in numerous studies [31, 73, 102–104]. However, comparison of this THP effect with digestion performance, points out that a higher solubilisation of biological sewage sludge does not necessarily lead to an improvement in biodegradability, especially at high temperatures [84, 85].

As expected, results indicate a substantial increase in soluble COD in digester effluent with increasing temperature of TH (: soluble COD (mg / g COD at digester inlet) versus THP temperature range on waste activated sludge (Figure 6). This increase has been statistically confirmed with a Kruskal-Wallis test ( $p$ -value < 0.05). Data used for this study are available in supplementary materials section (only waste activated sludge data were available). Pairwise comparison (Table 6) confirms that the difference in soluble COD concentration is significant between the [100-140]°C and ]160-200]°C temperature ranges and between the [140-160]°C and ]160-200]°C temperature ranges. However, no difference is perceived between [100-140]°C and [140-160]°C, thus demonstrating the substantial increase from 160°C upwards.

Concurrent increases in COD concentrations of the return liquor and THP operating temperature is well documented in the literature and has been demonstrated in full-scale applications [29]. The excess dissolved organic nitrogen (DON) and COD formed during THP is partly due to the production of refractory compounds and colour, which are resistant to biological anaerobic and aerobic degradation and can lead to potential deterioration of WWTP effluent quality [64]. In consequence, an increase in the cost of aerobic treatment of the WWTP to achieve the required COD discharge limit can occur. The formation of these compounds is related to the production of Maillard and Amadori products as previously described in §3.5.1.

On the other hand, excess DON and COD are inherent to the process itself and can be explained by (1) a higher concentration of sludge at the digester inlet and (2) a higher COD solubilisation yield [21].

Although the increase in soluble COD in the return liquor is significant when a THP is implemented upstream from the anaerobic digestion workshop, few articles in the literature describe the impact on the water treatment line. Chauzy et al. [57] revealed an increase in soluble COD concentration in rejected water ranging from 9 to 14 mg/L. Similarly, Phothilangka et al [64] found that 5.7 kgCOD/m<sup>3</sup> of inert soluble compounds were generated with THP compared to 0.9 kgCOD/m<sup>3</sup> with WAS. These inert soluble compounds are diluted in the main stream wastewater line and contribute by about 10 mg/L to the effluent COD. Therefore, COD removal efficiency decreased from 95% to 93%. These findings are in agreement with the results obtained at full scales, where a THP process had been implemented. For instance, the Hengelo WWTP (Netherlands) recorded an increase by 7mg/L of COD in the WWTP effluent once a THP had been installed [63].

Recent work from Toutian et al. [33] investigated the effect of THP operating temperatures on the biodegradability of the THP return liquor at lab scales. This is the first study to clearly establish the exponential relationship between THP operating temperatures and refractory soluble COD (sCOD<sub>ref</sub>). Moreover, the authors proposed an empirical equation where the increased production of sCOD<sub>ref</sub> (and consequently the WWTP effluent COD increase) can be predicted as a function of the THP operating temperature. Accordingly, results pointed out that sCOD<sub>ref</sub> increased by 3.9 to 8.4% with increasing THP operating temperatures from 130°C to 170°C, respectively.

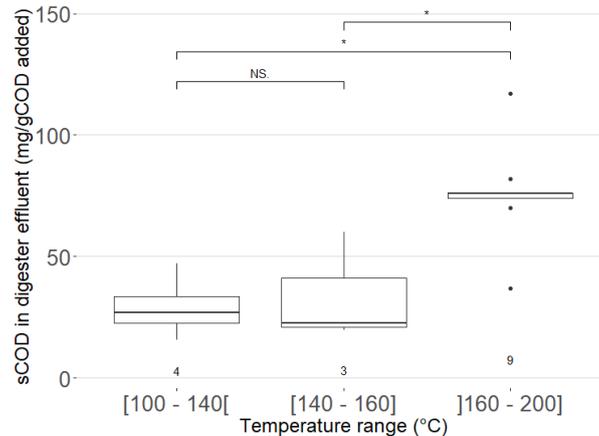
In a context of stringent discharged water quality, the understanding of the impact of this THP specific COD concentration on nutrient removal and recovery processes is paramount in order to continue operating a THP without deleterious environmental impact. In this respect, Fidgore et al. [107] revealed that THP return liquor contains compounds at sufficient concentrations to be inhibitory for the key microbial consortia involved in sidestream biological treatment processes. The authors suggested that inhibition could be associated with inert soluble COD which in turn depends on the characteristics of feed solids and on THP temperature. They observed acclimation of biological activity to the THP digestion sidestream. The THP pre-treated sidestream also presented inhibitory effects on the activity of anammox bacteria, and long-term acclimation could not eliminate inhibition even when diluted with wastewater [94]. Further research is thus required for better characterising the inhibition effects of these sidestreams on nitrogen removal processes [63]. There is growing interest for these issues, in a context where wastewater treatment plants are increasingly regarded as water resource recovery facilities (WRRFs).

Advanced oxidation processes (AOP) such as ozone or ozone with hydrogen peroxyde have proved to treat refractory compounds effectively [96, 108]. Further studies are still required to better understand the underlying mechanisms, optimise energy consumption and oxidant concentration of these treatments for such applications.

**Table 6 : p-value of Kruskal Wallis test for the impact of THP temperature range on sCOD performance indicator and p-value of the THP temperature range pairwise comparison using Wilcoxon test**

<i>Statistical tests</i>	<i>Parameters</i>	<i>Performance indicator</i>
		<b>WAS – sCOD</b>
<i>Kruskal Wallis</i>	<b>THP Temperature range</b>	0.0074 *
	<b>[100 – 140] °C vs. [140 – 160] °C</b>	1

<i>Pairwise comparison using Wilcoxon test</i>	<b>[100 – 140] °C vs. ]160 – 200] °C</b>	0.031*
	<b>[140 – 160] °C vs. ]160 – 200] °C</b>	0.051*



**Figure 6 : soluble COD (mg / g COD at digester inlet) versus THP temperature range on waste activated sludge digester effluent**

### 3.5.3 Nitrogen

Very limited data on nitrogen have been found in the literature, making statistical analysis impossible for this parameter. The literature on the subject is summarised in this section.

The dewatered sidestream from AD of TH pre-treated sludge contains a higher concentration in ammonium and dissolved organic nitrogen compared to conventional mesophilic processes [32, 64, 70, 91]. This THP return liquor is usually recycled back to the wastewater treatment plant and can significantly impact the nitrification and total nitrogen removal performance of secondary treatment units [57, 95]. For instance, according to Wilson et al. [32], the concentration of Total Ammonia Nitrogen (TAN) was 1330 mg/L downstream from a conventional mesophilic digestion. The TAN content increased up to 2510 mg/L when sludge underwent TH upstream from anaerobic digestion. This is as expected since TH favours (1) an increase in the sludge loading rate due to reduced viscosity, (2) a higher solubilisation rate and (3) an increase in biodegradation of organic matter and therefore proteins. This leads to increased release and concentration of ammonium in the digester. More generally, total ammonia release per mass of volatile solids destroyed is equivalent to conventional mesophilic AD and THP for any sludge type [32]. Nevertheless, Wilson and Novak [77] demonstrated that the protein content of WAS made it particularly prone to ammonification at hydrolysis temperatures above 170°C. They therefore suggested an operating temperature of 150°C to reduce TAN loading in the digester by 20-30%. This complies with results from Whang et al., who found that total nitrogen and ammonium concentrations in the liquid fraction of thermally hydrolysed sludge increased with temperature, whereas organic nitrogen presented a maximum within the 120 - 300°C range [109].

Although THP leads to an increase in TAN concentrations for the reasons explained above, the biological nitrogen removal systems employed in wastewater may effectively reduce ammonium in the effluent [70, 95]. However, the excess of dissolved organic nitrogen formed during THP is essentially refractory in nature and is not effectively removed by biological nitrogen removal processes [29, 60]. In

AD dewatering sidestreams, previous studies have estimated dissolved organic nitrogen to represent 20 to 85% of total nitrogen in effluents [57].

Higgins et al. [34] found that DON in the digester effluent was significantly lower than the influent due to the consumption of dissolved biodegradable proteins during anaerobic digestion. However, they also observed that THP temperature increased dissolved organic nitrogen concentrations in digester effluent from 380 mgN/L at 130°C to 470 mgN/L at 160°C, thus implying an overall 24% increase. A positive linear correlation was observed between the increase in THP operating temperatures (from 130°C to 170°C) and DON formation [70]. This increase could result from (1) a higher protein solubilisation with THP [101] and (2) the participation of amino acids in the Maillard reaction and therefore in the formation of compounds that are difficult to degrade or not readily biodegradable, i.e. typically melanoidins.

A comprehensive review by Zhang et al. [30] focusses on the formation of recalcitrant dissolved organic nitrogen (rDON) in thermal hydrolysis. The authors presented the current understanding of melanoidin formation mechanisms and effects in order to control rDON production in sludge during thermal pre-treatment. Four factors were found to impact the production of rDON in THP return liquor: reactant composition (typically sludge type and composition), THP operating temperature and time, pH, and the presence of metallic ions. Regarding the impact of temperature, the authors concluded that the first stage of the Maillard reaction was a rate limiting step that could accelerate at higher temperatures. Therefore, the lowering of the THP operating temperature could be a possible strategy to reduce melanoidin and rDON content in the sludge. Since the production of rDON depends upon multiple parameters [34, 77], the THP should operate at the lowest possible temperature, until a trade-off can be established between rDON production and AD and dewatering performances [30].

An alternative to temperature reduction is the sequestration of melanoidins by coagulation during the dewatering stage, as suggested by Wilson et al. [32]. The authors observed an effective DON and UV-quenching mitigation by using dual conditioning with cationic polymers and ferric chloride (0.10 gFeCl<sub>3</sub> per g total solids). Similarly, Penaud et al. [110] used acid precipitation and ion exchange resins in an effort to remove refractory organics created by thermal hydrolysis.

Melanoidins are known to exhibit antimicrobial and cytotoxic properties [111] Therefore, similarly to refractory COD, melanoidins may contribute to the observed inhibition of the side-stream nitrogen removal process when treating the THP return liquor [112]. Currently, a there is general consensus on the increase of total nitrogen concentrations in the THP return liquor with an increase in THP operating temperature. However, further investigation is still required to provide insights on the characterisation, quantification of rDON and DON present in the THP return liquor. This should allow for a better operation of both the thermal treatment and the side-stream biological nitrogen removal processes. It should also help to ensure that compliance with the regulatory discharge limit for total nitrogen is achieved.

### 3.5.4 Phosphorus

Phosphorus concentrations in the digester effluent are difficult to predict as they depend on the design of the wastewater treatment line (enhanced biological phosphorus removal and/or use of metallic ions such as magnesium, calcium, aluminium, and iron on the water treatment line). However, as THP increases phosphorus solubilisation [96], an increase in phosphate in AD dewatering sidestreams is expected in comparison to conventional digestion.

This has been observed in the work of Chauzy et al. [98] with an increase in total phosphorus concentrations of 0.5-0.8 mg/L in the THP return liquor compared to conventional AD. Conversely, in a study by Han et al. [113] the release of intracellular phosphorus increased during THP, although this phosphorus was immobilized by metallic ions such as Mg, Fe, Ca and Al. Therefore, the phosphorus content in the THP return liquor did not vary. Phosphorus modelling and equilibrium in water and sludge is a current issue, and not only related to the presence of THP in the sludge treatment line. Indeed, the scarcity of this nutrient and its increasing demand has led to particular interest towards achieving its recovery [114]. Hence, recovery of phosphorus release during AD of hydrolysed sludge could become a noteworthy source of revenue in the future.

### 3.6 Optimal THP Temperature Range

In the present literature review, a THP operating temperature range that would mitigate the process drawbacks while maintaining its advantages has been identified. Table 7 summarises the results obtained in the form of a coloured, weighted optimisation. Consequently, the [140-160] °C temperature range stands out as a trade-off to where AD and dewatering performances would be sustained, while the degradation of the filtrate quality would be mitigated.

**Table 7 : THP temperature weighted optimisation (green = optimum, orange = acceptable, red = unfavorable)**

Parameters	Impact on performances		
	100-140°C	140-160°C	160-200°C
Volatile solid removal			
Methane production			
Dewatering			
Filtrate quality			

## 4. Conclusion

The objective of this study was to compile anaerobic and dewatering-related performances at different THP operating temperatures higher than 100°C. It is noteworthy that THP temperature optimisation depends upon multiple parameters and should thus be carried out on a case by case basis. For instance,

dry solid concentrations at the digester inlet, mixing efficiency, viscosity and sludge volatile solid contents have not been considered herein. Besides, the opportunity of reducing digester HRT with a THP [98] was neither quantified nor analysed in this study. No significant differences in VSR and methane production were observed within the 140°C – 200 °C range. Yet, dewatering performances appear to reach a threshold above 160°C. This threshold value is inherent to the temperature ranges defined in this work. Indeed, as the quantity of data relative to sludge dewatering was limited, further discrimination within the 140 - 160°C range could not be possible. According to the cited studies, there is a general consensus that the temperature cut-off value below which dewatering performances become negatively impacted is closer to 150°C than to 160°C. Nevertheless, above 160°C, the filtrate quality tends to sharply deteriorate with a significant increase in refractory sCOD, rDON concentrations and colour. It is paramount to further maintain the development of thermal hydrolysis processes without deteriorating overall environmental impacts of such pretreatments. Consequently, a THP operating temperature in the [140-160°C] range is here proposed as an optimum range where expected performances can be sustained while the production of refractory compounds would be reduced.

## References

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1. Gherghel, A., Teodosiu, C., De Gisi, S.: A review on wastewater sludge valorisation and its challenges in the context of circular economy. *J. Clean. Prod.* 228, 244–263 (2019). <https://doi.org/10.1016/j.jclepro.2019.04.240>
2. Appels, L., Baeyens, J., Degreè, J., Dewil, R.: Principles and potential of the anaerobic digestion of waste-activated sludge. *Prog. Energy Combust. Sci.* 34, 755–781 (2008). <https://doi.org/10.1016/j.pecs.2008.06.002>
3. Carrère, H., Dumas, C., Battimelli, A., Batstone, D.J., Delgenès, J.P., Steyer, J.P., Ferrer, I.: Pretreatment methods to improve sludge anaerobic degradability: A review. *J. Hazard. Mater.* 183, 1–15 (2010). <https://doi.org/10.1016/j.jhazmat.2010.06.129>
4. Anjum, M., Al-Makishah, N.H., Barakat, M.A.: Wastewater sludge stabilization using pre-treatment methods. *Process Saf. Environ. Prot.* 102, 615–632 (2016). <https://doi.org/10.1016/j.psep.2016.05.022>
5. Gonzalez, A., Hendriks, A.T.W.M., van Lier, J.B., de Kreuk, M.: Pre-treatments to enhance the biodegradability of waste activated sludge: Elucidating the rate limiting step. *Biotechnol. Adv.* 36, 1434–1469 (2018). <https://doi.org/10.1016/j.biotechadv.2018.06.001>
6. Zhen, G., Lu, X., Kato, H., Zhao, Y., Li, Y.Y.: Overview of pretreatment strategies for enhancing sewage sludge disintegration and subsequent anaerobic digestion: Current advances, full-scale application and future perspectives. *Renew. Sustain. Energy Rev.* 69, 559–577 (2017). <https://doi.org/10.1016/j.rser.2016.11.187>
7. Nazari, L., Yuan, Z., Santoro, D., Sarathy, S., Ho, D., Batstone, D., Xu, C.C., Ray, M.B.: Low-temperature thermal pre-treatment of municipal wastewater sludge: Process optimization and effects on solubilization and anaerobic degradation. *Water Res.* 113, 111–123 (2017). <https://doi.org/10.1016/j.watres.2016.11.055>
8. Ferrer, I., Ponsá, S., Vázquez, F., Font, X.: Increasing biogas production by thermal (70 °C) sludge pre-treatment prior to thermophilic anaerobic digestion. *Biochem. Eng.*

- J. 42, 186–192 (2008). <https://doi.org/10.1016/j.bej.2008.06.020>
9. Xue, Y., Liu, H., Chen, S., Dichtl, N., Dai, X., Li, N.: Effects of thermal hydrolysis on organic matter solubilization and anaerobic digestion of high solid sludge. *Chem. Eng. J.* 264, 174–180 (2015). <https://doi.org/10.1016/j.cej.2014.11.005>
  10. Nielsen, H.B., Thygesen, A., Thomsen, A.B., Schmidt, J.E.: Anaerobic digestion of waste activated sludge – comparison of thermal pretreatments with thermal inter-stage treatments. 238–245 (2011). <https://doi.org/10.1002/jctb.2509>
  11. Battimelli, A., Millet, C., Delgenès, J.P., Moletta, R.: Anaerobic digestion of waste activated sludge combined with ozone post-treatment and recycling. *Water Sci. Technol.* (2003). <https://doi.org/10.2166/wst.2003.0222>
  12. Ge, H., Jensen, P.D., Batstone, D.J.: Temperature phased anaerobic digestion increases apparent hydrolysis rate for waste activated sludge. *Water Res.* 45, 1597–1606 (2011). <https://doi.org/10.1016/j.watres.2010.11.042>
  13. Kor-Bicakci, G., Eskicioglu, C.: Recent developments on thermal municipal sludge pretreatment technologies for enhanced anaerobic digestion. *Renew. Sustain. Energy Rev.* 110, 423–443 (2019). <https://doi.org/10.1016/j.rser.2019.05.002>
  14. Barber, W.P.F.: Thermal hydrolysis for sewage treatment: A critical review. *Water Res.* 104, 53–71 (2016). <https://doi.org/10.1016/j.watres.2016.07.069>
  15. Cano, R., Pérez-Elvira, S.I., Fdz-Polanco, F.: Energy feasibility study of sludge pretreatments: A review. *Appl. Energy.* 149, 176–185 (2015). <https://doi.org/10.1016/j.apenergy.2015.03.132>
  16. Chauzy, J., Cretenot, D., Bausseron, A., Deleris, S.: Anaerobic digestion enhanced by thermal hydrolysis: First reference Biothelys(R) at Saumur, France. *Water Pract. Technol.* 3, 2–9 (2008). <https://doi.org/10.2166/WPT.2008004>
  17. Abu-Orf, M., Goss, T.: Comparing Thermal Hydrolysis Processes (CAMBI™ and EXELYS™) For Solids Pretreatment Prior To Anaerobic Digestion. *Proc. Water Environ. Fed.* 2012, 1024–1036 (2012). <https://doi.org/10.2175/193864712811693272>
  18. Williams, T.O., B.P.: Thermal hydrolysis offerings and performances. In: *European Biosolids and Organic Resources Conference* (2016)
  19. Luning, J., Hol, L., Dijk, A. Van, Man, D.: Full scale experiences with TurboTec® continuous thermal hydrolysis at WWTP Venlo ( NL ) and Apeldoorn ( NL ) The TurboTec approach. In: *19th European Biosolids & Organic Resources Conference & Exhibition* (2014)
  20. Geraats, B.: LYSOTHERM® SLUDGE HYDROLYSIS Five year experience with a novel approach for operational savings. In: *19th European Biosolids & Organic Resources Conference & Exhibition LYSOTHERM®* (2014)
  21. Bougrier, C., Delgenès, J.P., Carrère, H.: Effects of thermal treatments on five different waste activated sludge samples solubilisation, physical properties and anaerobic digestion. *Chem. Eng. J.* 139, 236–244 (2008). <https://doi.org/10.1016/j.cej.2007.07.099>
  22. Carrère, H., Bougrier, C., Castets, D., Delgenès, J.P.: Impact of initial biodegradability on sludge anaerobic digestion enhancement by thermal pretreatment. *J. Environ. Sci. Heal. - Part A Toxic/Hazardous Subst. Environ. Eng.* 43, 1551–1555 (2008). <https://doi.org/10.1080/10934520802293735>
  23. Haug, R.T., Stuckey, D.C., Gossett, J.M., McCarty, P.L.: Effect of thermal pretreatment on digestibility and dewaterability of organic sludges. *J. Water Pollut. Control Fed.* 50, 73–85 (1978)

24. Li, Y.-Y., Noike, T.: Upgrading of Anaerobic Digestion of Waste Activated Sludge by Thermal Pretreatment. *Water Sci. Technol.* 26, 857–866 (1992). <https://doi.org/10.2166/wst.1992.0466>
25. Pinnekamp, J.: Effects of thermal pretreatment of sewage sludge on anaerobic digestion. *Water Sci. Technol.* 21, 1542–1543 (1989). <https://doi.org/10.2166/wst.1989.0214>
26. Pilli, S., Yan, S., Tyagi, R.D., Surampalli, R.Y.: Thermal pretreatment of sewage sludge to enhance anaerobic digestion: A review. *Crit. Rev. Environ. Sci. Technol.* 45, 669–702 (2015). <https://doi.org/10.1080/10643389.2013.876527>
27. Panter, K., THP Consultant Cambi, Fountain, P., Shana, A., Sludge process specialists, Thames Water UK: The Effects of Thermal Hydrolysis, Hydraulic Retention Time and Ammonia Concentration on Digestion Rates and Dewatering Across Digestion Sites in Thames Water UK. In: *WEF/IWA Residuals and Biosolids Conference 2019* (2019)
28. Choi, J.M., Han, S.K., Lee, C.Y.: Enhancement of methane production in anaerobic digestion of sewage sludge by thermal hydrolysis pretreatment. *Bioresour. Technol.* 259, 207–213 (2018). <https://doi.org/10.1016/j.biortech.2018.02.123>
29. Dwyer, J., Starrenburg, D., Tait, S., Barr, K., Batstone, D.J., Lant, P.: Decreasing activated sludge thermal hydrolysis temperature reduces product colour, without decreasing degradability. *Water Res.* 42, 4699–4709 (2008). <https://doi.org/10.1016/j.watres.2008.08.019>
30. Zhang, D., Feng, Y., Huang, H., Khunjar, W., Wang, Z.: Recalcitrant dissolved organic nitrogen formation in thermal hydrolysis pretreatment of municipal sludge. *Environ. Int.* 138, 105629 (2020). <https://doi.org/10.1016/j.envint.2020.105629>
31. Bougrier, C.: Optimisation du procédé de méthanisation par mise en place d'un co-traitement physico-chimique : Application au gisement de biogaz représenté par les boues d'épuration des eaux usées, (2005)
32. Wilson, C.A., Tanneru, C.T., Banjade, S., Murthy, S.N., Novak, J.T.: Anaerobic Digestion of Raw and Thermally Hydrolyzed Wastewater Solids Under Various Operational Conditions. *Water Environ. Res.* 83, 815–825 (2011). <https://doi.org/10.2175/106143011x12928814444934>
33. Toutian, V., Barjenbruch, M., Unger, T., Loderer, C., Remy, C.: Effect of temperature on biogas yield increase and formation of refractory COD during thermal hydrolysis of waste activated sludge. *Water Res.* 171, (2020). <https://doi.org/10.1016/j.watres.2019.115383>
34. Higgins, M.J., Beightol, S., Mandahar, U., Suzuki, R., Xiao, S., Lu, H.W., Le, T., Mah, J., Pathak, B., DeClippeleir, H., Novak, J.T., Al-Omari, A., Murthy, S.N.: Pretreatment of a primary and secondary sludge blend at different thermal hydrolysis temperatures: Impacts on anaerobic digestion, dewatering and filtrate characteristics. *Water Res.* 122, 557–569 (2017). <https://doi.org/10.1016/j.watres.2017.06.016>
35. Lu, D., Sun, F., Zhou, Y.: Insights into anaerobic transformation of key dissolved organic matters produced by thermal hydrolysis sludge pretreatment. *Bioresour. Technol.* 266, 60–67 (2018). <https://doi.org/10.1016/j.biortech.2018.06.059>
36. Zhang, Q., De Clippeleir, H., Su, C., Al-Omari, A., Wett, B., Vlaeminck, S.E., Murthy, S.: Deammonification for digester supernatant pretreated with thermal hydrolysis: overcoming inhibition through process optimization. *Appl. Microbiol. Biotechnol.* 100, 5595–5606 (2016). <https://doi.org/10.1007/s00253-016-7368-0>
37. Neyens, E., Baeyens, J.: A review of thermal sludge pre-treatment processes to improve dewaterability. *J. Hazard. Mater.* 98, 51–67 (2003).

[https://doi.org/10.1016/S0304-3894\(02\)00320-5](https://doi.org/10.1016/S0304-3894(02)00320-5)

38. Zhang, J., Li, N., Dai, X., Tao, W., Jenkinson, I.R., Li, Z.: Enhanced dewaterability of sludge during anaerobic digestion with thermal hydrolysis pretreatment: New insights through structure evolution. *Water Res.* 131, 177–185 (2018). <https://doi.org/10.1016/j.watres.2017.12.042>
39. Wett, B., Takács, I., Batstone, D., Wilson, C., Murthy, S.: Anaerobic model for high-solids or high-temperature digestion - Additional pathway of acetate oxidation. *Water Sci. Technol.* 69, 1634–1640 (2014). <https://doi.org/10.2166/wst.2014.047>
40. Metcalf & Eddy. Inc: *Wastewater energy: treatment and reuse*. (2003)
41. Armstrong, D.L., Rice, C.P., Ramirez, M., Torrents, A.: Influence of thermal hydrolysis-anaerobic digestion treatment of wastewater solids on concentrations of triclosan, triclocarban, and their transformation products in biosolids. *Chemosphere.* 171, 609–616 (2017). <https://doi.org/10.1016/j.chemosphere.2016.12.122>
42. Wang, M., Li, R., Zhao, Q.: Distribution and removal of antibiotic resistance genes during anaerobic sludge digestion with alkaline, thermal hydrolysis and ultrasonic pretreatments. *Front. Environ. Sci. Eng.* 13, 1–10 (2019). <https://doi.org/10.1007/s11783-019-1127-2>
43. Donoso-Bravo, A., Pérez-Elvira, S., Aymerich, E., Fdz-Polanco, F.: Assessment of the influence of thermal pre-treatment time on the macromolecular composition and anaerobic biodegradability of sewage sludge. *Bioresour. Technol.* 102, 660–666 (2011). <https://doi.org/10.1016/j.biortech.2010.08.035>
44. Mottet, A., Steyer, J.P., Déléris, S., Vedrenne, F., Chauzy, J., Carrère, H.: Kinetics of thermophilic batch anaerobic digestion of thermal hydrolysed waste activated sludge. *Biochem. Eng. J.* 46, 169–175 (2009). <https://doi.org/10.1016/j.bej.2009.05.003>
45. Sapkaite, I., Barrado, E., Fdz-Polanco, F., Pérez-Elvira, S.I.: Optimization of a thermal hydrolysis process for sludge pre-treatment. *J. Environ. Manage.* 192, 25–30 (2017). <https://doi.org/10.1016/j.jenvman.2017.01.043>
46. Abelleira-Pereira, J.M., Pérez-Elvira, S.I., Sánchez-Oneto, J., de la Cruz, R., Portela, J.R., Nebot, E.: Enhancement of methane production in mesophilic anaerobic digestion of secondary sewage sludge by advanced thermal hydrolysis pretreatment. *Water Res.* 71, 330–340 (2015). <https://doi.org/10.1016/j.watres.2014.12.027>
47. Ngwenya, Z., Beightol, S., NgoneOo, T., Vega, J., Pathak, B., Al-Omari, A., Zhu, K., Wadhawan, T., Murthy, S.N., Higgins, M.J.: A stoichiometric approach to control digester chemistry and ammonia inhibition in anaerobic digestion with thermal hydrolysis pretreatment model development. In: *WEF Residuals & Biosolids 2015*. Water & Environment Federation, Washington D.C. (2015)
48. Jeong, S.Y., Chang, S.W., Ngo, H.H., Guo, W., Nghiem, L.D., Banu, J.R., Jeon, B.H., Nguyen, D.D.: Influence of thermal hydrolysis pretreatment on physicochemical properties and anaerobic biodegradability of waste activated sludge with different solids content. *Waste Manag.* 85, 214–221 (2019). <https://doi.org/10.1016/j.wasman.2018.12.026>
49. Ennouri, H., Miladi, B., Diaz, S.Z., Güelfo, L.A.F., Solera, R., Hamdi, M., Bouallagui, H.: Effect of thermal pretreatment on the biogas production and microbial communities balance during anaerobic digestion of urban and industrial waste activated sludge. *Bioresour. Technol.* 214, 184–191 (2016). <https://doi.org/10.1016/j.biortech.2016.04.076>
50. Batstone, D., Lovett, R., Mieog, J., Mbamba, C.K., Yap, S.D., Pagliaccia, P.: Sludge treatment options analysis : Integrated analysis of thermal hydrolysis and recuperative thickening. In: *OZwater'18*. Australia's international water conference & exhibition

(2018)

51. Chen, S., Li, N., Dong, B., Zhao, W., Dai, L., Dai, X.: New insights into the enhanced performance of high solid anaerobic digestion with dewatered sludge by thermal hydrolysis: Organic matter degradation and methanogenic pathways. *J. Hazard. Mater.* 342, 1–9 (2018). <https://doi.org/10.1016/j.jhazmat.2017.08.012>
52. Batstone, D.J., Balthes, C., Barr, K.: Model assisted startup of anaerobic digesters fed with thermally hydrolysed activated sludge. *Water Sci. Technol.* 62, 1661–1666 (2010). <https://doi.org/10.2166/wst.2010.487>
53. Han, Y., Zhuo, Y., Peng, D., Yao, Q., Li, H., Qu, Q.: Influence of thermal hydrolysis pretreatment on organic transformation characteristics of high solid anaerobic digestion. *Bioresour. Technol.* 244, 836–843 (2017). <https://doi.org/10.1016/j.biortech.2017.07.166>
54. Liu, X., Wang, W., Gao, X., Zhou, Y., Shen, R.: Effect of thermal pretreatment on the physical and chemical properties of municipal biomass waste. *Waste Manag.* 32, 249–255 (2012). <https://doi.org/10.1016/j.wasman.2011.09.027>
55. Mills, N., Pearce, P., Farrow, J., Thorpe, R.B., Kirkby, N.F.: Environmental & economic life cycle assessment of current & future sewage sludge to energy technologies. *Waste Manag.* 34, 185–195 (2014). <https://doi.org/10.1016/j.wasman.2013.08.024>
56. Valo, A., Carrère, H., Delgenès, J.P.: Thermal, chemical and thermo-chemical pretreatment of waste activated sludge for anaerobic digestion. *J. Chem. Technol. Biotechnol.* 79, 1197–1203 (2004). <https://doi.org/10.1002/jctb.1106>
57. Graja, S., Chauzy, J., Fernandes, P., Patria, L., Cretenot, D.: Reduction of sludge production from WWTP using thermal pretreatment and enhanced anaerobic methanisation. *Water Sci. Technol.* 52, 267–273 (2005). <https://doi.org/10.2166/wst.2005.0527>
58. Pérez-Elvira, S.I., Fdz-Polanco, M., Fdz-Polanco, F.: Enhancement of the conventional anaerobic digestion of sludge: Comparison of four different strategies. *Water Sci. Technol.* 64, 375–383 (2011). <https://doi.org/10.2166/wst.2011.593>
59. Perez-Elvira, S.I., Sapkaite, I., Fdz-Polanco, F.: Evaluation of thermal steam-explosion key operation factors to optimize biogas production from biological sludge. *Water Sci. Technol.* 72, 937–945 (2015). <https://doi.org/10.2166/wst.2015.294>
60. Phothilangka, P., Schoen, M.A., Huber, M., Luchetta, P., Winkler, T., Wett, B.: Prediction of thermal hydrolysis pretreatment on anaerobic digestion of waste activated sludge. *Water Sci. Technol.* 58, 1467–1473 (2008). <https://doi.org/10.2166/wst.2008.726>
61. Goldhardt, J., Knoerle, U., CaDavid, G.: Continuous Thermal Hydrolysis without Steam or Chemicals. In: *WEF/IWA Residuals and Biosolids Conference 2019*. pp. 484–499 (2019)
62. Camacho, P., Ewert, W., Kopp, J., Panter, K., Perez-Elvira, S.I., Piat, E.: Combined experiences of thermal hydrolysis and anaerobic digestion – latest thinking on thermal hydrolysis of secondary sludge only for optimum dewatering and digestion. *WEFTEC 2008*. 1964–1978 (2008). <https://doi.org/10.2175/193864708788733972>
63. Oosterhuis, M., Ringoot, D., Hendriks, A., Roeleveld, P.: Thermal hydrolysis of waste activated sludge at Hengelo Wastewater Treatment Plant, the Netherlands. *Water Sci. Technol.* 70, 1–7 (2014). <https://doi.org/10.2166/wst.2014.107>
64. Phothilangka, P., Schoen, M.A., Wett, B.: Benefits and drawbacks of thermal pre-hydrolysis for operational performance of wastewater treatment plants. *Water Sci. Technol.* 58, 1547–1553 (2008). <https://doi.org/10.2166/wst.2008.500>

65. Barjenbruch, M., Kopplow, O.: Enzymatic, mechanical and thermal pre-treatment of surplus sludge. *Adv. Environ. Res.* 7, 715–720 (2003)
66. Lu, H.-W., Xiao, S., Le, T., Al-Omari, A., Higgins, M., Boardman, G., Novak, J., Murthy, S.: Evaluation of Solubilization Characteristics of Thermal Hydrolysis Process. In: *WEFTEC 2014* (2014)
67. Fjorside, C.: Full Scale Experience of Retrofitting Thermal Hydrolysis To an Existing Anaerobic Digester for the Digestion of Waste Activated Sludge. In: *Residuals and biosolids management conference* (2002)
68. Kepp, U., Machenbach, I., Weisz, N., Solheim, O.E.: Enhanced stabilisation of sewage sludge through thermal hydrolysis - Three years of experience with full scale plant. *Water Sci. Technol.* 42, 89–96 (2000). <https://doi.org/10.2166/wst.2000.0178>
69. Strong, P.J., McDonald, B., Gapes, D.J.: Combined thermochemical and fermentative destruction of municipal biosolids: A comparison between thermal hydrolysis and wet oxidative pre-treatment. *Bioresour. Technol.* 102, 5520–5527 (2011). <https://doi.org/10.1016/j.biortech.2010.12.027>
70. Ahuja, N.: *Impact of Operating Conditions on Thermal Hydrolysis Pre-Treated Digestion Return Liquor.* (2015)
71. Bishnoi, P.: *Effect of thermal hydrolysis pre-treatment on anaerobic digestion of sludge,* (2012)
72. Fdz-Polanco, F., Velazquez, R., Perez-Elvira, S.I., Casas, C., del Barrio, D., Cantero, F.J., Fdz-Polanco, M., Rodriguez, P., Panizo, L., Serrat, J., Rouge, P.: Continuous thermal hydrolysis and energy integration in sludge anaerobic digestion plants. *Water Sci. Technol.* 57, 1221–1226 (2008). <https://doi.org/10.2166/wst.2008.072>
73. Perez-Elvira, S.I., Fdz-Polanco, M., Fdz-Polanco, F.: Increasing the performance of anaerobic digestion: Pilot scale experimental study for thermal hydrolysis of mixed sludge. *Front. Environ. Sci. Eng. China.* 4, 135–141 (2010). <https://doi.org/10.1007/s11783-010-0024-5>
74. Pérez-Elvira, S.I., Fdz-Polanco, F.: Continuous thermal hydrolysis and anaerobic digestion of sludge. Energy integration study. *Water Sci. Technol.* 65, 1839–1846 (2012). <https://doi.org/10.2166/wst.2012.863>
75. Pérez-Elvira, S., Fdz-Polanco, F.: Improving Anaerobic Digestion by Physico-Chemical Pretreatment. In: *International Workshop on Anaerobic Digestion.* pp. 1–9 (2009)
76. Tobias Prochnow: *Elaboration of an operation manual for the thermal sewage sludge disintegration plant in the WWTP Grevesmühlen and bench and pilot scale investigations supporting the start-up.* (2015)
77. Wilson, C.A., Novak, J.T.: Hydrolysis of macromolecular components of primary and secondary wastewater sludge by thermal hydrolytic pretreatment. *Water Res.* 43, 4489–4498 (2009). <https://doi.org/10.1016/j.watres.2009.07.022>
78. Dohányos, M., Zábranská, J., Kutil, J., Jeníček, P.: Improvement of anaerobic digestion of sludge. *Water Science and Technology.* *Water Sci. Technol.* 49, 89–96 (2004). <https://doi.org/10.2166/wst.2004.0616>
79. Switzenbaum, M.S., Farrell, J.B., Pincince, A.B.: Relationship Between the Van Kleeck and Mass-Balance Calculation of Volatile Solids Loss. *Water Environ. Res.* 75, 377–380 (2003). <https://doi.org/10.2175/106143003x141187>
80. Stuckey, D.C., McCarty, P.L.: The effect of thermal pretreatment on the anaerobic biodegradability and toxicity of waste activated sludge. *Water Res.* 18, 1343–1353 (1984). [https://doi.org/10.1016/0043-1354\(84\)90002-2](https://doi.org/10.1016/0043-1354(84)90002-2)

81. Neyens, E., Baeyens, J., Dewil, R., De Heyder, B.: Advanced sludge treatment affects extracellular polymeric substances to improve activated sludge dewatering. *J. Hazard. Mater.* 106, 83–92 (2004). <https://doi.org/10.1016/j.jhazmat.2003.11.014>
82. Girault, R., Tosoni, J., Reverdy, A.-L., Marion, R., Baudez, J.: Déshydratation mécanique des boues d'épuration - Etat des lieux des filières en France métropolitaine. Rapport du projet ONEMA. (2014)
83. Higgins, M., Murthy, S.N., Schafer, P., Cooper, A., Kasirga, E., Lee, K., Machisko, J., Fountain, P., Kelleher, K.: Dewatering Characteristics of Cambi Thermal Hydrolysis Biosolids: Centrifuges vs. BFPs. In: WEFTEC 2011 (2011)
84. Fisher, R.A., Swanwick, S.J.: High temperature treatment of sewage sludge. *Water Pollut.* 255–370 (1971)
85. Higgins, M.J., Beightol, S., Mandahar, U., Xiao, S., Lu, H.W., Le, T., Mah, J., Patha, B., Novak, J., Al-Omari, A., Murthy, S.N.: Effect of thermal hydrolysis temperature on anaerobic digestion, dewatering and filtrate characteristics. In: 87th Annual Water Environment Federation Technical Exhibition and Conference, WEFTEC 2014. pp. 2027–2037 (2014)
86. Nielsen, P.H., Jahn, A.: Extraction of EPS. In: *Microbial Extracellular Polymeric Substances*. pp. 49–72. Springer Berlin Heidelberg (1999)
87. Flemming, H.-C.: EPS - Then and now. *microorganisms*. (2016). <https://doi.org/10.3390/microorganisms4040041>
88. Keiding, K., Wybrandt, L., Nielsen, P.H.: Remember the water - a comment on EPS colligative properties. *Water Sci. Technol.* 43, 17–23 (2001). <https://doi.org/10.2166/wst.2001.0330>
89. Hasan, M., Zhang, Q., Riffat, R., Al-Omari, A., Murthy, S., Higgins, M.J., Clippeleir, H. De: Mechanistically Understanding the Dewatering Fundamentals: Impact of Biological Systems and Thermal Hydrolysis on Cake Total Solids and Polymer Demand. *Proc. Water Environ. Fed.* 2017, 997–1001 (2017). <https://doi.org/10.2175/193864717821495537>
90. Tian, Y., Zheng, L., Sun, D.-Z.: Functions and behaviors of activated sludge extracellular polymeric substances (EPS): a promising environmental interest. *J. Environ. Sci. (China)*. 18, 420–7 (2006)
91. Figdore, B., Bowden, G., Bodniewicz, B., Bailey, W., Derminassian, R., Kharkar, S., Murthy, S.: Impact of Thermal Hydrolysis Solids Pretreatment on Sidestream Treatment Process Selection at the DC Water Blue Plains AWTP. *Proc. Water Environ. Fed.* (2010). <https://doi.org/10.2175/193864710798193950>
92. Suárez-Iglesias, O., Urrea, J.L., Oulego, P., Collado, S., Díaz, M.: Valuable compounds from sewage sludge by thermal hydrolysis and wet oxidation. A review. *Sci. Total Environ.* 584–585, 921–934 (2017). <https://doi.org/10.1016/j.scitotenv.2017.01.140>
93. Zhang, Q., Vlaeminck, S.E., Debarbadillo, C., Su, C., Al-omari, A., Wett, B., Pümpel, T., Shaw, A.: Supernatant organics from anaerobic digestion after thermal hydrolysis cause direct and / or diffusional activity loss for nitrification and anammox. *Water Res.* 143, 270–281 (2020). <https://doi.org/10.1016/j.watres.2018.06.037>
94. Gu, Z., Li, Y., Yang, Y., Xia, S., Hermanowicz, S.W., Alvarez-Cohen, L.: Inhibition of anammox by sludge thermal hydrolysis and metagenomic insights. *Bioresour. Technol.* 270, 46–54 (2018). <https://doi.org/10.1016/j.biortech.2018.08.132>
95. Constantine, T.A.: North American Experience with Centrate Treatment Technologies for Ammonia and Nitrogen Removal. In: WEFTEC 2006. Water Environment

Federation (2006)

96. Khunjar, W.O., Horne, M. Van, Pace, G., Bilyk, K., Sveum, K.: Strategies for treating biodegradable and recalcitrant nutrient fractions generated from THP processes. WEF/IWA Residuals Biosolids Conf. 2019. 343–344 (2019)
97. Wilson, C.A., Novak, J., Takacs, I., Wett, B., Murthy, S.: The kinetics of process dependent ammonia inhibition of methanogenesis from acetic acid. *Water Res.* 46, 6247–6256 (2012). <https://doi.org/10.1016/j.watres.2012.08.028>
98. Chauzy, J., Graja, S., Gerardin, F., Crétenot, D., Patria, L., Fernandes, P.: Minimisation of excess sludge production in a WWTP by coupling thermal hydrolysis and rapid anaerobic digestion. *Water Sci. Technol.* 52, 255–263 (2005). <https://doi.org/10.2166/wst.2005.0701>
99. Nursten, H.: The chemistry of nonenzymic browning. *The Maillard Reaction.* (2005)
100. Dignac, M.F., Ginestet, P., Rybacki, D., Bruchet, A., Urbain, V., Scribe, P.: Fate of wastewater organic pollution during activated sludge treatment: Nature of residual organic matter. *Water Res.* 34, 4185–4194 (2000). [https://doi.org/10.1016/S0043-1354\(00\)00195-0](https://doi.org/10.1016/S0043-1354(00)00195-0)
101. Wilson, C.A., Novak, J.T., Boardman, G.D., Chen, J.-S., Higgins, M.J., Murthy, S.N.: Mechanisms of Methanogenic Inhibition in Advanced Anaerobic Digestion. (2009)
102. Aboulfoth, A.M., El Gohary, E.H., El Monayeri, O.D.: Effect of thermal pretreatment on the solubilization of organic matters in a mixture of primary and waste activated sludge. *J. Urban Environ. Eng.* 9, 82–88 (2015). <https://doi.org/10.4090/juee.2015.v9n1.082088>
103. Climent, M., Ferrer, I., Baeza, M. del M., Artola, A., Vázquez, F., Font, X.: Effects of thermal and mechanical pretreatments of secondary sludge on biogas production under thermophilic conditions. *Chem. Eng. J.* 133, 335–342 (2007). <https://doi.org/10.1016/j.cej.2007.02.020>
104. Kim, J., Park, C., Kim, T.-H., Lee, M., Kim, S., Kim, S.-W., Lee, J.: Effects of various pretreatments for enhanced anaerobic digestion with waste activated sludge. *J. Biosci. Bioeng.* 95, 271–275 (2003). [https://doi.org/10.1016/S1389-1723\(03\)80028-2](https://doi.org/10.1016/S1389-1723(03)80028-2)
105. Müller, J.A.: Prospects and problems of sludge pre-treatment processes. *Water Sci. Technol.* 44, 121–128 (2001). <https://doi.org/10.2166/wst.2001.0598>
106. Penaud, V., Delgenes, J.P., Moletta, R.: Influence of thermochemical pretreatment conditions on solubilization and anaerobic biodegradability of a microbial biomass. *Environ. Technol. (United Kingdom)*. 21, 87–96 (2000). <https://doi.org/10.1080/09593332108618141>
107. Figdore, B., Wett, B., Hell, M., Murthy, S.: Deammonification of Dewatering Sidestream from Thermal Hydrolysis-Mesophilic Anaerobic Digestion Process. *Proc. Water Environ. Fed.* 2011, 1037–1052 (2011). <https://doi.org/10.2175/193864711802867289>
108. Dwyer, J., Kavanagh, L., Lant, P.: The degradation of dissolved organic nitrogen associated with melanoidin using a UV/H<sub>2</sub>O<sub>2</sub> AOP. *Chemosphere.* 71, 1745–1753 (2008). <https://doi.org/10.1016/j.chemosphere.2007.11.027>
109. Wang, L., Chang, Y., Liu, Q.: Fate and distribution of nutrients and heavy metals during hydrothermal carbonization of sewage sludge with implication to land application. *J. Clean. Prod.* 225, 972–983 (2019). <https://doi.org/10.1016/j.jclepro.2019.03.347>
110. Penaud, V., Delgenès, J.-P., Moletta, R.: Characterization of Soluble Molecules from Thermochemically Pretreated Sludge. *J. Environ. Eng.* 126, 397–402 (2000).

[https://doi.org/10.1061/\(ASCE\)0733-9372\(2000\)126:5\(397\)](https://doi.org/10.1061/(ASCE)0733-9372(2000)126:5(397))

111. Chandra, R., Naresh, R., Rai, V.: Melanoidins as major colourant in sugarcane molasses based distillery effluent and its degradation. *Biore.* 99, 4648–4660 (2008). <https://doi.org/10.1016/j.biortech.2007.09.057>
112. Lemaire, R., Veuillet, F., Bausseron, A., Chastrusse, S., Monnier, R., Christensson, M., Zhao, H., Thomson, C., Ochoa, J.: ANITA™ Mox deammonification process for COD-rich and THP reject water. 88th Annu. Water Environ. Fed. Tech. Exhib. Conf. WEFTEC 2015. 3, 3266–3279 (2015). <https://doi.org/10.2175/193864715819539849>
113. Han, X., Wang, F., Zhou, B., Chen, H., Yuan, R., Liu, S., Zhou, X., Gao, L., Lu, Y., Zhang, R.: Phosphorus complexation of sewage sludge during thermal hydrolysis with different reaction temperature and reaction time by P K-edge XANES and <sup>31</sup>P NMR. *Sci. Total Environ.* 688, 1–9 (2019). <https://doi.org/10.1016/j.scitotenv.2019.06.017>
114. Qadir, M., Drechsel, P., Jiménez Cisneros, B., Kim, Y., Pramanik, A., Mehta, P., Olaniyan, O.: Global and regional potential of wastewater as a water, nutrient and energy source. *Nat. Resour. Forum.* 1–12 (2020). <https://doi.org/10.1111/1477-8947.12187>