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Improvement of the energy recovery from the digestion of waste activated sludge (WAS) through intermediate treatments: the effect of the hydraulic retention time (HRT) of the first-stage digestion

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Abstract

Reduced biodegradability of waste activated sludge (WAS) limits the production of methane and the consequent energy recovery in an anaerobic digestion (AD) process. Pre-treatments are a solution to increase the biodegradability of bacteria cell biomass, but a large part of poorly degradable organic matter is left after digestion. The utilization of intermediate hydrolysis treatments (IHTs) may help in converting even the most recalcitrant parts of organic matter in methane.

This study employed a three-phase experimentation to assess the effect of the hydraulic retention time (HRT) of the digestion first stage, on the overall performance of a two-stage digestion process, with an in-between treatment, carried out on WAS. The three phases of the experimentation included a first-stage digestion (with HRTs = 5, 10 and 15 days), performed in a semi-continuous 10L-reactor, followed by a thermal (90°C) or a hybrid (thermal 90°C + chemical, 4% NaOH) IHT, completed by a second-stage digestion carried out in a batch mode. Both the digestion processes were performed in mesophilic conditions (38°C).

The obtained results revealed that, in the presence of a thermal IHT and by fixing the duration of the second stage to 20 days, the overall specific methane production (SMP) tended to a constant value, in the order of 0.205 Nm^3/kg VS added, irrespective of the duration of the first stage. Conversely, when a hybrid treatment was applied, the difference between a short (5 days) and a medium (10-15 days) duration of the digestion first stage became evident, with SMPs in the order of 0.247 and 0.230 Nm^3/kg VS added, respectively.

Energy and economic sustainability of the application of IHTs at a full scale plant required an adequate thickening of sludge / digestate matrices and an efficient heat exchange between donor (sludge after treatment) and acceptor (cold sludge before digestion) agents. It was demonstrated that for separated or joined digestion processes of primary sludge (7.0% TS) and treated digestates, with heat recovery and different combinations of the duration of the first and second stage of AD, TS contents in the order of

4%, 6% and 8-9% were required to make the thermal balance neutral for thermal exchanges efficiencies of 100%, 70% and 50%, respectively.

Keywords: intermediate hydrolysis treatments; two-stage digestion; energy balance; economic sustainability; digestate

1. Introduction

Anaerobic digestion (AD) is employed worldwide as the oldest and most important process for stabilization of both primary sludge (PS) and waste activated sludge (WAS) generated in wastewater treatment plants (WWTPs). However, it is well known that the aim of the AD process carried out in the sludge line of a WWTP is not only reducing the volume of sludge and making it stable, but primarily producing an amount of biogas sufficient to self-sustain the AD process and possibly supplying the WWTP and the close users with energy in the form of hot water and electricity. In this context, the reduced biodegradability of WAS represents a drawback because it limits the methane production and volatile solid (VS) reduction. In fact, most of the biodegradable material of WAS is either enclosed in the microbial cell wall or enmeshed in an extracellular polymeric matrix (Grübel and Suschka, 2015).

Various pre-treatment processes (e.g. thermal, mechanical, chemical and biological) have been developed with the aim of increasing the biodegradability of bacteria cell biomass and reducing volume and retention time of digesters. Extensive reviews of the effectiveness of each of the afore-mentioned types of process in terms of resulting properties of treated sludge, digester performance, energy balances, environmental benefits and current state of real-world application are reported among others in Carrère et al., 2010; Cano et al., 2015; Zhen et al., 2017; Wacławek et al., 2019. Pre-treatments, as their name suggests, are performed prior to the process of AD. In this way, the different fractions of organic matter, that is both complex compounds and readily bio-degradable substances (Jimenez et al., 2015), undergo the pre-treatment (Wang and Li, 2016). Therefore, the material that is already available to be converted into methane enters the pre-treatment despite it does not need to and does not benefit from such a treatment (Svensson et al., 2018). After pre-treatment, the treated substrate enters the anaerobic digesters where degradable organic matter is converted into methane, still leaving a large fraction of poorly degradable organic matter, that comes out from the digester and does not fully harness its energy potential (Aragón-Briceño et al., 2017). Some authors hypothesized that, in a digestion process involving WAS, this poorly degradable fraction was associated with the extracellular polymeric substance (EPS), that is released from WAS during digestion because of microbial metabolism, self-protective reaction and cell lysis (Shana et al., 2013; Williams et al., 2015). Consequently, the use of sludge pre-treatment technologies may only help to hydrolyze the EPS in the sludge feedstock but it is not of any benefit for the fraction produced during an AD process.

A possible solution to deal with the EPS generated during sludge digestion and other poorly degradable organic matter fractions is to make use of treatments that follow AD (named post-treatments, inter-stage treatments, intermediate hydrolysis treatments, IHTs). Such treatments only concentrate on the slowly degradable parts of the sludge, in contrast to pre-treatment methods (Shana et al., 2015). In this new configuration, the AD process converts the readily biodegradable organic matter into methane, in the first step, and the residual digestate containing the recalcitrant or more complex material undergoes the IHT in a second phase. The ‘‘inter-treated” digestate enters the anaerobic digester either by recirculating a certain fraction or, as a whole, entering a second-stage AD process. This will reduce the residual organic matter and increase the energy benefit of the treatment (Ortega-Martinez et al., 2016).

Despite the possible advantages, IHTs of WAS have until now received little attention in comparison to pre-treatments. Moreover, due to the nature of the organic matter that remains after an AD process, most part of the recent applications of IHTs have used high energy-demanding processes like hydrothermal treatments (HTTs). The examples reported in the follow point out that some authors have focused only on post-treatments, searching for the best operating conditions to extract the highest amount of biogas from the second stage of digestion. Conversely, a limited number of authors have compared the performances of HTTs used as a pre- or post-treatment.

With reference to the first group of studies, Aragón-Briceño and coauthors (2016) evaluated the effect of the temperature parameter (160, 220 and 250 °C) in HTTs carried out on digestate samples collected from Leeds (UK) WWTP. The performance of the IHTs was studied with respect to product yields (that is the production of hydrochar), biomethane potential and solubilisation of organic carbon. They obtained the highest hydrochar yields at 220°C. The solubilisation of carbon increased from 4.62% in the raw feedstock to 31.68%, 32.56% and 30.48% after HTTs at 160, 220 and 250 °C, respectively. Finally, the HTT enhanced the potential methane production up to 58% for both, the whole fraction (hydrochar + processed water) and processed waters. Bjerg-Nielsen and coauthors (2018) used an anaerobically digested WAS (HRT = 15 days, T = 55°C) from Aalborg Vest WWTP (Denmark), with and without wheat straw as a co-substrate, to study the effect of time (30 and 60 min) and temperature (120–190 °C) on the performances of thermal IHTs. They observed that, compared to non-treatment, IHTs increased the secondary step methane yield from 52 to 222 L CH_4/kg VS and from 147 to 224 L CH_4/kg VS for pre-digested WAS and pre-co-digested WAS respectively, at an optimum of 170 °C and 30 min.

Conversely, Svensson and coauthors (2018) compared the performances of pre- and post-treatments that were carried out in a non-homogeneous way: low temperature (70°C) and HTT (134-175°C), respectively. Specifically, they used a Cambi steam explosion unit to treat digestate samples from source separated food waste (SSFW) digested in thermophilic conditions and from a mixture of sewage sludge treated in mesophilic conditions. The thermal hydrolysis was found to be more efficient, on WAS digestate than on SSFW digestate, in relation to methane yields and dewatering capacity. For temperatures above 165°C, the authors observed increases in volumetric methane yield and COD reduction of 7% and 70% respectively, compared to a conventional pre-treatment carried out at 70°C.

Ortega-Martinez and coauthors (2016) demonstrated that intermediate HTTs could improve the methane production of a mixed sludge by 45% and 20% compared to a conventional AD process and pre-treatments followed by AD, respectively. They carried out pre- and inter-treatments in a laboratory-scale thermal steam explosion system at temperatures from 110 to 200 °C and for contact times of 10, 30 and 50 min. Recently, Yuan and coauthors (2019) compared the efficiency of HTTs (130-210 °C) as pre- and post-treatment on PS and anaerobically digested sludge. They demonstrated that, although post-treatments achieved higher total methane yield and solids reduction, the increment of methane yield was found to be similar through both strategies (pre- and post-treatments) compared to their control counterparts (no HTT). The decomposition of insoluble organic carbon was also similar via both strategies.

Finally, Rennuit and coauthors (2018) tested a thermophilic (55°C) aerobic digestion (TAD) as a pre- and inter-stage treatment of sludge and digestates samples coming from the Ejby Mølle WWTP (Denmark, treating capacity 385,000 population equivalent, p.e.). Digestates were originated from a mixture of primary (60%), dewatered secondary sludge (40%) and highly degradable organic waste (depending on availability). The authors demonstrated that TAD as a pre-treatment decreased the methane yield (up to −70%), due to oxidation losses, whereas, when used as an inter-stage treatment, it slightly improved the overall methane yield (+2.6%) and the total COD removal (+5%) compared to control.

From the review of the existing studies, it can be seen that most part of the processes employed for IHTs are high energy-demanding processes, like HTT Cambi, carried out at 140-180 °C. Moreover, all the mentioned experiences used digested substrates from full scale plants (with a fixed HRT) or from batch tests, in the case a comparison between pre- and post- treatments was carried out. In the specific context of the application of low-temperature IHTs (that is treatments that employ only a limited amount of energy compared to HTTs), the HRT of the AD process that generates the intermediate digestate is a key issue. In fact, the HRT of the digestion first stage affects the amount of biogas produced in the first stage, the state/characteristics of the organic matter after the partial digestion and the volume of substrate to be fed to the second stage. All these elements concur in the biogas production and consequent energy recovery from the whole (two-stage) digestion process.

In a previous study (Campo et al., 2018), the effect of low-temperature thermo-alkali IHTs on two digestate samples with different HRTs (7 and 15 days) was compared. However, the two substrates used for the study had different origin, the Castiglione Torinese full scale WWTP (Italy, 2,000,000 p.e.) and a pilot scale (300-L) digester fed with the WAS collected from the same WWTP, respectively. In the afore-mentioned study, the performances of the IHTs were compared with those of a process that included a first stage of pre-treatment (same operating conditions as the IHT) and a subsequent stage of batch digestion, both carried out on a WAS sample.

The aim of this new study was analyzing the effect of the duration of the first stage of an AD process on the overall energy recovery, firstly in the form of biogas and consequently in the form of electric energy and heat, from a system made of a two-stage AD with an in-between digestate treatment. To achieve the prefixed aim, controlled-HRT (5, 10 and 15 days) digestate samples were produced in a semi-continuous pilot scale apparatus (10 L), subsequently, they were treated (90°C, 4% NaOH, 90 minutes) and, finally, the residual methane production was quantified in a batch AD apparatus. To the best of our knowledge, this is the first study that makes use of a first-stage controlled-HRT digestate to evaluate the energy and economic sustainability of an AD process made of two stages. The results obtained in these tests will subsequently be used to evaluate the future performances of the sludge line of Castiglione Torinese plant, the largest Italian WWTP, once intermediate treatments are implemented. The increase in the energy production, in the form of electricity that can be sold to external users, compared to the present situation, of which the WWTP can benefit from the implementation of the novel IHTs is the key point that will drive the WWTP managers in the decision to go for the renovation of the sludge line.

2. Materials and Methods

2.1 Reactors set up and operation

The scheme of the system used for the study is shown in Figure 1. The study was articulated in three phases, each of which was repeated three times, one for each of the tested HRTs (5-10-15 days). The first phase was a semi-continuous digestion process, followed by an in-between treatment phase of the digestate and completed by a second-stage digestion process carried out in a batch mode. The experimental system consisted of a first semi-continuous anaerobic digester with a useful volume of 10 L, 500-ml jars for IHTs and a series of six 6 L batch anaerobic digesters (4.5 L working volume) for the second-stage AD process.

2.1.1 Phase 1 – Semi-continuous tests on untreated WAS samples

In the first phase, a semi-continuous AD process was run on WAS samples collected from Castiglione Torinese SMAT WWTP (see Section 2.2). The test was carried out in mesophilic conditions (38°C). Once a day, aliquots of digestate were extracted from the digester and, subsequently, samples of fresh WAS were used to feed the reactor, for a number of days equal to at least three times the fixed HRT (5-10-15 days). This procedure was followed in order to reach a steady-state condition into the digester and obtain a digestate product representative of the fixed HRT. The OLR for each of the three HRTs, 5, 10 and 15 days, was of 6, 3 and 2 g TS/l·d, respectively.

At the end of each run, the digester was emptied and the digestate was used for the subsequent operations of IHTs and the second phase of the AD process in the batch apparatus (see Sections 2.1.2 and 2.1.3). Inoculum and fresh WAS (see Section 2.2) were subsequently added to the digester to start a new digestion run with a different HRT.

The temperature in the 10 L semi-continuous reactor was kept at 38°C (mesophilic conditions) by a temperature controlled water jacket. The biogas produced in each of the three digestion runs (HRT = 5-

10-15 days) was collected in a gas bag. The volume of the gas was quantified and characterized daily as described in Ruffino et al. (2016). Briefly, the volumetric composition of the biogas in terms of CH_4, CO_2, O_2 was obtained by flushing 500 mL of biogas through a biogas analyzer (Biogas Check, Geotechnical Instruments Ltd). The residual volume of the biogas after characterization was measured by displacing volumes of water with the residual gas and referring the obtained value to the normal conditions (273.15 K and 101.325 kPa). TS and VS concentrations of both the fed sludge (WAS) and digested product from the 10 L semi-continuous reactor were measured every working day in order to determine the extent of TS and VS destruction.

2.1.2 Phase 2 – Intermediate hydrolysis treatments (IHTs)

After three HRTs, the AD process was stopped, the semi-continuous anaerobic digester was emptied and the digestate was subjected to the second phase of the study. The controlled-HRT digestate was divided into three aliquots of the same volume. One aliquot was subjected to a low-temperature thermal treatment (90 min, 90°C), another to a hybrid (thermo-alkali) treatment (90 min, 90°C, NaOH 4% TS) and the third was used as a reference (untreated) sample. The conditions for the treatments and the protocol followed for the tests came from the results of a previous study that involved samples of digestate generated from the same WAS (Campo et al., 2018). For each of the tested conditions, three replicates were carried out.

The efficacy of the IHTs (low-temperature thermal and thermo-alkali) was preliminarily assessed by using the disintegration rate (DR) parameter, as in Equation (1)

$$DR = \frac{sCOD_l - sCOD_0}{tCOD_0 - sCOD_0} \quad (1)$$

Where:

$sCOD_l$ = soluble COD after the lysis process;

$sCOD_0$ = soluble COD of the untreated sample;

$tCOD_0$ = total COD.

The DR parameter has been largely employed in the literature to evaluate the efficacy of lysis processes (Dohányos et al. 1997). It relates the soluble COD released by the lysis treatment to the COD of the particulate fraction (tCOD–sCOD), that is, the fraction that can be potentially hydrolyzed during the treatment.

2.1.3 Phase 3 – Batch tests on the digestate

After the IHT, each aliquot of treated digestate was divided into two sub-aliquots and subjected to the third phase of the study, that is a second-stage AD in a batch mode. The AD process was carried out in the 6 L batch reactors already used in previous experimentations (see Campo et al., 2017). Treated and untreated digestate was mixed with the inoculum described in Section 2.2., according to a ratio in the order of 1:1 on a VS basis (specifically, digestate : inoculum ratio = 0.99; 1.11 and 1.09 for HRT of 5; 10 and 15, respectively). No buffer agents and no nutrients were added to the digesters.

The biogas produced from each of the batch digester was collected in a gas bag and quantified and characterized as described in Section 2.1.1. Blank tests were carried out in batch reactors with a working volume of 2 liters, in order to evaluate the amount of biogas generated by the inoculum in each experimental run (with 5-, 10- or 15-d digestate). In blank tests the volume of inoculum added to the reactors was the same of that used for the tests with digestates.

2.2 Substrate and inoculum

The sludge used in this study was collected from the secondary settlers of the SMAT WWTP located in Castiglione Torinese (20 km from Turin, NW Italy). Details of the WWTP were provided in a previous paper (Ruffino et al., 2014). The WWTP has a standard configuration: preliminary treatments (grating and sand/oil removal), primary settling, pre-denitrification, biological oxidation with a SRT in the order of 25 days, secondary settling and final filtration on a gravel and anthracite bed.

The WWTP has a treatment capacity of approximately 2,000,000 p.e. and generates 4320 kg TS/h of sludge, 64% of this amount is made of primary sludge. The mass flow rate of WAS is in the order of 1555 kg TS/h, with an average TS content of 0.8%. Pre-thickeners located in the sludge line of the WWTP increase the solid content (TS) of the WAS from 0.8% to approximately 2-3%.

Ten liters of WAS were collected weekly, for the whole duration of the study, and were used to feed the semi-continuous 10 L digester. The testing conducted in this study spanned approximately nine months (270 days) of the WWTP operation and hence it was anticipated that there were seasonal variations in the raw sludge properties. As shown in Figure 2, the ratio between volatile and total solids (% VS/TS), from the beginning to the end of the study, was appreciably variable. VS/TS ratio was in the order of 70-71% during the first digestion run (HRT = 5 days), it decreased to 66% during the second digestion run (HRT = 15 days) and, finally, it rose to 68% during the third digestion run (HRT = 10 days).

Inocula employed for the semi-continuous and batch tests were obtained from a digester of the WWTP that was fed with sole primary sludge. The average TS content of the inoculum aliquots used for the experimental runs was in the order of 2%, the VS/TS ratio was of 58%. The inoculum aliquots were considered ready for the experimentation when their daily biogas production was of less than 1% of the overall production recorded during the whole period of permanence into the digesters used for the experimentation (VDI 4630, 2006).

2.3 Analytical methods

All the analytical parameters monitored in the tests (TS, VS, pH, electric conductivity (EC), soluble COD (sCOD) were determined according to Standard Methods (APHA, AWWA, WEF, 2012). Soluble COD is the fraction of COD separated after a centrifugation at 4000 rpm for 15 min and a subsequent filtration on a 0.45 mm nylon membrane, as recommended by Roeleveld and van Loosdrecht (2002).

The FOS/TAC parameter was obtained by a potentiometric titration, according to the Nordmann method (Nordmann, 1977), by using a SI Analytics automatic titrator. FOS/TAC parameter is the ratio between volatile organic acids and alkaline buffer capacity; FOS/TAC is an easy-to-do and reliable measure of the risk of acidification of a biogas plant.

3. Results and Discussion

3.1 Phase 1 – Semi-continuous tests on untreated WAS samples

Figure 3 presents the evolution of the specific methane production (SMP) from the semi-continuous first-stage digester in the three digestion runs (HRTs = 5, 10 and 15 days). It could be observed that in all cases a steady production of methane was obtained after three HRTs. Values of SMP and VS reduction from the three digestion runs, and pH and FOS/TAC values of the digestate samples with HRT of 5, 10 and 15 days are reported in Table 1.

Table 1. Values of SMP and VS reduction from the three digestion runs, and pH and FOS/TAC values of the digestate samples

HRT (d)	SMP Nm^3/kg VS added	VS reduction (%)	pH	FOS/TAC
5	0.069±0.001	13	7.29±0.06	0.12±0.02
10	0.095±0.001	22	7.35±0.13	0.11±0.01
15	0.109±0.001	26	7.35±0.09	0.11±0.01

The standard deviation of the specific production of methane from the average value was calculated over the period in which a steady production was observed (see the circles in Figure 3). From these results it can be concluded that the HRT of the first stage of the AD process played a fundamental role in the methane production, especially for a low biodegradable substrate such a sludge from biological origin.

Both pH and FOS/TAC values indicated a good stability of the digestion process, even for short HRTs and without the addition of buffering agents.

Sludge samples collected from the same WWTP and digested in a 300 L semi-continuous apparatus, working with a HRT of 20 days, generated a SMP in the order of 0.120 Nm^3/kg VS added (Campo et al., 2018). During the test carried out at a pilot scale (300 L) the average VS reduction was in the order of 19%. In a similar experience carried out in a semi-continuous reactor (OLR = 2.1 gVS/l·d, HRT = 15 days), Wei and coauthors (2018) observed a SMP of 0.132 ± 0.005 m^3/kg VS added and a VS reduction of 29.2 ± 0.9% for an untreated WAS. Leite and coauthors (2017) measured a SMP of 0.067 Nm^3/kg VS added in a semi-continuous mesophilic reactor (HRT = 7 days, ORL = 1.9 kg VS/m^3·d) fed with a WAS with a VS/TS ratio in the order of 70%. In that experience $NaHCO_3$ was added to the fed sludge in order to prevent bacteria inhibition or digester souring in the subsequent phase of thermophilic digestion. In a test where sonication was used to pre-treat WAS before a semi-continuous AD process, Córdova Lizama and coauthors (2018) obtained daily biogas productions for the raw WAS of 0.040 L/d and 0.033 L/d for OLRs of 1 and 3 kg VS/m^3·d respectively, that correspond to approximately 0.050 – 0.014 m^3/kg VS fed in a 0.8 L working volume reactor. The production recorded for a HRT of 10 days (0.037 L/d or 0.023 m^3/kg VS fed) was approximately five times lower than that of this study.

Table 2. Characteristics of the digestate samples used for the IHTs and the second-stage digestion process and results of the intermediate treatments

HRT (d)	TS (%)	VS/TS (%)	sCOD (mg/l)	thermal IHT DR (%)	hybrid IHT DR (%)
5	3.37±0.20	66.74±0.36	1005±9	21.5±1.1	32.1±1.3
10	2.65±0.07	63.72±0.12	453±6	14.5±0.8	29.2±1.5
15	2.65±0.05	61.29±0.46	823±7	13.3±0.6	19.4±1.0

3.2 Phase 2 – Intermediate hydrolysis treatments (IHTs)

The amount of TS, VS, sCOD in the three samples of digestate used for IHTs and for the second-stage digestion process are shown in Table 2. The effect of thermal and hybrid treatments on the samples of digestate collected from the 10-L semi-continuous digester was preliminarily evaluated by using the DR parameter. DR quantifies the sCOD released after the treatment with respect to the maximum amount of potentially hydrolysable COD (that is the particulate COD). Table 2 resumes the effect of IHTs in terms of DR values. From these results, it could be seen that, on average, the only thermal treatment was less efficient in COD solubilizing than the hybrid treatment. The efficiency of both thermal and hybrid treatments decreased with increasing HRTs, consequently, the degree of solubilization seemed to be dependent on the feedstock (Svensson et al., 2018). IHTs had probably a weaker effect on more stable structures, like those that are formed during an AD process (Rennuit et al., 2018). During an IHT, heat and/or alkali changed the floc structure of the digestate, caused the rupture of cell walls and/or extracellular polymeric substances (EPS) with the release of intracellular and/or extracellular dissolved organic compounds to the aqueous phase. These organic compounds are carbohydrates, proteins, lipids and VFAs, that have been proposed to be responsible for the significant increases in sCOD after treatment (Bjerg-Nielsen et al., 2018).

DRs found in this work were similar to the values that were reported for sewage sludge (Ruffino et al., 2016) and for digestates in previous studies (Campo et al., 2018; Ortega Martinez et al., 2016). Ortega

Martinez and coauthors (2016) reported solubilization degrees in the order of 25-40% for digestate samples treated in a laboratory-scale thermal steam explosion system. However, a limited number of information on the variation of the solubilization degree of digestate sludge subjected to IHTs is available in the published literature.

3.3 Phase 3 – Batch tests on the digestate

In the third phase of the experimentation, the residual methane production of the digestates generated in Phase 1, subsequently subjected to thermal or hybrid IHTs, was determined in a batch apparatus. According to VDI 4630 rule (2006), the batch AD test was considered concluded when the daily volume of the produced biogas was 1% or less of the volume produced during the whole test. All the batch tests had a duration of approximately 25 days.

Figure 4 compares the overall SMP produced by the three systems (first stage HRT = 5, 10, 15 days). Each bar combines the SMP obtained in the first-stage semi-continuous digestion (red bar), the SMP obtained in the second-stage batch digestion from the untreated digestate and the SMP increments (incr) due to the only thermal or hybrid IHT carried out on the digestate samples. It is necessary to point out that the SMP obtained from the second-stage digestion was referred to the overall production (25 days) of the batch tests, until exhaustion of the methane generation of the substrate (VDI 4630 rule, 2006).

It can be seen that the SMP from the second-stage batch digestion of untreated 5-, 10- and 15-day digestates was of 0.096±0.003, 0.072±0.002 and 0.053±0.003 Nm^3 CH_4/kg VS added, respectively. Not surprisingly, digestates with a lower HRT in the first stage had a methane production higher than that of the digestates that underwent longer digestion periods. In fact, as said in Section 3.1, the first-stage digestion process consumed only 13%, 22% and 26% of the VS preliminarily available in the sludge samples subjected to 5-, 10- and 15-day HRT digestion, respectively. The SMP values generated from digestates in batch AD tests, after thermal IHTs, were of 0.135±0.006, 0.112±0.004 and 0.100±0.004 Nm^3 CH_4/kg VS added. Consequently, it could be stated that the only thermal treatment determined an increase in the second-stage SMP of 41%, 56% and 89%, with respect to the untreated digestate, for 5-, 10- and 15-day digestates, respectively. Recently, Bjerg-Nielsen and coauthors (2018) observed that thermal IHTs carried out at 120°C for 60 min on a predigested sludge (HRT = 15 days, thermophilic conditions) determined a triplication in the SMP, that passed from 0.052 to 0.147 m^3/kg VS added.

Conversely, the SMP values that resulted from the digestates treated with the hybrid IHT were of 0.178±0.008, 0.136±0.004 and 0.120±0.007 Nm^3 CH_4/kg VS added. The hybrid treatment determined an increase in the SMP of 85%, 89% and 126%, in comparison with the untreated digestate, for 5-, 10- and 15-day digestates, respectively. Hybrid IHTs proved to be more efficient in releasing biodegradable substances and subsequent methane generation in the second-stage AD process than the only thermal treatment. These observations were in agreement with the results obtained in a previous study (Campo et al., 2018) carried out on WAS and digestates samples collected from the same WWTP and tested in AD processes after low-temperature thermal and hybrid pre- and intermediate treatments. Compared with IHTs carried out in other studies, the increase found in the present study was high. Takashima and Tanaka (2014) observed increases in methane production in the order of 14-21% after acidic thermal post-treatments carried out for 1 h at 170°C and pH 5-6. Rennuit and coauthors (2018) in a recent study found very small increases in methane production (in the order of 2-3%) after aerobic biological inter-stage treatments.

The comparison among the three systems (first-stage HRT = 5, 10, 15 days) showed that the production developed in the second-stage AD, irrespective of the presence of an IHT, tended to bring the overall production (first plus second stage) to an approximately constant value. In fact, the sum of the production of the two stages, without an IHT, was of 0.165, 0.167 and 0.162 Nm^3 CH_4/kg VS added for the systems characterized by a first-stage HRT equal to 5, 10 and 15 days, respectively. In the case of a thermal IHT, the overall SMP values were of 0.204, 0.207 and 0.209 Nm^3 CH_4/kg VS added for the systems characterized by a first-stage HRT equal to 5, 10 and 15 days, respectively. Finally, where a hybrid IHT was applied to the digestate samples, the overall SMP values were of 0.247, 0.231 and 0.229 Nm^3 CH_4/kg VS added, respectively. Only in this last case the difference between a short and a medium duration of the first-stage AD on the overall SMP became evident. As it can be seen in Figure 5, the combination of the two digestion processes with an in-between hybrid treatment, determined an overall VS removal of 45%, 44% and 44% for the systems with a first-stage HRT equal to 5, 10 and 15 days, respectively. Digestates extracted and characterized after the second-stage digestion process had an average VS/TS ratio in the order of 55% (data not shown). The systems that included the thermal treatments experienced an overall VS removal of 37%, 40% and 41% with a final digestate VS/TS ratio of 58%, 56% and 54% for first-stage HRTs equal to 5, 10 and 15 days, respectively (data not shown).

These results demonstrated that a shorter first phase of the AD process (i.e. in the order of five days) was more profitable in terms of the overall SMP than a longer one. This result became more evident if the overall SMP was obtained by summing the SMP generated in the first-stage process (with a duration of 5, 10 or 15 days), with the SMP obtained after having stopped the second-stage digestion after a number of days equal to 15, 10 and 5, respectively, so that the overall duration of the two-stage AD process was of 20 days. In this way, it could be seen (Figure 6) that, for the untreated digestate samples, the useful SMPs were of 0.091, 0.059 and 0.028 Nm^3 CH_4/kg VS added, only 95%, 82% and 53% of the values obtained at the end of the tests. That determined overall SMPs, after a 20-day, two-stage digestion, of 0.160, 0.154 and 0.137 Nm^3 CH_4/kg VS added for the systems with a first-stage HRT of 5, 10 and 15 days, respectively.

It can be pointed out that the above-reported overall SMPs for the untreated WAS samples were obtained by summing the SMP generated in a semi-continuous (SC) test and the SMP generated in the subsequent batch (B) test, with different durations, that is (5-d SC + 15-d B), (10-d SC + 10-d B) and

(15-d SC + 5-d B). Consequently, values of SMP that increased from 0.137 to 0.160 Nm^3 CH_4/kg VS added, by combining shorter first-stage HRTs and longer durations of the batch test, were obtained for the same substrate (untreated WAS). The observed trend was in good agreement with the well-known result that batch tests produce from 15 to 30% more biogas than semi-continuous tests with the same duration in terms of HRT (Ruffino et al., 2015b; Zhang et al., 2013; Zupančič and Jemec, 2010).

For the same digestion conditions considered before, that is (5-d SC + 15-d B), (10-d SC + 10-d B) and (15-d SC + 5-d B), with an in-between thermal treatment, the overall SMPs, after a 20-day, two-stage digestion, were of 0.204, 0.194 and 0.196 Nm^3 CH_4/kg VS added, respectively. Finally, the overall SMPs, after a 20-day, two-stage digestion, with an in-between hybrid treatment, were of 0.244, 0.211 and 0.213 Nm^3 CH_4/kg VS added.

Tests carried out on a raw WAS sample, collected from the same WWTP, returned a SMP of 0.274 Nm^3 CH_4/kg VS added when treated with a hybrid (90°C, NaOH 4% TS content, 90 minutes, same conditions of this work) pre-treatment and digested in the same batch apparatus used in this work (Campo et al., 2018). In the same study, a sample of digestate that came from a 7-day digestion process in the WWTP digesters and was subsequently subjected to a hybrid IHT (90°C, NaOH 4% TS content, 90 minutes), produced an overall methane amount of 0.317 Nm^3/kg VS added. The result obtained in the present study for the most promising system (0.244 Nm^3 CH_4/kg VS added, first-stage HRT = 5 days) was well below the values obtained in the previous experience, that is -11% if compared with the pre-treatment and -23% if compared with the IHT. This demonstrates that batch tests were useful to make a comparison between systems subjected to different treatment conditions but they were not adequate to return a methane production value that can be representative of the productions of a plant working in (semi-) continuous conditions.

3.4 Future application of IHTs to the sludge line of a full scale WWTP

3.4.1 Technical assessments

In this section a technical analysis based on heat balances was carried out to evaluate the effects of the introduction of IHTs in the sludge line of Castiglione Torinese SMAT WWTP. The three scenarios schematized in Figure 7 were considered for the analysis. In Scenario 1 and Scenario 2 primary and secondary sludge are digested in separated lines. More in detail, in Scenario 1 the heat from the IHT of secondary sludge is recovered for heating primary sludge before digestion. Conversely, in Scenario 2, no heat recovery is applied. Finally, Scenario 3 considers that the primary sludge and the (X-day HRT) digestate from the secondary sludge are mixed after the IHT, and the mixture of the two substrates is subsequently digested in a reactor with HRT equal to 20 days. Both first- and second- stage digesters, for all the considered Scenarios, work in mesophilic conditions (38°C).

First of all, a "zero" Scenario, that refers to the current operating conditions of the sludge line in the WWTP, was presented and its performances were evaluated. Currently, the six 12,000 m^3 anaerobic digesters of Castiglione Torinese SMAT WWTP treat the sludge that come from both primary and secondary settlers. The ratio between primary and secondary sludge, generated in the water line and subsequently digested, is 65:35 on a TS basis (see Section 2.2).

At present, three of the five working digesters (one is periodically in maintenance) are employed for primary sludge and two for secondary sludge. As demonstrated in a previous study (Ruffino et al., 2014), the present modality of management of the sludge line, that includes a preliminary thickening with gravity devices able to thicken sludge to a final TS content of approximately 3.0% in both primary and secondary sludge, and a subsequent AD with no pre- or intermediate treatments, does not guarantee the complete thermal self-sustainment of the digesters, especially during the cold season.

Figure 8 shows the TS content that should be reached in both primary and secondary sludge, through an enhanced thickening process, in order to obtain the complete thermal self-sustainment of the digesters.

The key data used for this assessment were:

- volumes and characteristics of primary and secondary sludge currently produced and treated in the WWTP, that are listed in Table 3;
- SMP of primary and secondary sludge fixed to 0.280 and 0.090 Nm^3/kg VS, respectively. These data were obtained from measurements of biogas and methane production, on the WWTP digesters fed with primary and secondary sludge, that have been carried out daily for approximately three years;
- the electrical and thermal efficiency of the combined heat and power (CHP) unit used in the WWTP that was of 41.9% and 42.4%, respectively;
- HRT of one-stage digesters fixed to 20 days;
- three values of heat exchange efficiency (50-70-100%) referred to the transfer of the heat generated in the CHP unit to the digesters.

Table 3. Volumes and characteristics of primary and secondary sludge currently produced and treated in the WWTP

	Primary sludge	Secondary sludge
Volumetric flow rate (m^3/h)	39.5	51.8
Mass flow rate (kg TS/h)	2765	1554
VS/TS ratio	0.72	0.69
Sludge average temperature (°C)	15	15

As shown in Figure 8, the analysis revealed that TS contents of 4.5%, 6.5% and 9.1%, in both primary and secondary sludge, were necessary to make the thermal balance of the sludge line neutral in the case of heat exchange efficiencies of 100%, 70% and 50%, respectively.

The thermal and economic assessment of the three Scenarios that consider the introduction of IHTs was carried out by considering that the primary sludge is digested with a HRT of 20 days as it is, after being thickened to 7% TS. This value came from the results of the analysis carried out on the “zero” Scenario that revealed that the self-sustainment of the sludge line, under a mid-thermal efficiency condition (70%), could be obtained provided that both primary and secondary sludge were thickened to a TS content in the order of 7%.

This assessment considers that the secondary sludge is partially digested for X days (with X = 5, 10 and 15 days, the three HRTs considered in this study), subsequently the first-stage digestate undergoes a IHT and, finally, it is digested in a second-stage process with a (20-X)-day HRT. For the assessment, the TS content of secondary sludge was made to change from 3% to 11% and the efficiency of the thermal exchange was fixed to 50%, 70% and 100%, considering that the heat produced from the biogas had to be used for the IHTs and the self-sustainment of the digestion processes in mesophilic conditions. Furthermore, it could be demonstrated that, in the present conditions, the heat lost in the sludge/digestate treatment, that is the heat necessary to compensate the exchange with the external environment and to maintain the requested temperature (90°C) for all the duration of the treatment (90 minutes), was only a small fraction (less than 1%) of the heat required for the process (Ruffino et al., 2014).

Figure 9, 10 and 11 and Table S1, S2 and S3 refer to Scenario 1, 2 and 3, respectively. Letters A, B and C in Figures 9, 10 and 11 refer to HRTs of the first-stage digestion of 5, 10 and 15 days, respectively.

Figure 9 highlights two main outcomes of the analysis. On the one hand, it could be observed that the efficiency with which the heat produced from the CHP unit was transferred to the processes that need heating (IHTs and digestion processes) heavily affected the performances of the whole system. On the other hand, irrespective of the thermal exchange efficiency, the solution that considered a 5-day long first-stage digestion, followed by an IHT and completed by a final 15-day digestion, for secondary sludge, proved to be the most efficient. More in detail, in the presence of a thermal exchange efficiency of 100%, a TS content of secondary sludge from 4.1 to 4.3%, depending on the combination of the duration of the first and second stage of AD, was sufficient to make the thermal balance neutral. If the thermal exchange efficiency decreased to 70% or 50%, the TS content had to be increased to approximately 6% or 8%, respectively. It has to be taken into account that, in the case of IHTs application, only the second-stage digestion process could benefit of the improved flowing capacity of the sludge samples that originated from the application of hybrid treatments. In a previous study, it was demonstrated that thermal treatments, carried out at 90°C for at least 90 minutes, made the flowing capacity of a 9% TS sludge equal to the one of a sludge with half TS content (Ruffino et al., 2015a). It is worth mentioning that a Cambi process can easily work with sludge with a TS content in the order of 16-17% (Svensson et al., 2018).

Not surprisingly, the results of Scenario 2 (shown in Figure 10), compared to those of Scenario 1, highlighted that when the heat recovered from the IHT was not used to heat the primary sludge before digestion, higher TS contents were required to make the thermal balance neutral. Theoretically, in the case of a thermal exchange efficiency of 50%, the TS content of secondary sludge rose to values in the order of 25%.

Results of Scenario 3, shown in Figure 11, were quite similar to those of Scenario 1 in terms of TS content of secondary sludge necessary to make the thermal balance of the overall system neutral. The required TS content of secondary sludge was of approximately 4.2, 6.1 and 8.7% for thermal exchange efficiencies of 100%, 70% and 50%, respectively. Even in this case, due to the weight of the thermal exchange efficiency on the overall process, the distribution of the length of the first and second stage of the AD process was only a minor issue in defining the TS content of secondary sludge. Notwithstanding the similarities of Scenario 1 and Scenario 3 in the TS content of secondary sludge, Scenario 3 had to be preferred to Scenario 1 because of the best conditions that could be found in the second-stage digester. In fact, it is necessary to mention the effect of hybrid processes on ammonia release (Kuglarz et al., 2013; Ruffino et al., 2016) and final pH of the treated substrates. The mixing with primary sludge helps in controlling ammonia concentration, adjusts pH to neutral values and dilutes the load of sodium ions introduced with the NaOH used for the treatment (Pinto et al., 2016; Sarwar et al., 2018).

3.4.2 Economic balance

The economic assessment carried out in this section of the study was aimed to:

- identify the cost of the initial investment that can be borne by the WWTP and recovered after a fixed time because of the gain that would derive from the application of the novel IHTs under Scenarios 1 or 3;
- compare the revenues that the WWTP presently obtains from the production of electricity with the gain that would derive from the implementation of one of the new Scenarios (1 or 3).

The Net Present Value (NPV) method was employed for the economic analysis. The NPV is the summation of the present value of a series of present and future cash flows as shown in Equation 2. Because NPV accounts for the time value of money, NPV provides a method for evaluating and comparing products with cash flows spread over many years.

$$NPV(i,N) = \sum_{t=0}^{N} \frac{R_t}{(1+i)^t} \quad (2)$$

Where

t - time of the cash flow;

i – discount rate, i.e. the return that could be earned per unit of time on an investment with similar risk;

R_t – net cash flow (i.e cash inflow minus cash outflow), at time t

In this study the unknown cost of the initial investment (I_0) was evaluated by using Equation 3 and assuming a pay-back time of three or five years

$$NPV(i,N) = -I_0 + \sum_{t=0}^{N} \frac{R_t}{(1+i)^t} \quad (3)$$

Where the time of cash flow and the discount rate were fixed to 20 years and 5%, respectively.

For the estimation of cash inflows and outflows, the four items listed in the follow were considered:

1. the cost of NaOH used for the application of the novel IHTs, fixed equal to 0.450 €/kg (Solvay 2018);
2. the avoided cost of the methane, used as an auxiliary fuel, of which the WWTP must presently be supplied to keep the energy balance of the sludge line neutral, especially in the worst weather conditions – i.e. winter time (Ruffino et al., 2014);
3. the cost of energy to thicken the sludge in a dynamic centrifuge and, finally,
4. the bonus granted for the production of green energy.

With reference to item n. 3, it has to be considered that TS contents in the order of 6% or more, like those required to make the energy balance neutral under Scenario 1 or 3, can be obtained only by using specific dynamic thickeners. The average volumetric flow rate of sludge generated in the WWTP (approximately 540 m^3/h), with their average TS content (from 0.8 to 2.5% TS, depending on the sludge type, primary or secondary), requires a rotary screw thickener with a specific power of 35 W/m^3. This piece of equipment consumes on average 0.03 kWh/m^3 to thicken sludge to TS values up to 7%. It is worthwhile to note that the hourly unit cost for the energy required for the dynamic thickener is of approximately one order of magnitude less than the hourly unit cost required for the NaOH used in IHTs (2.35 €/h vs. 28.0 €/h).

With reference to item n. 4, it must be mentioned that in Italy the electric energy produced from renewable sources benefits from incentives. According to DM 6/07/2012, biogas from AD of sewage sludge that fuels endothermic engines is included in renewable sources. From 2016, the bonuses granted for the production of green energy are calculated by using GRIN application (GSE, 2018). According to this method, the incentive rate (IR) is calculated by the formula

$$IR = k \cdot (180 - Re) \cdot 0.78$$

Where

- 180 is the reference value of a green certificate (equal to 180 €/MWh);
- Re is equal to the sale price of electricity defined by the Authority annually on the basis of the economic conditions recorded on the market in the previous year. For year 2016 Re was of 42.38 €/MWh;
- k is a constant, the value of which depends on the type of used renewable source; for the case considered in this study k was equal to 0.8.

The WWTP considered in this study will be having access to incentives until May 2023. Furthermore, for the economic analysis here reported, the value of Re was kept constant and equal to the value fixed (42.38 €/MWh) for 2016 (i.e. the first year in which GRIN application started to be used).

From the outcomes of the economic analysis, as can be seen in Figure 12, it resulted that the WWTP could bear investments in the order of 4.7 M€ and 7.2 M€, that could be recovered after three or five years respectively, to enhance the performances of the sludge line. The change of the curves' slope recorded after 2023 depended on the expiration of incentives.

In the current operating condition, the AD process carried out on the sludge from the WWTP generated 2730 kWe. Future potential increases in the amount of produced energy, that could derive from the application of IHTs under Scenario 1 or Scenario 3, and in the consequent revenues from the produced electricity are detailed in Table 4.

Table 4. Increases in the amount of produced energy and in the revenues from the produced electricity deriving from the application of IHTs under Scenario 1 or Scenario 3

Scenario	Energy increase (%)	Revenue increase (%)	AD stage I duration (d)	AD stage II duration (d)
1	25.3	34.3	5	15
1	19.9	28.2	10	10
1	20.2	28.6	15	15
3	25.8	34.8	5	20
3	23.1	31.9	10	20
3	22.8	31.5	15	20

It can be seen from Table 4 that, under both Scenarios, the introduction of IHTs determined an average energy increase in the order of 20%; the consequent increase in the revenues from the production of electricity was of 30% or more.

Conclusions

This study wanted to analyze the effect of the duration of the first-stage AD on the overall performance of a system made of two stages of digestions with an in-between digestate treatment. The three digestates with controlled HRTs (5 – 10 and 15 days) were obtained by using a 10L semi-continuous mesophilic reactor.

The obtained results demonstrated that:

- A two-stage digestion, with a low-temperature (90°C) thermal IHT, produced a SMP in the order of 0.205 Nm^3/kg VS added, irrespective of the duration of the first stage, when the duration of the second-stage digestion was fixed to 20 days.
- In the presence of an IHT, the difference between a short (5 days) and a medium (10-15 days) duration of the first-stage digestion became evident, with SMPs in the order of 0.247 and 0.230

Nm^3/kg VS added, respectively. Even in this case the duration of the second-stage digestion was fixed to 20 days.

- When the duration of the overall process was fixed to 20 days (i.e. not all the methane generated in the batch tests was used to calculate the overall production), the effect of the first-stage HRT was amplified, with a SMP reduction, from short to medium first-stage durations, in the order of 15%.
- Thermal sustainability of the application of IHTs at a full scale plant required an adequate thickening of sludge and an efficient heat exchange between donor (sludge after treatment) and acceptor (cold sludge before digestion) agents. In the case of separated digestion of raw primary, thickened to 7%, and treated digestate from secondary sludge (Scenario 1), a TS content of secondary sludge in the range 4.1-4.3%, depending on the combination of the duration of the first and second stage of AD, was required to make the thermal balance neutral, with a thermal exchange efficiency equal to 100%. If the thermal exchange efficiency decreased to 70% or 50%, the TS content had to be increased to 6% or 8%, respectively.
- When primary sludge (7%) was mixed with the treated digestate from secondary sludge to undergo a final 20-day digestion (Scenario 3), TS contents of secondary sludge similar to those of Scenario 1 (that is of 4.2, 6.1 and 8.7%, for thermal exchange efficiencies of 100%, 70% and 50%, respectively) were required.
- Irrespective of the considered Scenario (1 or 3), the introduction of IHTs in the WWTP would determine an average energy increase in the order of 20% and a consequent increase in the revenues from the production of electricity of 30% or more.

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Figure

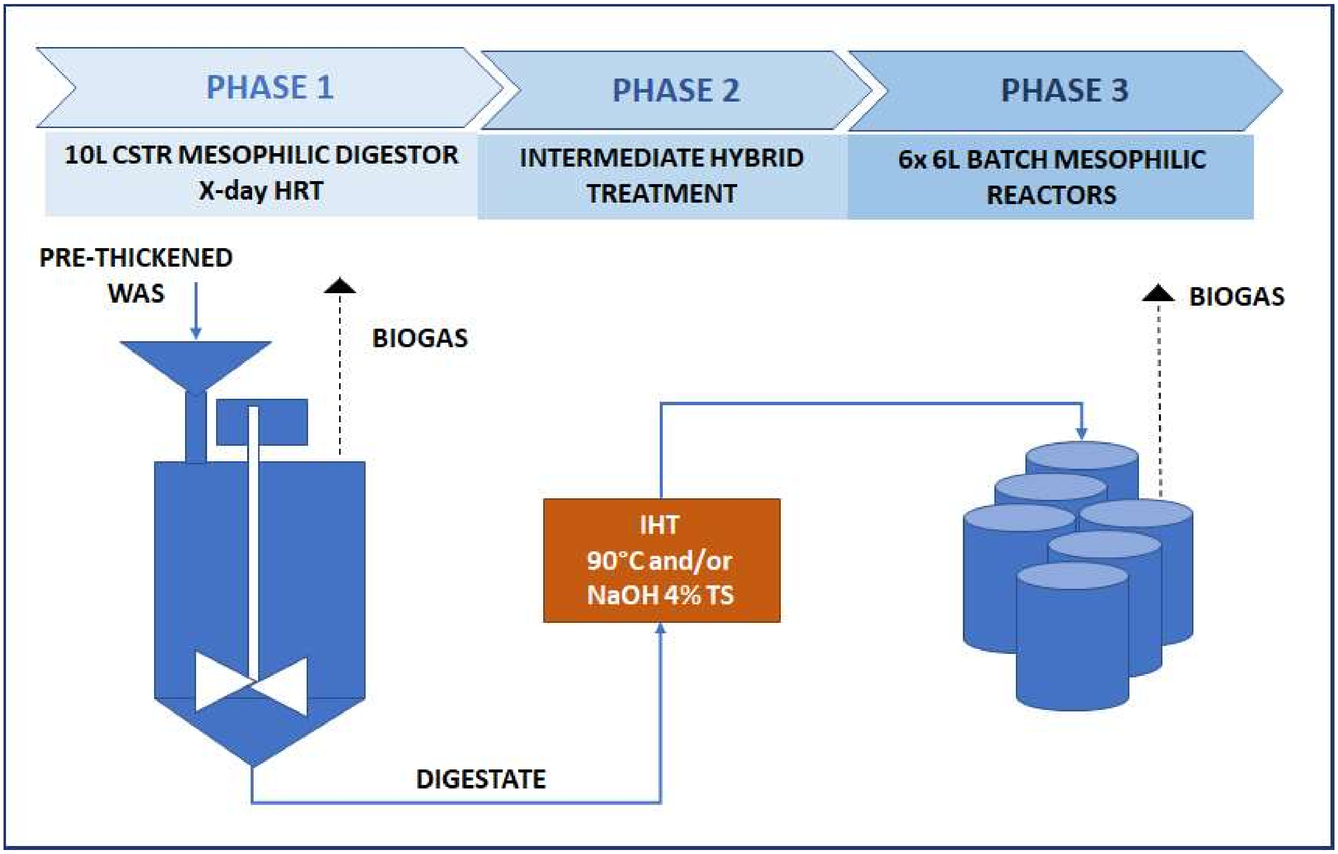

Figure 1. Scheme of the system used for the study

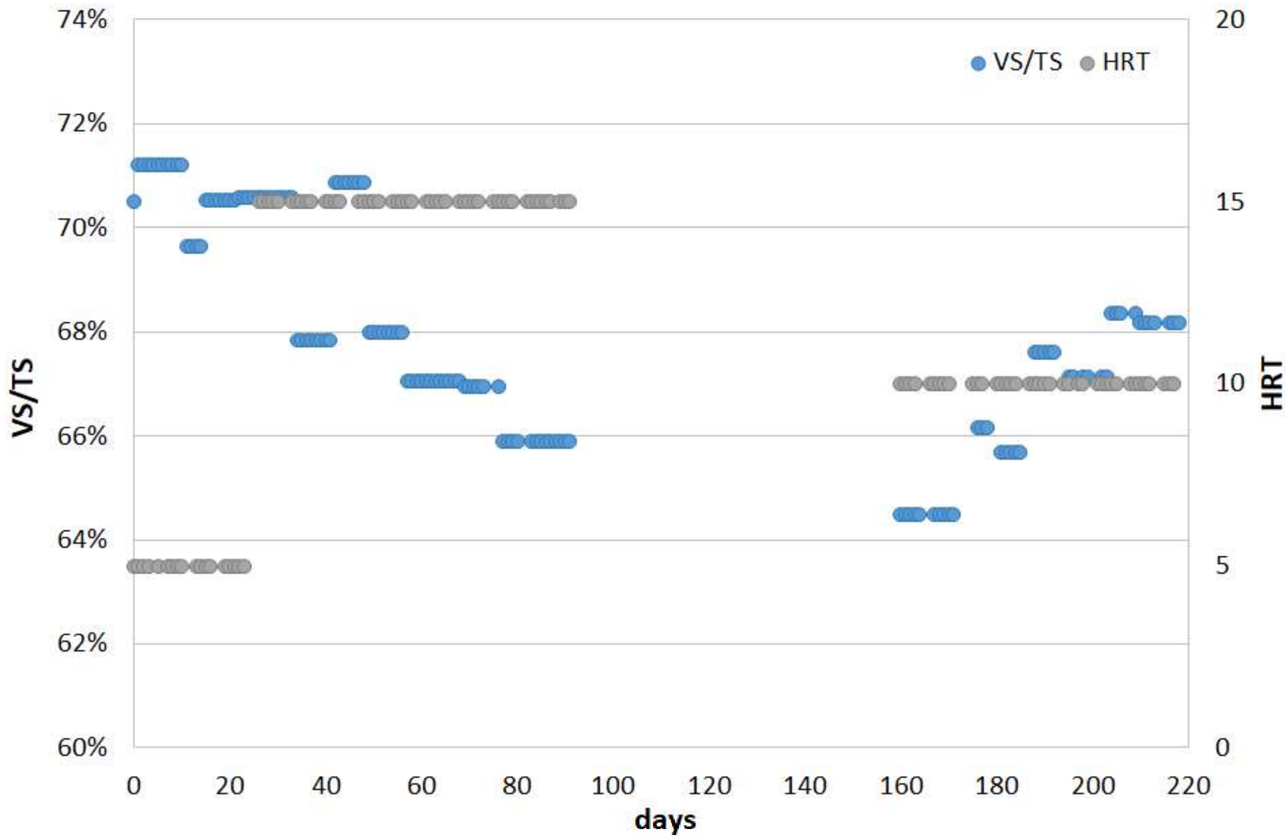

Figure 2. Ratio between volatile and total solids (% VS/TS) for the whole duration of the study

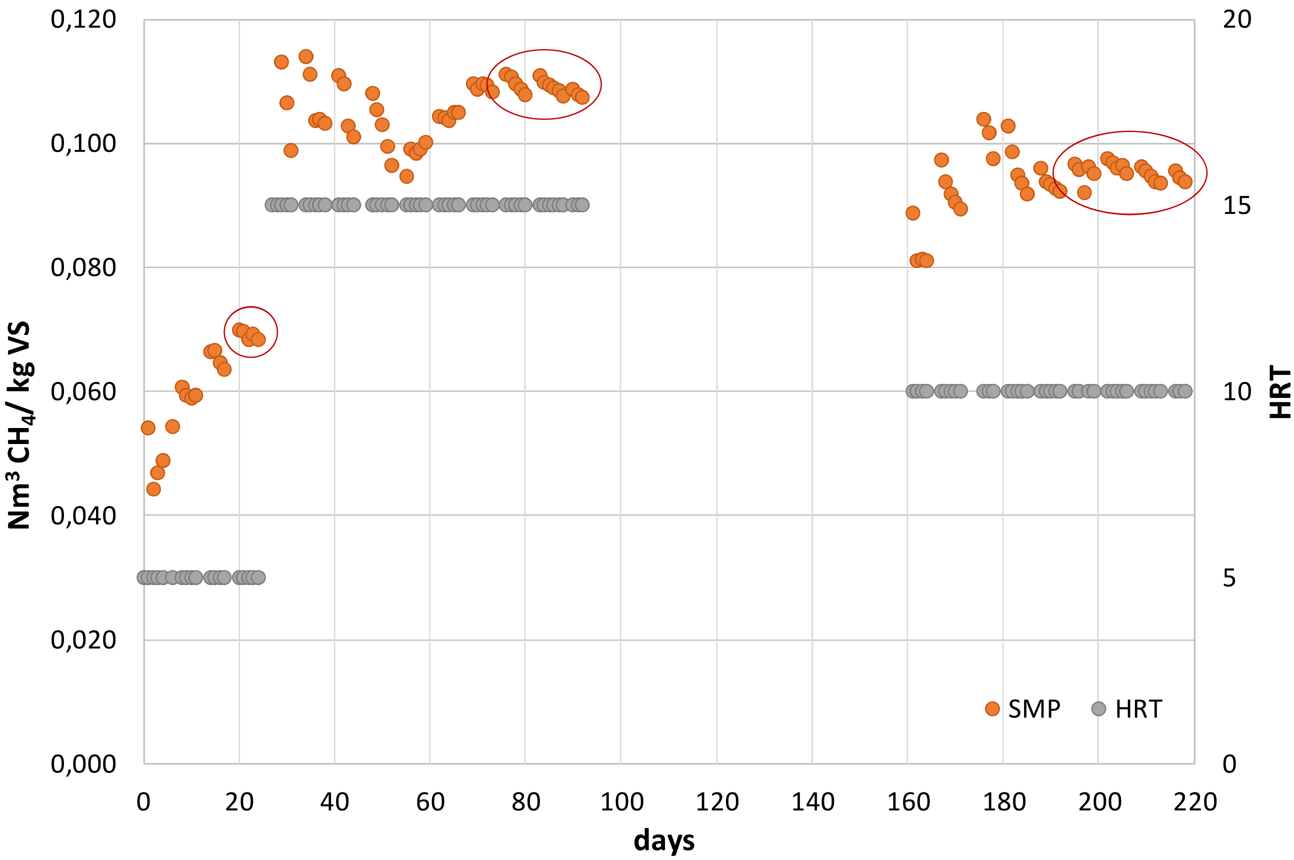

Figure 3. Evolution of the SMP from the semi-continuous first-stage digester in the three digestion runs (HRTs = 5, 10 and 15 days)

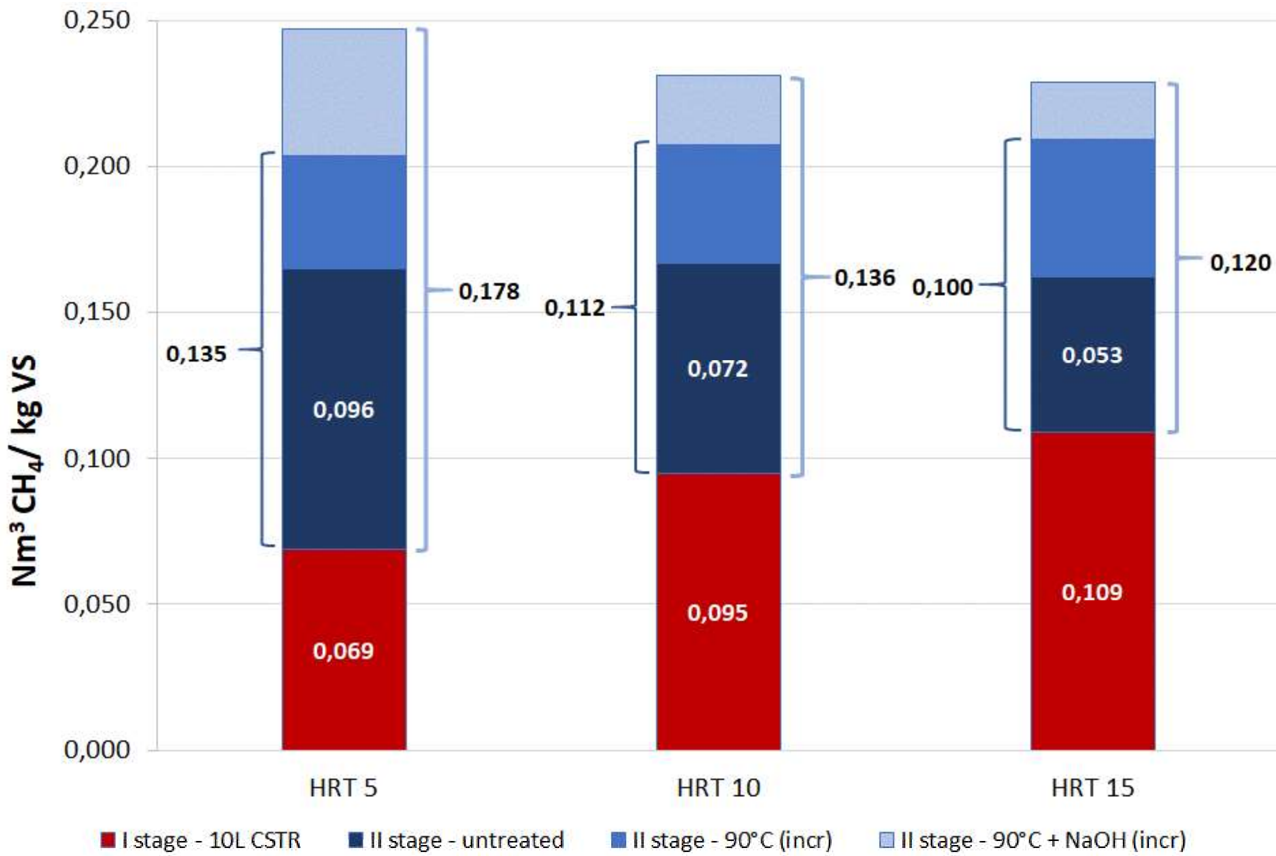

Figure 4. Overall SMP generated by the three systems (first stage HRT = 5, 10, 15 days)

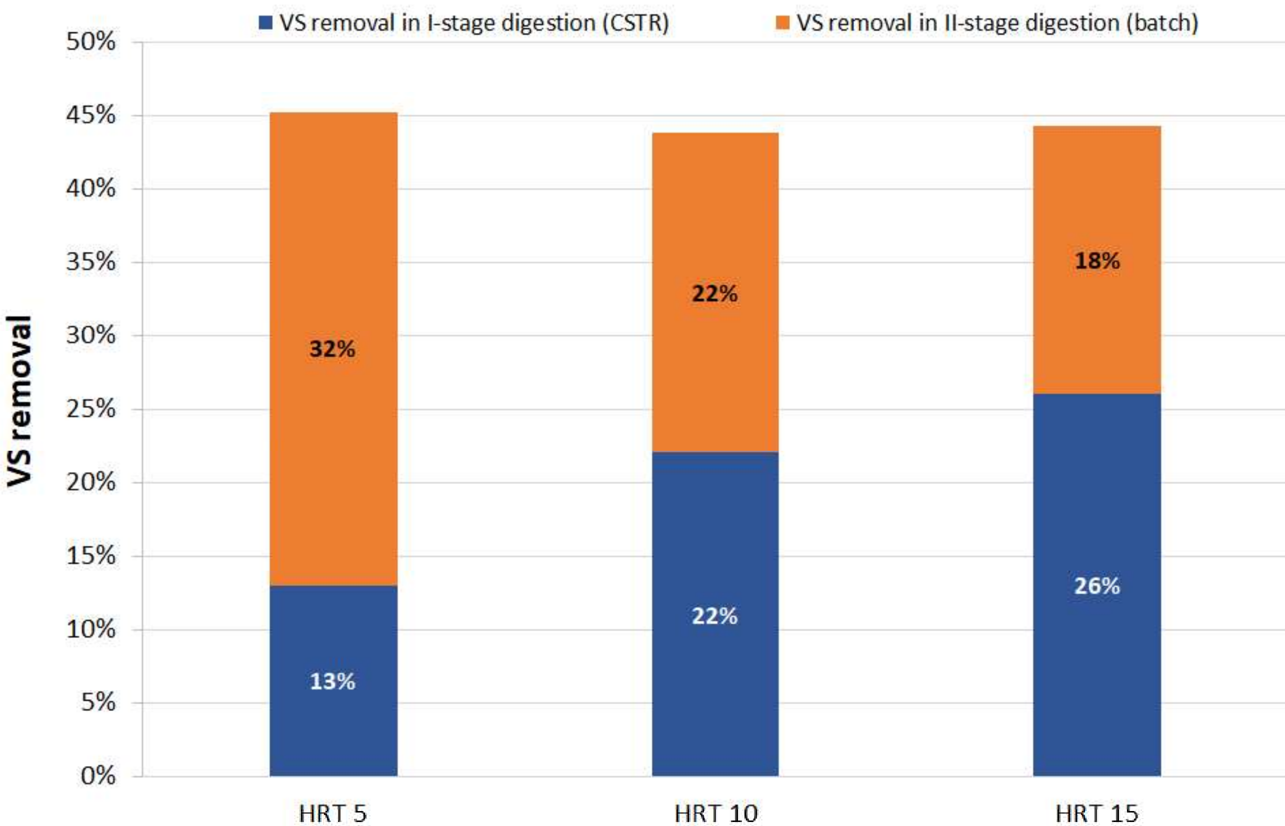

Figure 5. Overall VS removal obtained in the combination of the two digestion processes, with an in-between hybrid treatment, for the systems with a first-stage HRT equal to 5, 10 and 15 days

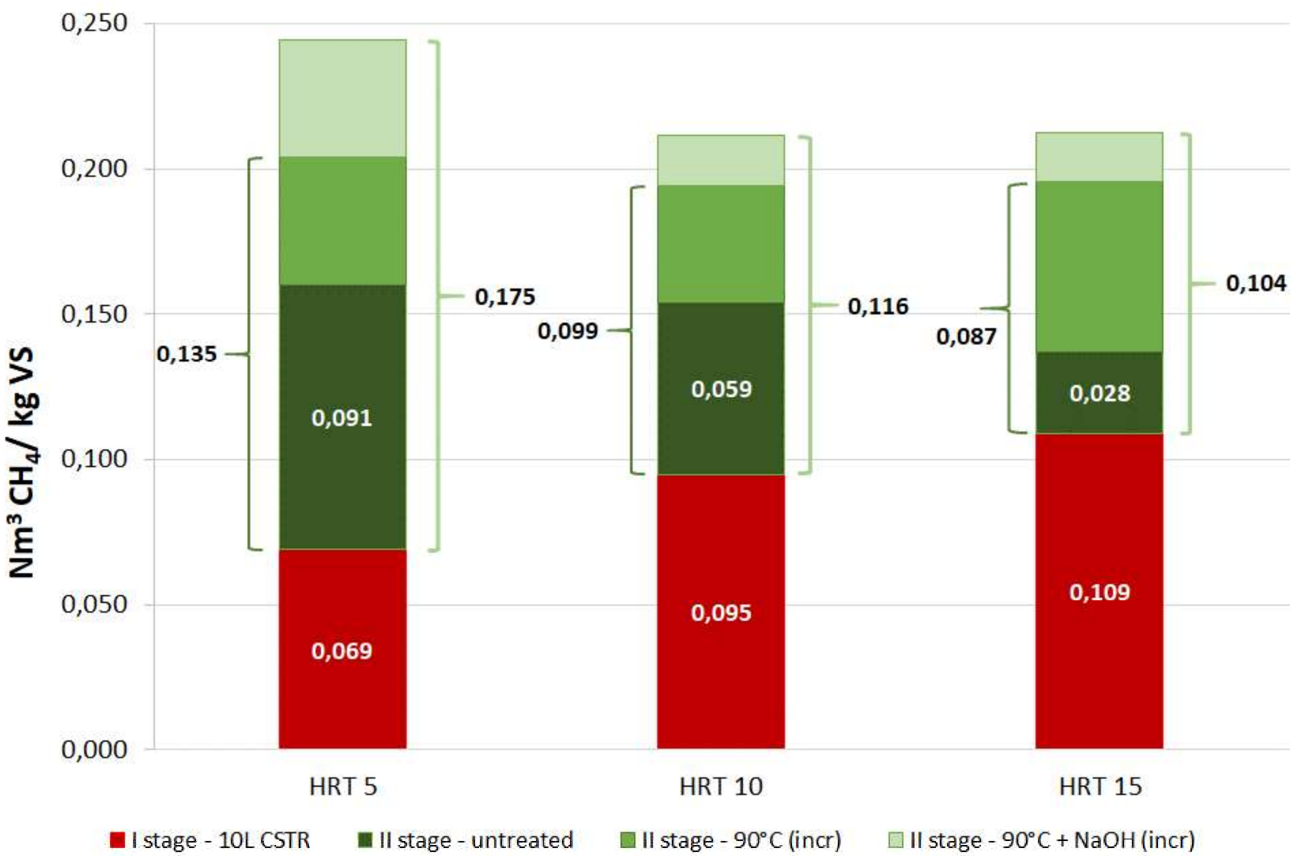

Figure 6. Overall SMP generated by the three systems (first stage HRT = 5, 10, 15 days) when the overall duration of the two-stage system was fixed to 20 days.

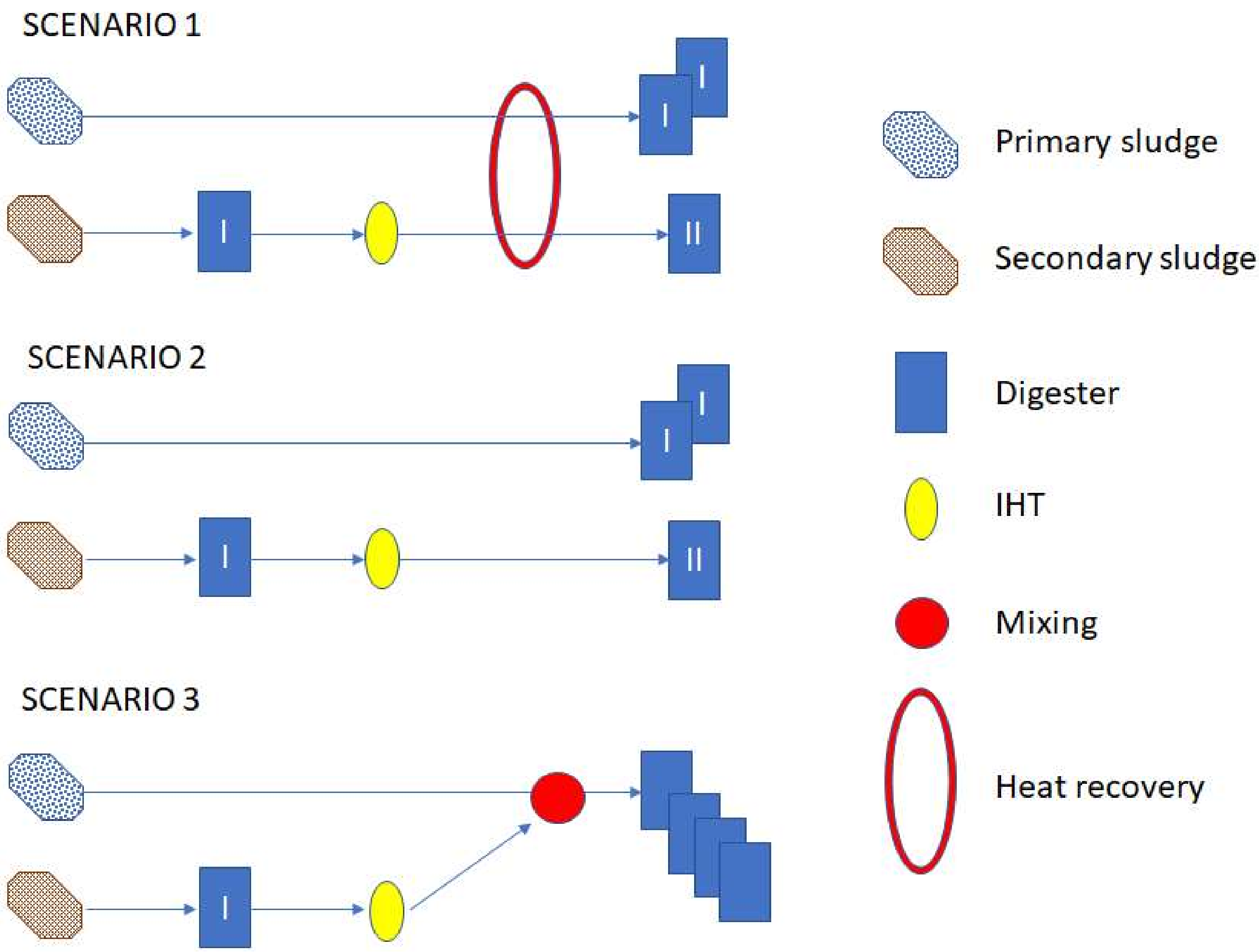

Figure 7. Scenarios considered for the technical and economic assessments at a full scale

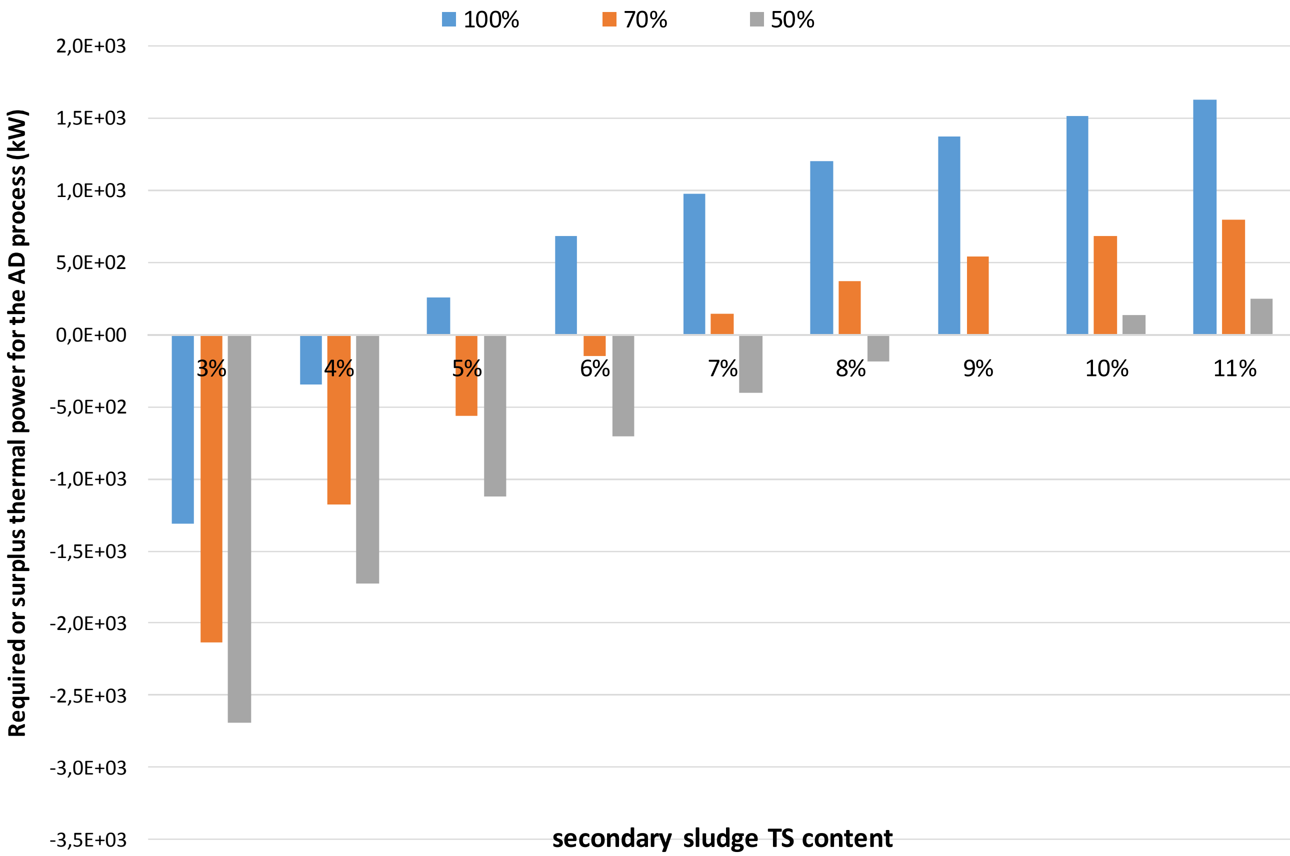

Figure 8. Required or surplus thermal power for the AD process for Scenario 0 that results from the technical assessment. Percentage values (100% - 70% - 50%) refer to the efficiency with which the heat produced from the CHP unit was transferred to the processes that need heating.

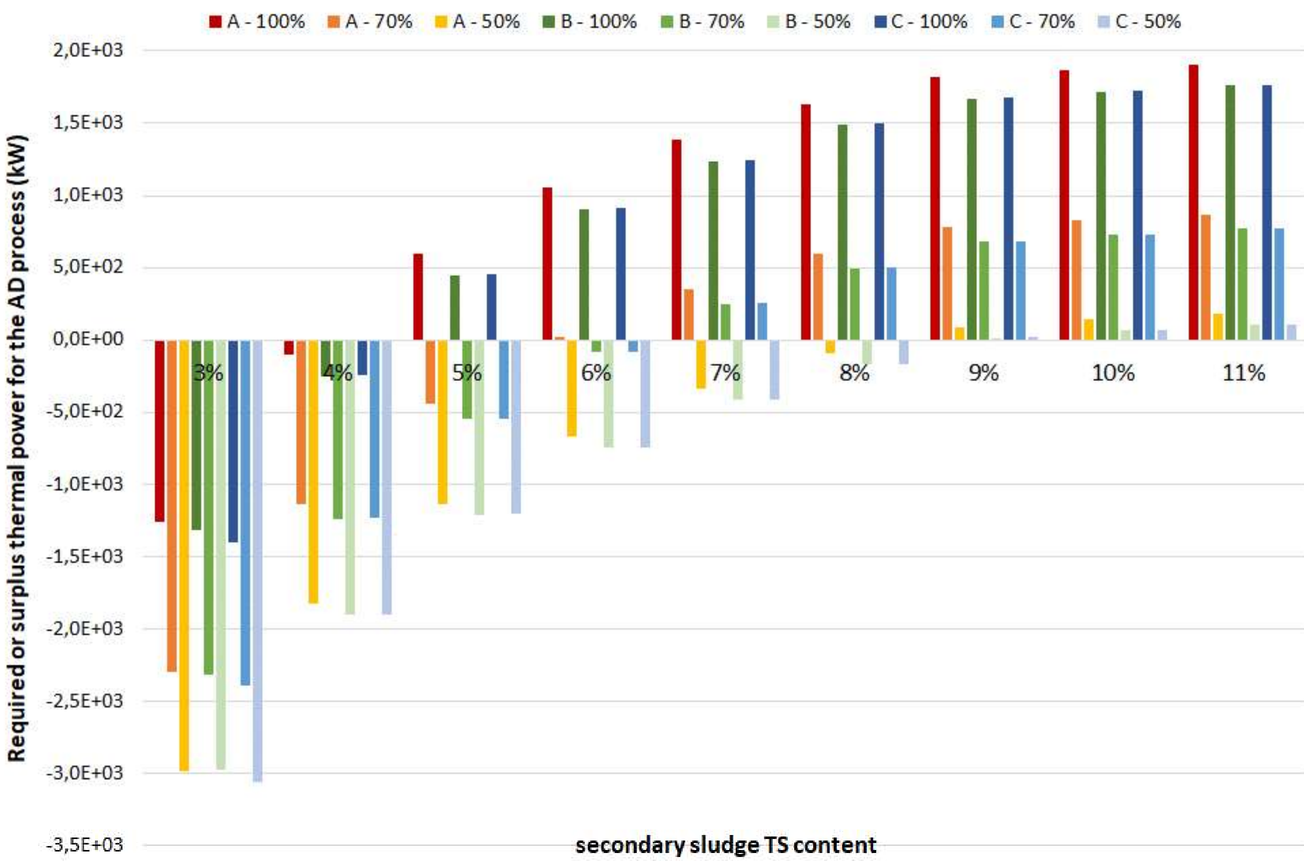

Figure 9. Required or surplus thermal power for the AD process for Scenario 1 that results from the technical assessment. Letters A, B and C refer to HRTs of the first-stage digestion of 5, 10 and 15 days, respectively. Percentage values (100% - 70% - 50%) refer to the efficiency with which the heat produced from the CHP unit was transferred to the processes that need heating (IHTs and digestion processes).

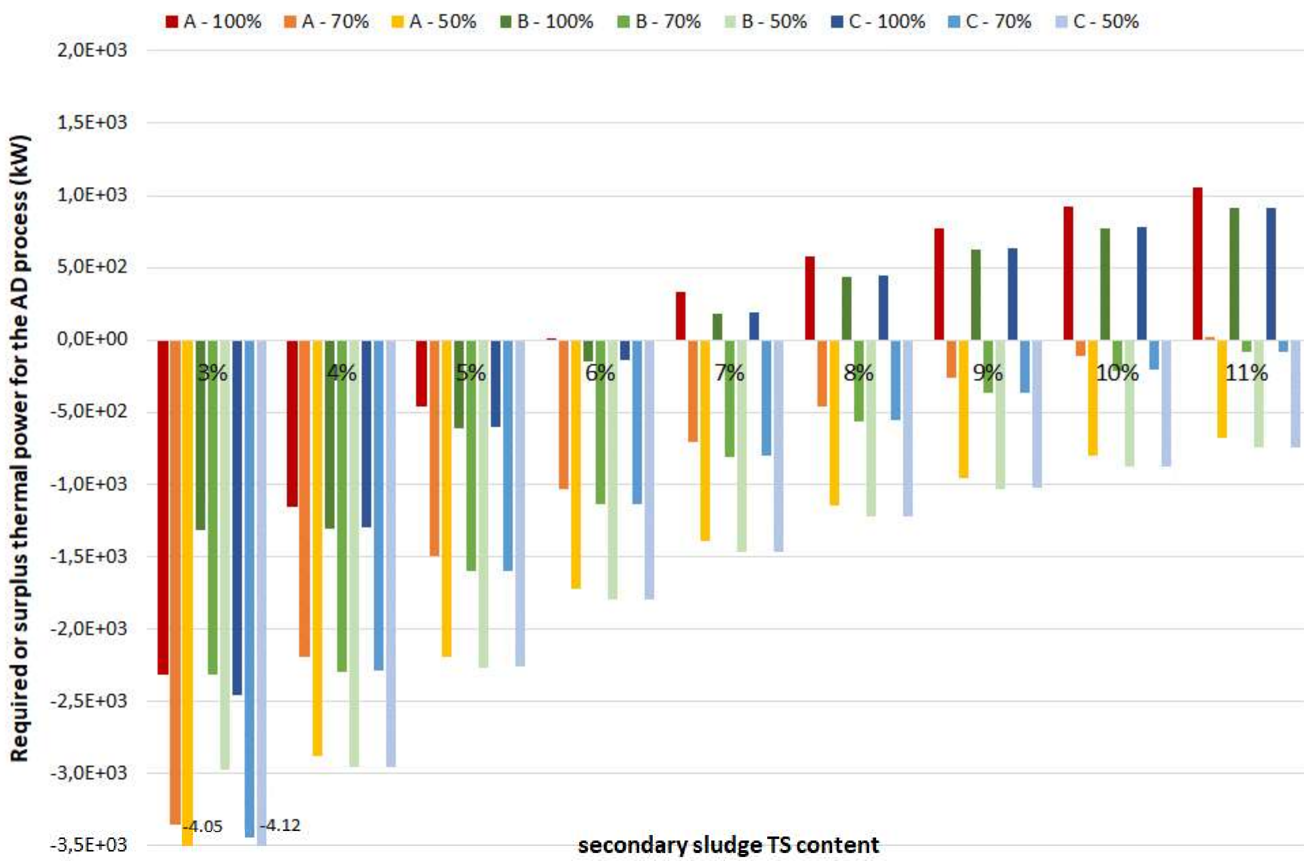

Figure 10. Required or surplus thermal power for the AD process for Scenario 2 that results from the technical assessment. Letters A, B and C refer to HRTs of the first-stage digestion of 5, 10 and 15 days, respectively. Percentage values (100% - 70% - 50%) refer to the efficiency with which the heat produced from the CHP unit was transferred to the processes that need heating (IHTs and digestion processes).

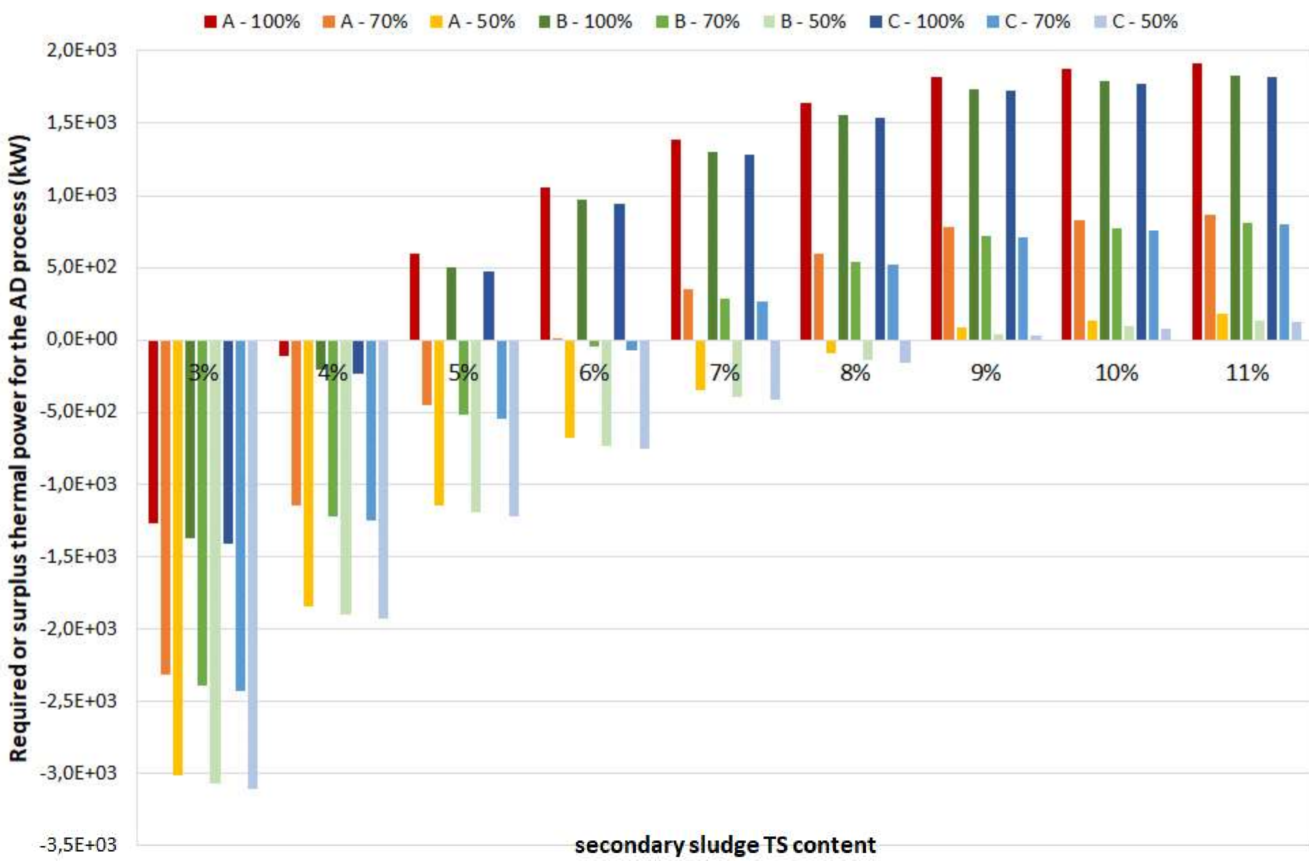

Figure 11. Required or surplus thermal power for the AD process for Scenario 3 that results from the technical assessment. Letters A, B and C refer to HRTs of the first-stage digestion of 5, 10 and 15 days, respectively. Percentage values (100% - 70% - 50%) refer to the efficiency with which the heat produced from the CHP unit was transferred to the processes that need heating (IHTs and digestion processes).

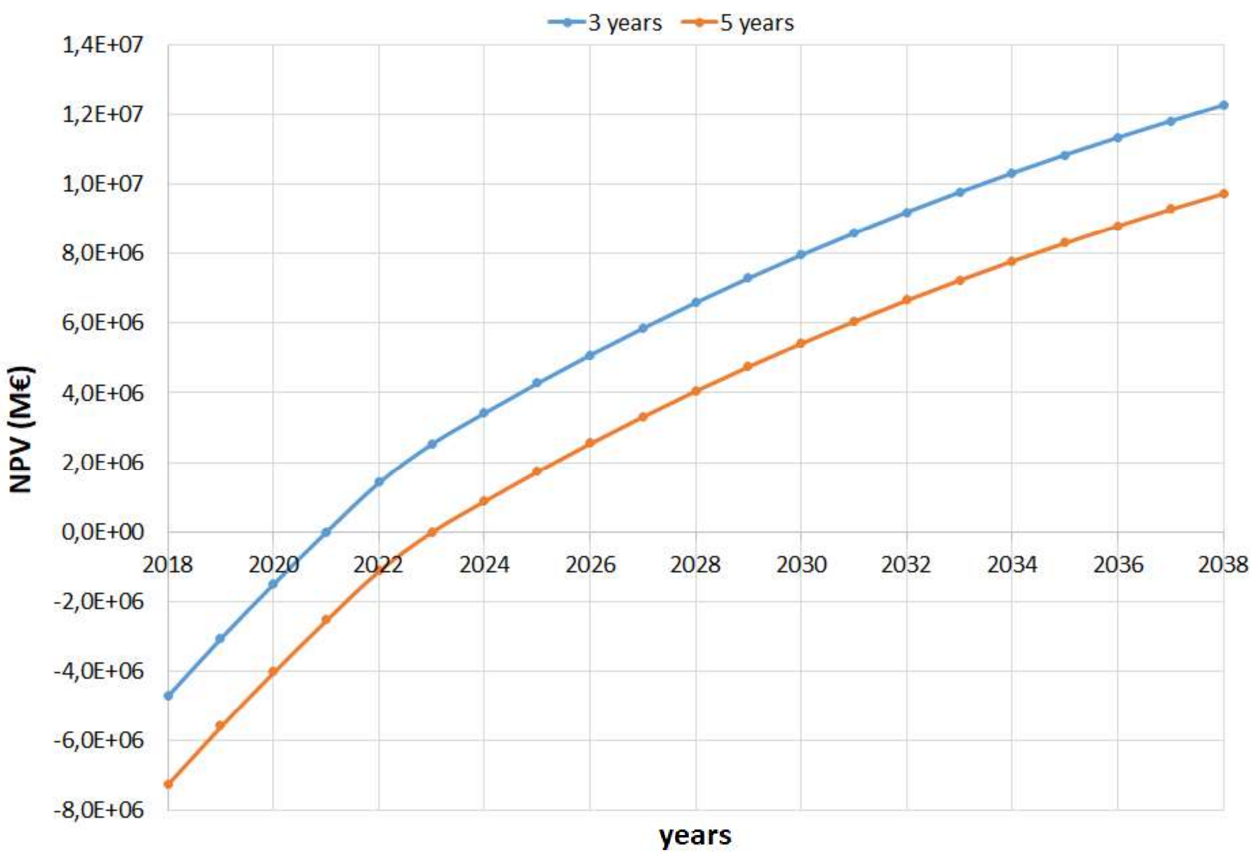

Figure 12. Net Present Value analysis for the assessment of the initial investment that the WWTP can bear, with pay-back times of three and five years, to renew the sludge line

Table

Table S1. Main results of the technical assessment carried out for Scenario 1 (Primary sludge TS content 7%)

First-stage digestion N. of digesters	Second-stage digestion N. of digesters	Primary sludge digestion N. of digesters	Total number of digesters	Heat exchange efficiency	%TS secondary sludge (*)	Duration of the first and second stage digestion (days)
1	2	2	5	100%	4.12	5+15
1	1	2	4		4.31	10+10
2	1	2	5		4.30	15+5
1	1	2	4	70%	5.95	5+15
1	1	2	4		6.23	10+10
1	1	2	4		6.21	15+5
1	1	2	4	50%	8.46	5+15
1	1	2	4		8.86	10+10
1	1	2	4		8.84	15+5

(*) that made the thermal balance neutral

Table S2. Main results of the technical assessment carried out for Scenario 2 (Primary sludge TS content 7%)

First-stage digestion N. of digesters	Second-stage digestion N. of digesters	Primary sludge digestion N. of digesters	Total number of digesters	Heat exchange efficiency	%TS secondary sludge (*)	Duration of the first and second stage digestion (days)
1	1	2	4	100%	6.00	5+15
1	1	2	4		6.41	10+10
1	1	2	4		6.38	15+5
1	1	2	4	70%	10.9	5+15
1	1	2	4		11.8	10+10
1	1	2	4		11.8	15+5
1	1	2	4	50%	23.7	5+15
1	1	2	4		27.1	10+10
1	1	2	4		26.9	15+5

(*) that made the thermal balance neutral

Table S3. Main results of the technical assessment carried out for Scenario 3 (Primary sludge TS content 7%)

First-stage digestion N. of digesters	Second-stage digestion (**) N. of digesters	Primary sludge digestion N. of digesters	Total number of digesters	Heat exchange efficiency	%TS secondary sludge (*)	Duration of the first and second stage digestion (days)
1	4	-	5	100%	4.13	5+20
1	4	-	5		4.24	10+20
2	4	-	6		4.28	15+20
1	4	-	5	70%	5.96	5+20
1	4	-	5		6.13	10+20
1	4	-	5		6.19	15+20
1	3	-	4	50%	8.47	5+20
1	3	-	4		8.72	10+20
1	3	-	4		8.80	15+20

(*) that made the thermal balance neutral
(**) digestion of the mixture made of primary sludge and treated digestate from secondary sludge