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Improvement of anaerobic digestion of sewage sludge in a wastewater treatment plant by means of mechanical and thermal pre-treatments: performance, energy and economical assessment

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Abstract

Performances of mechanical and low-temperature (<100°C) thermal pre-treatments were investigated to improve the present efficiency of anaerobic digestion (AD) carried out on waste activated sludge (WAS) in the largest Italian wastewater treatment plant (2,300,000 p.e.). Thermal pre-treatments returned disintegration rates of one order of magnitude higher than mechanical ones (about 25% vs. 1.5%). The methane specific production increased of 21% and 31%, with respect to untreated samples, for treatment conditions of respectively 70 and 90°C, 3h. Thermal pre-treatments also decreased WAS viscosity. Preliminary energy and economic assessments demonstrated that a WAS final total solid content of 5% was enough to avoid the employment of auxiliary methane for the pre-treatment at 90°C and the subsequent AD process, provided that all the heat generated was transferred to WAS through heat exchangers. Moreover, the total revenues from sale of the electricity produced from biogas increased of 10% with respect to the present scenario.

Keywords: waste activated sludge, pre-treatments, anaerobic digestion, rheology, energy balances

1. INTRODUCTION

Waste activated sludge (WAS) is an important source of biological matter that is commonly produced in secondary processes of wastewater treatment plants (WWTPs). According to Wilson and Novak [1] and Cho et al. [2], WAS handling is one of the most difficult and expensive items for a WWTP, since it accounts for 30-40% of capital costs and 50% of operating costs of the plant. Anaerobic digestion (AD), with its four steps of hydrolysis, acidogenesis, acetogenesis and methanogenesis, is one of the most common and widely adopted methods for WAS treatment and stabilization. In fact, in addition to reducing the overall amount of biosolids to be disposed of by about 40%, and stabilizing organic substance, AD produces biogas, the two main components of which are methane and carbon dioxide, that can be exploited for energy recovery. Other beneficial features of the AD process include inactivation and reduction of pathogens and improvement of sludge dewaterability [3].

Nevertheless, AD is not completely effective towards WAS from WWTPs because the complexity of the WAS structure limits the biological process. Low biodegradability of WAS is due to the retarded hydrolysis, because microbial cells, cell walls and membrane in the WAS are strong barriers that do not easily permit the penetration of hydrolytic enzymes [2, 4].

In order to accelerate the step of hydrolysis, thus improving degradability of sludge with subsequent lower digester retention time and higher methane production rates, several types of pre-treatments have been tested with the purpose of:

- reducing macromolecule dimensions and avoiding presence of macro-flocks inside the sludge;
- breaking bacteria cell walls with release of intracellular organic matter.

WAS pre-treatments can be classified on the basis of the lysis system employed. Pre-treatments can be grouped into biological, thermal, mechanical, chemical as well as different combination of these [5-12]. In the wide scenario of lysis technologies, mechanical and thermal pre-treatments are the most common systems available on the market, working in full scale plants with good results in terms of COD solubilization and AD performance improvement [13-22]. Soluble COD (SCOD), that is the amount of COD in the liquid phase (after 0.45 µm filtration), is a parameter of easy analytic determination strictly related to sludge degradability.

In the field of mechanical pre-treatments, several systems for mechanical disintegration are available, such as lysis-thickening centrifuge, stirred ball mills, high pressure homogenizers, high pressure jet and collision, rotor-stator disintegration system. The aim of the mechanic disintegration is to enhance sludge solubilization as a consequence of bacteria cell disintegration and disaggregation of biological flocs. In general, at low applied energy only floc disintegration is observed, while high energy is required to damage microbial cells [5, 8]. Although not many results are available for mechanical pre-treatments in respect to the other methods, it is demonstrated that the efficiency of most of the technologies in improving AD of sewage sludge is rather low, if not coupled with other methods [2, 19-21].

In thermal pre-treatments sludge is generally heated to temperature values in the range 150-200°C, although lower temperatures have also been tested [5, 15, 23]. The most significant drawback of high temperature pre-treatments is the high energy requirement. The surplus of energy that can be recovered because of the increase in biogas production is largely balanced by the high energy requirements for rising the temperature of sludge to the temperature of the thermal process. This largely reduces the overall profitability of the process. Application of low temperature thermal pre-treatments (<100°C) could be an alternative to overcome this drawback. However, at low temperature values, treatment time plays a more dominant role than treatment temperature [5]. Lysis pre-treatments, other than having effect on COD solubilization and subsequent methane production rate, are able to affect the structure of sludge thus lowering its viscosity with the consequence of improving sludge handling and pumpability in the WWTP [24-25].

In this study, in the view of improving the efficiency of the AD process performed on the WAS produced in the largest Italian WWTP (2,300,000 p.e., population equivalent), located near Turin, in the north of Italy, the experimental activities listed in the follow were carried out:

- comparison of the performances obtained from mechanical and low-temperature thermal pre-treatments on WAS samples thickened to final total solid (TS) contents of 2, 4 and 9% b.w. (by weight). The TS values employed in this study were in the range that may be obtained using dynamic thickeners;
- evaluation of the influence of temperature (70-80-90°C) and treatment time (from 1 to 15 hours) on the performances of low-temperature pre-treatments;
- evaluation of the increase in biogas and methane generation from AD of WAS, due to thermal pre-treatments, using batch tests at semi-pilot scale;
- evaluation of the effect of low-temperature thermal pre-treatments on the rheology of sludge samples at different TS values;
- preliminary assessment of the energy and economic feasibility of full scale low-temperature thermal pre-treatments.

2. MATERIALS AND METHODS

2.1 The WWTP and the present performance of sludge AD

The largest Italian WWTP treats municipal and industrial wastewater with a capacity of about 2,300,000 p.e. (about 1.5 million of civil inhabitants, over 1,000 industrial discharges and also tank truck wastewater). It consists of four parallel lines devoted to wastewater treatment and a modular sludge treatment. Each line for wastewater treatment, with an average flow rate of about 25,000 m^3/h, is made up of the following processes: grid screens, grit and grease removal, primary sedimentation, pre-denitrification, biological oxidation, secondary sedimentation, phosphorous removal and final filtration. The wastewater treatment process generates an average amount of primary and secondary sludge equal to about 300 – 350 m^3/h, with an average TS content of 1%, that is sent to the sludge treatment. The sludge treatment line is made up of the following unit operations: pre-thickening, mesophilic AD, post-thickening and final dewatering. The pre-thickening process reduces the amount of sludge to be treated by AD to about 110 m^3/h, with an average TS content of 2.75% for both primary and secondary sludge. The pre-thickening process is carried out by means of gravity devices with the addition of polyelectrolyte for thickening of the secondary sludge. The total sludge amount is treated in six anaerobic digesters, one of them periodically in maintenance. Each digester has a volume of 12,000 m^3 (for a total volume useful to the digestion process of 60,000 m^3), a D/H (diameter, height) ratio of 26/30, a filling coefficient of 0.8, an hydraulic retention time (HRT) of about 17 days, a fed sludge amount of 23.5 m^3/h with a TS content of 2.75%, for a mass flow rate of dry substance of 650 kg/h.

The digestion process is carried out in mesophilic conditions, at the temperature value of 38°C. In order to heat the sludge from the average temperature of 15°C (ambient temperature) to 38°C and keep the process temperature constant, each digester is coupled with a double-tube heat exchanger fed by the hot water (70°C) circuit. Four cogeneration engines (GE-Jenbacher JMS 420 GS-B.L.), that produce heat and electricity by burning the biogas generated in the AD process, supply heat to the hot water circuit. The cogeneration engines have thermal and electrical efficiency of 42%.

In the present working condition (no pre-treatments applied to WAS), each digester requires 2,425 MJ/h to heat the sludge to the process temperature (38°C) and offset the thermal losses with the outside. Each digester, fed with 50% primary sludge and 50% secondary sludge, produces about 1,900 MJ/h (yearly average), considering that the average methane production of the primary sludge is of 0.385 $Nm^3/kgVS$ and the average methane production of the secondary sludge is of 0.167 $Nm^3/kgVS$ (data supplied from the WWTP). In the present situation the AD of the mix made of primary and secondary sludge has an economic revenue of 530 €/h from sale of electricity. However, the thermal balance is negative because the AD process requires an average consumption of auxiliary methane of about 35 Sm^3/h (with peaks of 40 Sm^3/h in the winter season).

2.2 WAS collection and characterization

Samples of WAS were collected from the WWTP described as in Section 2.1. Raw WAS samples were firstly sieved at 0.295 mm (using a 48-mesh Tyler Standard sieve), in order to remove coarse particles and impurities, and then thickened from an initial TS content of about 0.8% to the final value of 2 or 4 or 9% b.w. established for the pre-treatment tests. Sieving operations resulted in a near 10% b.w. loss of coarse sludge solids.

Samples were stored at 4°C until utilization, in all cases no later than 48 hours from collection. The parameters (TS, VS, TCOD – that is the COD of the whole sludge sample, SCOD – that is the COD in the liquid phase after filtration on a 0.45 μm acetate-cellulose membrane; pH, EC – electric conductivity and soluble ammonia) were determined according to standard methods [26]. Sludge samples had an average TS content of 0.823%, a VS/TS ratio of 67.7%, a TCOD content of 7,200 mg/L, a SCOD of 31 mg/l, a pH value of 7.05, a EC value of 1,270 μS/cm and a soluble ammonia (NH_4^+) content of 38.4 mg/L.

2.3 Mechanical pre-treatments

Mechanical pre-treatments were carried out on samples of WAS using a high shear mixer (UltraTurrax T-25, IKA) that operated at 5,000 rpm for 10 minutes. Mixer operating values were chosen in order to simulate working conditions and residence time of a full-scale centrifuge as in [16]. WAS samples were preliminarily thickened to TS values of 2, 4 and 9% b.w. and then put inside a test-tube with diameter size of some millimeters larger than that of the mixer head, in order to optimize the contact between the sludge and the mixing device. The mechanical disintegration and grinding of WAS particles was developed by a rotor-stator system similar to that of full scale machines. Sludge was pressed through a cylindrical space by an agitator inducing shear stresses of sufficient magnitude to break the bacterial cell walls.

The sludge liquid phase was separated from the corpuscolate phase by means of centrifugation (4,000 rpm, 15 min) and subsequent filtration on 0.45 μm acetate-cellulose membranes. On all samples, TCOD and SCOD, before and after treatment, were determined according to standard methods [26].

2.4 Thermal pre-treatments

The effect of temperature and contact time on the low-temperature thermal pre-treatment performances was tested using WAS samples with TS content of about 4% b.w. treated at temperature values of 70, 80, 90°C for contact times of 1, 2, 3, 6, 9 and 15 hours. Moreover, a direct comparison between the performances of mechanical and thermal pre-treatments was carried out on WAS samples thickened to TS content of 2, 4 and 9% b.w. and treated at 90°C for 3 hours.

Thermal pre-treatments were carried out on WAS samples of 300-ml that were introduced into 500-ml glass laboratory bottles equipped with screw caps. The bottles were dipped in a thermostatic bath preheated at the temperature value established for the test. Contact times of more than 15 hours were not considered because of the significant increase in the energy requirements that those values would have implied. At the end of the thermal treatment, samples were allowed to cool and the liquid phase was separated and then characterized. The sludge liquid phase was separated from the corpuscolate phase by means of centrifugation (4,000 rpm, 15 min) and subsequent filtration on 0.45 µm acetate-cellulose membranes. On the liquid phases that came from the different treatment conditions, pH, EC, SCOD and soluble ammonia were determined according to [26].

2.5 Batch tests at semi-pilot scale for digestibility assessment

Because of the best performances of thermal pre-treatments over mechanical pre-treatments in the COD solubilization (see Section 3), digestibility batch tests were carried out only on thermal treated WAS samples.

Batch tests were performed on treated and untreated (control) WAS samples in order to assess the effect of different pre-treatment conditions (temperature, treatment time) on WAS digestibility and verify the correspondence between the increase in soluble COD and the increase in biogas and methane production. Due to the limited availability of pilot-scale digestors (5), the anaerobic digestibility tests on the WAS samples were conducted on two series. Following the outcomes found in the thermal pre-treatment tests (see Section 3), among the 18 tested systems, four (70°C, 3h; 70°C, 15h; 80°C, 3h; 90°C, 3h) were chosen to undergo digestibility tests in batch mode.

In the first series of test, biogas and methane production of the WAS samples treated at 80°C and 90°C for 3 hours were compared with the control. The second series of tests involved samples treated at 70°C for 3 and 15 hours and the relative untreated sample. Digestibility tests were performed in batch mode (2 replicates for treated samples, 1 replicate for untreated samples, UNT), under mesophilic conditions (35°C), using 6-L poly methyl methacrylate (PMMA) digesters placed in a thermostatic bath. The anaerobic environment inside the digesters was prepared by filling them with water, then replacing water with nitrogen. This procedure also ensured that the reactors were leak free. In order to simulate real digestion conditions, pH value was not adjusted and no nutrients were added.

For digestibility tests in batch mode, 2,400 ml of on-purpose treated WAS (or an equal volume of the control, see Table 1) were put in contact with an inoculums, according to a volumetric ratio of 1:1 and a digester volume filling level of 80%. The inoculums was prepared from 1,000 mL of sludge collected from the anaerobic digesters of the WWTP described as in Section 2.1. The inoculums was progressively fed with amounts of primary sludge, coming from the same WWTP, to reach a total volume of 2,400 ml. The inoculums was considered ready to start digestibility tests when its daily biogas production was of less than 0.5% of the total production registered throughout the whole period of preparation. The inoculums had a TS content of 1.58 ± 0.16%, a VS/TS ratio of 58.9 ± 0.5% and a pH value of 7.23 ± 0.01 (average value on 5 replicates ± standard deviation)

For each replicate, the produced biogas was collected in two 5-L Tedlar® bags connected in parallel. The characterization and measure of the volume of the produced biogas was carried out daily, throughout the whole duration of the test. The characterization, that is the volumetric composition of the biogas in terms of CH_4, CO_2, O_2 and "balance" (the fraction made up of gases that are different from the first three) 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 replacing volumes of water with the residual gas and referring the obtained value to normal conditions.

All the samples involved in digestibility tests were characterized, before and after the test, with reference to TS, VS/TS ratio, SCOD and soluble ammonia according to standard methods [26].

2.6 Rheological characterization

A rheological characterization of WAS samples at 2, 4 and 9% TS content before and after thermal treatments (90°C, 3 h) was performed using an ANTON PAAR PHYSICA MCR 301 rheometer with the purpose of investigating the effect of pre-treatments toward viscosity values in shear conditions. WAS samples were tested using parallel plates device (PP50) in the range 10^{-3} – 10^{2} s^{-1} of shear rate.

2.7 Energy assessment and preliminary cost-benefit analysis

The energy assessment was carried out for the present situation (as described in Section 2.1) and for the possible future scenario, that planned the introduction of low-temperature thermal pre-treatments of WAS. In the energy assessment the comparison between the amount of heat produced by the AD process and the amount of heat required for heating and maintaining the sludge at the process temperature (70, 80 or 90°C), and offsetting thermal losses during the AD process, was made.

The heat losses with the outside were evaluated by taking into account the geometry of the digester, the materials employed for its construction and the temperature of pre-thickened sludge (15°C), soil (15°C) and outside (exterior environment, as monthly averages as reported in the UNI 10349 rule).

The future scenario that planned the introduction of low-temperature thermal pre-treatments included the following operations:

- thickening of primary sludge to a TS content of 3.50% b.w. using all the gravity devices available in the WWTP;
- thickening of secondary sludge to a range of TS from 3.5% b.w. to 8% b.w. using dynamic thickeners that will be on-purpose acquired;

- low-temperature thermal pre-treatment of thickened secondary sludge at temperature values of 70, 80 or 90°C. For the development of the energy balance, the average methane production of the primary sludge was fixed to 0.385 Nm^3/kgVS (see Section 2.1) and the average methane production of the WAS respectively to 0.202, 0.216 and 0.219 Nm^3/kgVS, depending on the temperature of the pre-treatment process, 70, 80 or 90°C (see Section 3).
- final mixing of the thermal pre-treated WAS with thickened primary sludge, with the aim of homogenizing the sludge sent to digesters and using the heat of the treated secondary sludge to increase the temperature of the mix.

The thermal pre-treatment included two phases: a first phase in which the sludge was heated from the ambient temperature (15°C) to the temperature value at which the pre-treatment was carried out (70, 80 or 90°C) and a second phase in which the process temperature value was maintained for the whole duration of the thermal treatment (3 hours, see Section 3.2).

The energy balance could be described using the following three equations (1, 2 and 3):

$$T_r + \Delta T = \frac{F_s T_s + F_p T_p}{F_s + F_p} \qquad (1)$$

$$\Delta T = \frac{nQ_a}{c_p(F_s + F_p)} \qquad (2)$$

$$F_s = \frac{nQ_a}{c_p(T_s - T_r)} + F_p \frac{(T_r - T_p)}{(T_s - T_r)} \qquad (3)$$

Where the equation (3) was obtained from (1) and (2) and the parameters of equation (3) are listed in the follow:

Tr = temperature of the mesophilic digestion process (38°C);

Tp = temperature of primary sludge after thickening (15°C, as yearly average);

Ts = temperature of secondary sludge after pre-treatment (70, 80 or 90°C);

ΔT, as in the Equation (2), is the difference between the temperature of the mix made of primary and secondary sludge thermally treated and the temperature inside the digester (38°C). This temperature difference was necessary to offset the thermal losses with the outside (Qa).

Cp, specific heat capacity of sludge. The specific heat capacity of sludge was based on proportion of water and solids in the sludge. The values of heat capacity used for calculations amounted to 4.18 and 1.95 kJ/kg °C for water and solids, respectively. The sludge density was fixed to 1,010 kg/m^3 for converting the unit volume of sludge into unit mass of volatile solids.

n = number of digesters employed for the AD of the mix.

Fp = flow rate of primary sludge (3.5% TS)

Fs, unknown, minimum flow rate of secondary sludge to be mixed with primary sludge because Tr was at least of 38°C.

The final outcome of the energy balance carried out for the future scenario was the flow rate (and, consequently, the TS amount) of thermal pre-treated WAS that, after mixing with primary sludge (TS 3.5%, at ambient temperature), was able to maintain the resulting mix at the process temperature value (38°C) by offsetting the thermal losses with the outside.

From the outcomes of the energy balance, a preliminary economical assessment of the whole AD process was performed. That assessment took into account the price of the methane employed as an auxiliary fuel, equal to 0.40 €/Sm^3 and the revenue from the electricity sale, equal to the sum of the electricity price, 0.14 €/kWh and green certificates, 0.077 €/kWh (data from Electrical Service Company), for a total of 0.217 €/kWh. The economical assessment here performed did not consider the costs (purchase cost and energy cost) for the new dynamic thickeners and the avoided costs for the polyelectrolyte purchase.

3. RESULTS AND DISCUSSION

3.1 Effect of mechanical and thermal pre-treatments on COD solubilization

The lysis effect of both mechanical and thermal pre-treatments was preliminarily assessed in terms of the disintegration rate (DR) parameter. DR is one of the most frequently employed indicators to compare the effectiveness of different WAS pre-treatments [16]. The employed formula was the following (4):

$$DR = \frac{SCOD_l - SCOD_0}{TCOD \; - SCOD_0} \cdot 100\% \qquad (4)$$

where the terms TCOD, $SCOD_0$ and $SCOD_l$ referred respectively to the total COD of sludge, the soluble COD before lysis and after lysis.

The results obtained from mechanical and thermal pre-treatments carried out at 90°C for 3 hours on WAS samples thickened to 2, 4 and 9% b.w. TS content are compared in Table 2. From the results of Table 2 it can be seen that thermal pre-treatments, if compared to mechanical ones, gave higher values of DR, which were quantifiable in more than one order of magnitude (about 25% vs. 1.5%). In the case of mechanical pre-treatments, the DR value increased of about 16% when the TS content increased from 2 to 4% b.w. and of about 35% when the TS content increased from 4 to 9% b.w. On the other hand, in the case of thermal pre-treatments, the DR value increased of about 17% when the TS content increased from 2 to 4% b.w. but of only about 15% when the TS content increased from 4 to 9% b.w.

The effect of temperature and contact time on the performance of thermal pre-treatments on WAS samples with a TS content of about 4% b.w. was evaluated in terms of DR, the values of which are shown in Figure 1. The results presented in Figure 1 show that not only the pre-treatment temperature but also the duration of the exposure determined the level of COD solubilization. For the considered treatment times (1-15 hours), an increase in the DR parameter from 15 to 25% for the temperature value of 70°C, from 15 to 28% for the temperature of 80°C and from 19 to 30% for the temperature of 90°C, was observed. Figure 1 highlights only a slight difference in terms of DR among the results obtained in the treatments performed at 70 and 80°C for durations of less than 3 hours. For durations of 6, 9 and 15 hours, the efficiency of the treatment carried out at 80°C was nearly at the midpoint between the results obtained at 70 and 90°C. Moreover, the trend of the results in terms of DR seemed to suggest a change in the curve slope for treatments that lasted respectively more and less than 3 hours. Although the efficiency of the thermal treatment, in terms of SCOD release, decreased for durations of more than 3 hours, for the tested treatment durations the DR trend seemed not to reach an asymptotic value.

The DR values obtained for treatments with a duration of 1 hour were compared with the results presented by Appels et al. [3]. In their work the authors tested the efficiency of low temperature thermal pre-treatments with a maximum duration of 1 hour (15, 30, 60 minutes) on sludge (probably a mixture of primary and secondary sludge) with TS equal to about 6.5% b.w. and VS/TS ratio of 70%. They obtained a SCOD release, assessed by means of the parameter "degree of solubilization" (DS, that is the ratio between the increase of SCOD due to treatment and the total COD of the sludge sample), respectively equal to 1.36%, 14.1% and 17.8% for the temperature of 70, 80 and 90°C. The DS parameter can correspond, with good accuracy, to the DR parameter because, especially for high TS values, the SCOD of the untreated sludge is negligible if compared to the total COD content (in this work 173 mg/l vs. 46,000 mg/l). There was good correspondence between the results obtained by Appels et al. [3] and those of this work for the temperature values of 80 and 90°C. On the contrary, with reference to the result obtained for 70°C, the efficiency observed by Appels et al. [3] seemed to be of one order of magnitude lower than that found in this work.

With reference to the work of Dhar et al. [23], where thermal pre-treatments performed at 50, 70 and 90°C with a duration of 30 minutes were applied to activated sludge characterized by TS equal to about 2% b.w. and VS/TS ratio of 75%, the authors observed an increase in the SCOD/TCOD ratio between the untreated sample and the treated one of 18-19% for the temperature of 70°C and equal to 35-37% for the temperature of 90°C. Notwithstanding the difference between the DR parameter and the SCOD/TCOD ratio and keeping in mind the shorter duration of the thermal treatment (30 minutes), the efficiency observed by Dhar et al. [23] had to be considered at least double of that found in this work. The differences between the results found in this work and those obtained in the afore mentioned experiences [3, 23] could be ascribed to the different characteristics (composition, TS and VS/TS content) of sludge subjected to thermal pre-treatments.

The trend of pH value, EC and soluble ammonia (NH_4^+) for treated and untreated systems is shown in Table 3. The decrease in the pH value noticed as the duration of the treatment lengthened had to be ascribed to the progressive release of organic acids in the liquid phase. The highest EC values observed for the systems treated at 70°C for 6, 9 and 15 hours were related to the soluble ammonia content in the liquid phase. With reference to soluble ammonia, all pretreated samples contained amounts of soluble ammonia higher than control. However, at elevated pre-treatment temperatures (>75°C), a significant decrease in soluble ammonia was observed. The reason for the reduction in the soluble ammonia concentrations of samples near boiling point is currently unknown but has been observed in other studies [27-29]. Some authors [27-29] tried to explain the decrease in the ammonia concentration as the result of caramelization or Maillard reactions occurring at temperature above 80°C. Because of the Maillard reactions, hydrolyzed ammonia is involved in polymerization with reducing sugar and thus transformed back to the particulate phase, resulting in a decrease in soluble ammonia in the pretreated samples.

3.2 Digestibility batch tests and rheological measurements

Following the outcomes found in the thermal pre-treatment tests, among the 18 tested systems, four (70°C, 3h; 70°C, 15h; 80°C, 3h; 90°C, 3h) were chosen to undergo digestibility tests in batch mode. The three systems treated for 3 hours (70, 80 and 90°C) were chosen for digestibility tests because of the slope change in the DR curve observed at that specific time. The system treated at 70°C for 15 hours was chosen because of the small difference between its DR value and the DR value observed for the system (90°C, 3 h).

With reference to the results of digestibility tests, Figure 2 and 3 show the daily trend of the biogas production (NmL) and the trend of the cumulative production (NmL) over the whole test period for treated and control systems. Both series of tests had a duration of 20 days, after such time the daily biogas production dropped below values of less than 1% of the cumulative production (1% criterion of the German Guideline VDI 4630, [30]).

With reference to the results of the first series of tests (80°C, 3h; 90°C, 3h; UNT1) shown in Figure 2, the untreated sample (UNT1) reached a daily biogas production of 3,500 NmL one day after the test start. Later, the biogas production value experienced a gradual reduction until it reached a value of less than 1% of the cumulative production 20 days after the test start. On the other hand, the treated samples (two replicates for each operating condition) showed a near constant daily biogas production, ranging from 2,500 to 3,500 NmL, during the first week of test. From day 7 a fast decrease was observed that led, at day 10, to a daily production of less than 500 NmL.

In the second series of tests (70°C, 3h; 70°C, 15h; UNT2, see Figure 3), the general trend of the daily biogas production for treated and untreated systems was approximately equal to that of the first series. Nevertheless some differences could be observed in the maximum value of the daily biogas production, in the duration of the phases characterized by constant production and in the biogas cumulative production reached at the end of the test. These differences could be due both to different characteristics of sludge employed in the first and in the second series of tests (mainly their TS content), and to the effect of operating conditions of the thermal pre-treatments. With reference to the second aspect, we may hypothesize that lower temperature treatments (70°C) promoted the starting phase of the digestibility process, thus causing a greater daily biogas production in the first days of the test. On the other hand, in the presence of higher temperature treatments (80 and 90°C), initially the digestibility process evolved more slowly, thus causing the highest values in the daily biogas production to appear only a week after the test start. However, the biogas and methane amounts produced by the systems treated at 80 and 90°C were higher than those from systems treated at 70°C. Biogas specific production (per unit mass of volatile solids), methane specific production, average methane content in the biogas and the increase of the aforementioned parameters compared to controls are shown in Table 4.

In order to make the biogas and methane specific production independent on the TS content of sludge employed in the test, only the percentage increase with respect to control was taken into account. Comparing the results coming from the first series of digestibility tests, samples treated at 80°C and 90°C for 3 hours showed, respectively, an increase of 29.2% and 31.2% in the methane production with respect to the untreated sample. With reference to the second series of tests, the sample treated at 70°C for 3 and 15 hours showed, respectively, an increase of 21.0% and 18.9% in the methane production compared to the untreated sample. From these outcomes and by imposing the UNT1 specific methane production equal to 0.167 Nm^3/kgVS (equal to UNT2), the specific methane production for systems treated at 80 and 90°C (3 hours) were respectively equal to 0.216 and 0.219 Nm^3/kgVS. These figures were employed for the energy assessment, the results of which were reported in Section 3.3.

In order to compare the obtained biogas and methane specific productions with the maximum production predicted by theoretical calculations according to the Buswell and Neave model [31], it was necessary to refer to the theoretical formula of activated sludge, that is $C_5H_7O_2N$. According to the amount of carbon provided by that formula, activated sludge subjected to anaerobic digestion would give rise to a biogas specific production of 0.99 Nm^3/kgVS and a methane specific production of 0.50 Nm^3/kgVS. With reference to biogas, the specific production observed on the whole, for treated and untreated samples, ranged from about 24% to 32% of the maximum theoretical production. With reference to methane, the specific production observed on the whole, for treated and untreated samples, ranged from about 28% to 43% of the maximum theoretical production.

Figure 4 provides a comparison between the amounts of TS and VS removed during AD with the increase in biogas and methane production with respect to the untreated samples. The amounts of TS and VS removed were calculated as in the equation (5):

$$\% \, TS \, (or \, VS) \, removed = \left(1 - \frac{M_d - M_i}{M_f}\right) \cdot 100\% \qquad (5)$$

where M_d was the mass of TS (or VS) in the digestate, M_i was the mass of TS (or VS) in the inoculums (under the hypothesis that the mass of inoculums did not change from start to end of the test) and M_f was the mass of TS (or VS) in the sludge fed to the digester.

The diagrams highlight that not all the cases examined showed an increase in the biogas production that corresponded to a proportional increase in the VS consumption.

With reference to soluble COD and ammonia concentrations before and after digestibility tests, the AD process reduced the SCOD values, for the thermal treated samples, by about one order of magnitude. On the other hand, at the end of digestibility tests the concentration of soluble ammonia, for all systems, reached values of about 1,000 mg/L. Final soluble ammonia concentrations were higher for the treated systems that had a higher biogas and methane specific production (data not shown).

The rheological tests carried out on thermally treated and untreated samples (TS content of 2, 4 and 9% b.w.) demonstrated the importance of using pre-treatments in order to decrease sludge viscosity, before AD processes, especially when they were carried out at high TS concentration. In fact, sludge viscosity is directly related with its pumpability and flowing ability through the WWTP. Sludge rheological behavior (see Figure 5) was influenced by pre-treatments especially at low shear rates (i.e. low applied stresses) where the structure and chemical forces inside sludge macromolecules are predominant (one order of magnitude decrease). That meant that thermal pre-treatments had the effect of deeply modify the structure of sludge. The effect of thermal pre-treatments on the rheological behavior of sludge could be simply summarized as it follows: the viscosity of a thermally treated WAS was equal to that of the sludge with half TS content. For instance, the viscosity of a 9% b.w. TS thermally treated sludge was equal to that of the untreated sludge at 4% TS b.w., with evident advantages in terms of flowing ability and management.

3.3 Energy assessment and preliminary cost-benefit analysis

In order to evaluate the energy and economic sustainability of thermal pre-treatments, some assessments were carried out. As said in Section 2, in the present working condition the AD of both primary and secondary sludge produced in the WWTP had an economic revenue of 530 €/h from sale of electricity. However, the thermal balance of the AD process was negative, in fact the process required an average consumption of auxiliary methane of about 35 Sm^3/h, with peaks of 40 Sm^3/h in the winter season.

The main outcomes of the energy balance performed as described in Section 2.3 and applied to the new scenario, that planned low temperature thermal pre-treatments of WAS, are detailed in Table 5. Under the conditions of flow rate and TS content detailed in Table 5, thermal pre-treated secondary sludge, after mixing with primary sludge, were able to maintain the resulting mix at the process temperature value, 38°C, by offsetting the thermal losses with the outside. The outcomes given back from the energy assessment of the new scenario showed that the balance between the heat necessary to pre-treat sludge, and maintain the system at the process temperature, and the heat generated from the produced biogas was positive, that is an extra thermal energy amount was available for plant requirements. That extra thermal energy was equal to about 950 MJ/h for the sludge treated at 70°C, to about 2,500 MJ/h for the sludge treated at 80°C and to 3,000 MJ/h for the sludge treated at 90°C. The performed calculations also demonstrated that the minimum flow rate of secondary sludge able to return positive values of the thermal balance decreased from 39.7 m^3/h (with a maximum TS content of 4.07%) to 23.4 m^3/h (with a maximum TS content of 6.90%), when the pretreatment temperature increased from 70 to 90°C. That meant that in the case of pretreatment at 90°C only 3 digesters could be employed.

From this starting condition, other calculations were done in order to search for the condition in which the balance between the heat required and the heat generated from biogas was equal to zero, that is all the generated heat was employed for the support of the AD process. For each pre-treatment temperature value, the main features of the researched condition are detailed in Table 6.

The results of Table 6 demonstrated that if all the heat generated by the burned biogas was transferred to the secondary sludge through heat exchangers, a TS content of 3.70% was enough to make the thermal pre-treatment at 70°C independent of the employment of an auxiliary fuel. The required TS content for secondary sludge rose to 4.25% and 4.90% for pre-treatment temperatures respectively of 80 and 90°C.

The implementation of this last scenario may guarantee, other than the complete energetic sustainability of the process, total revenues from sale of electricity that increased of amounts ranging from 35 €/h (that is + 6.7% with respect to the present scenario) to 53 €/h (that is + 10% with respect to the present scenario), depending on the pre-treatment temperature.

4. CONCLUSIONS

Low-temperature thermal pre-treatments on WAS may actually improve the performances of the AD process carried out in the largest Italian WWTP. Methane specific production increased of 21% and 31% for WAS samples treated for 3h at 70 and 90°C, respectively. Thermal pre-treatments also decreased WAS viscosity. Preliminary energy and economic assessments demonstrated that a WAS final TS content of 5% b.w. was enough because the pre-treatment at 90°C and the subsequent AD process were fully self-sustainable. Moreover, the total revenues from sale of the electricity produced from biogas increased of 10% with respect to the present situation.

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Table legends

Table 1. Characteristics of treated/untreated sludge subjected to digestibility tests

Table 2. Results of mechanical and thermal pretreatment on WAS at 2, 4 and 9% TS

Table 3. Trend of pH value, EC and soluble ammonia for thermally treated and untreated samples

Table 4. Results of the digestibility tests

Table 5. Main outcomes of the energy balance carried out on the mix made of primary sludge (TS 3.50%) and thermal pre-treated secondary sludge

Table 6. Main outcomes of the energy balance carried out on the mix made of primary sludge (TS 3.50%) and thermal pre-treated secondary sludge – balance between the produced heat and the required heat equal to zero

Figure legends

Figure 1. DR obtained in the low-temperature thermal pre-treatments

Figure 2. Daily trend of biogas production (NmL, **a**) and trend of the biogas cumulative production (NmL, **b**) for treated (80°C, 3h and 90°C, 3 h) and control systems

Figure 3. Daily trend of biogas production (NmL, **a**) and trend of the biogas cumulative production (NmL, **b**) for treated (70°C, 3h and 70°C, 15 h) and control systems

Figure 4. Comparison between the amounts of TS and VS removed during AD tests with the increase in biogas and methane production

Figure 5. Effect of thermal lysis (90°C, 3 hours) towards the rheological behavior of WAS at different TS content (yellow 2%, red 4%, green 9%). The upper curves refer to the untreated samples, while the lower ones refer to the thermal treated samples.

Table 1.

	TS (%)	VS/TS (%)	TCOD (mg/L)	SCOD (mg/L)	DR (%)	pH	EC (μS/cm)	NH_4^+ (mg/L)
UNT 1	4.74	72.8	48500	146	-	7.03	1300	37.7
80°C, 3h	4.74	72.8	48500	10550	21.5	6.37	2060	193
90°C, 3h	4.74	72.8	48500	11590	23.7	6.41	2400	197
UNT 2	3.82	70.1	40000	354	-	6.94	1590	53.3
70°C, 3h	3.82	70.1	40000	8370	20.2	6.49	2260	178
70°C, 15h	3.82	70.1	40000	11340	27.7	6.68	4860	712

Table 2.

TS (%)	Treatment modality	TCOD (mg/L)	$SCOD_0$ (mg/L)	$SCOD_1$ (mg/L)	DR (%)
1.96	Mechanical	17100	26	160	0.936
4.23	Mechanical	37600	32	420	1.12
9.31	Mechanical	84200	45	1450	1.72
2.04	Thermal (90°C)	18300	28	3700	20.2
4.43	Thermal (90°C)	39300	32	9600	24.4
8.59	Thermal (90°C)	78100	39	22500	28.8

Table 3.

Treatment time (h)	Treatment temperature (°C)	pH	EC (μS/cm)	NH_4^+ (mg/L)
0	-	7.05	1270	8.4
1	70	6.95	1790	155
2	70	6.90	1875	151
3	70	6.84	1945	149
6	70	6.63	3730	460
9	70	6.62	4710	614
15	70	6.97	5370	821
1	80	6.83	1555	79.6
2	80	6.79	1625	73.1
3	80	6.70	1682	93.8
6	80	6.67	1826	81.6
9	80	6.62	1864	88.7
15	80	6.74	1897	84.9
1	90	7.11	1503	78.5
2	90	7.06	1591	75.5
3	90	7.01	1669	84.0
6	90	6.84	1800	104
9	90	6.98	1783	88.3
15	90	6.97	1902	112

Table 4.

	Biogas production (Nm^3/kgVS)	Biogas production increment (%)	CH_4 production (Nm^3/kgVS)	CH_4 production increment (%)	CH_4 average content (%, v/v)	CH_4 average content increment (%)
UNT 1	0.234	-	0.138	-	59.2	-
80°C, 3h	0.287±0.0075	22.7	0.179±0.0062	29.2	62.3	5.24
90°C, 3h	0.295±0.0034	26.1	0.182±0.0025	31.4	61.6	4.05
UNT 2	0.262	-	0.167	-	63.8	-
70°C, 3h	0.312±0.0078	18.9	0.202±0.0113	21.0	64.8	1.57
70°C, 15h	0.302±0.0092	15.1	0.199±0.0063	18.9	65.7	2.98

Table 5.

Pre-treatment temperature (°C)	70	80	90
Minimum flow rate (m^3/h, secondary sludge)	39.7	30.3	23.4
Heat from the digester to the outside, maximum value on yearly basis (MJ/h)	220.5	220.5	220.5
Number of employed digesters	4	4	3
HRT (days)	18.6	21.0	17.3
Maximum TS content (%)	4.07	5.34	6.90
Required heat (MJ/h)	9,146	7,894	7,357
Produced heat (MJ/h)	10,105	10,352	10,412
Balance (MJ/h)*	959	2,458	3,055
Increased revenues from electricity (€/h)°	35.3	49.9	53.5

*positive values indicate that extra thermal energy is available
°with respect to the present scenario

Table 6.

Pre-treatment temperature (°C)	70	80	90
Minimum flow rate (m^3/h, secondary sludge)	43.9	38.1	33.2
Heat from the digester to the outside, maximum value on yearly basis (MJ/h)	220.5	220.5	220.5
Number of employed digesters	4	4	4
HRT (days)	17.8	19.0	20.2
Maximum TS content (%)	3.69	4.25	4.88
Required heat (MJ/h)	10,105	10,352	10,412
Increased revenues from electricity (€/h)°	35.3	49.9	53.5

°with respect to the present scenario

Figure 1.

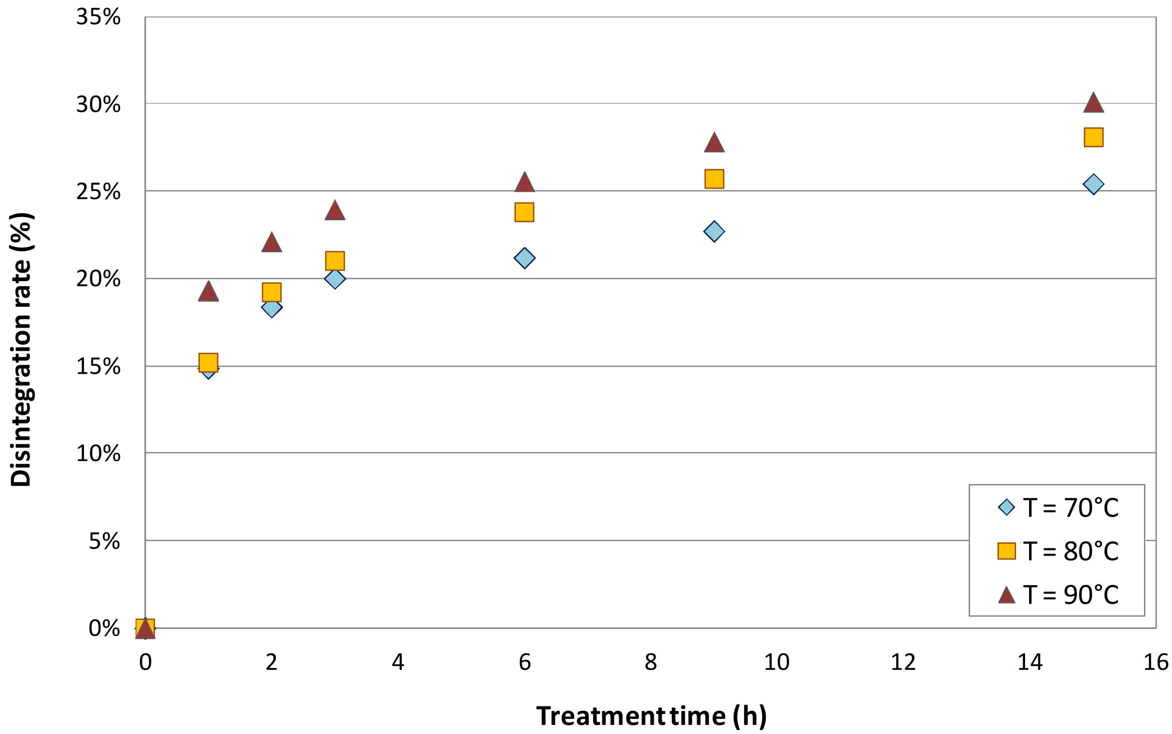
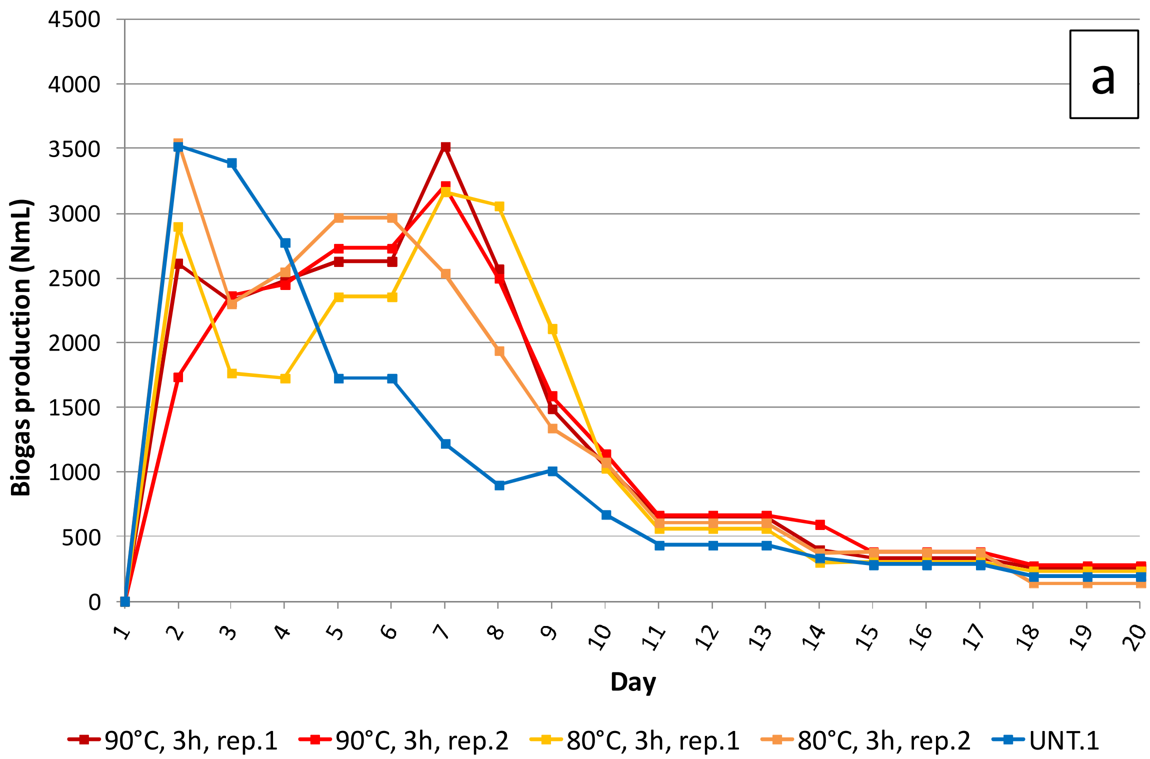
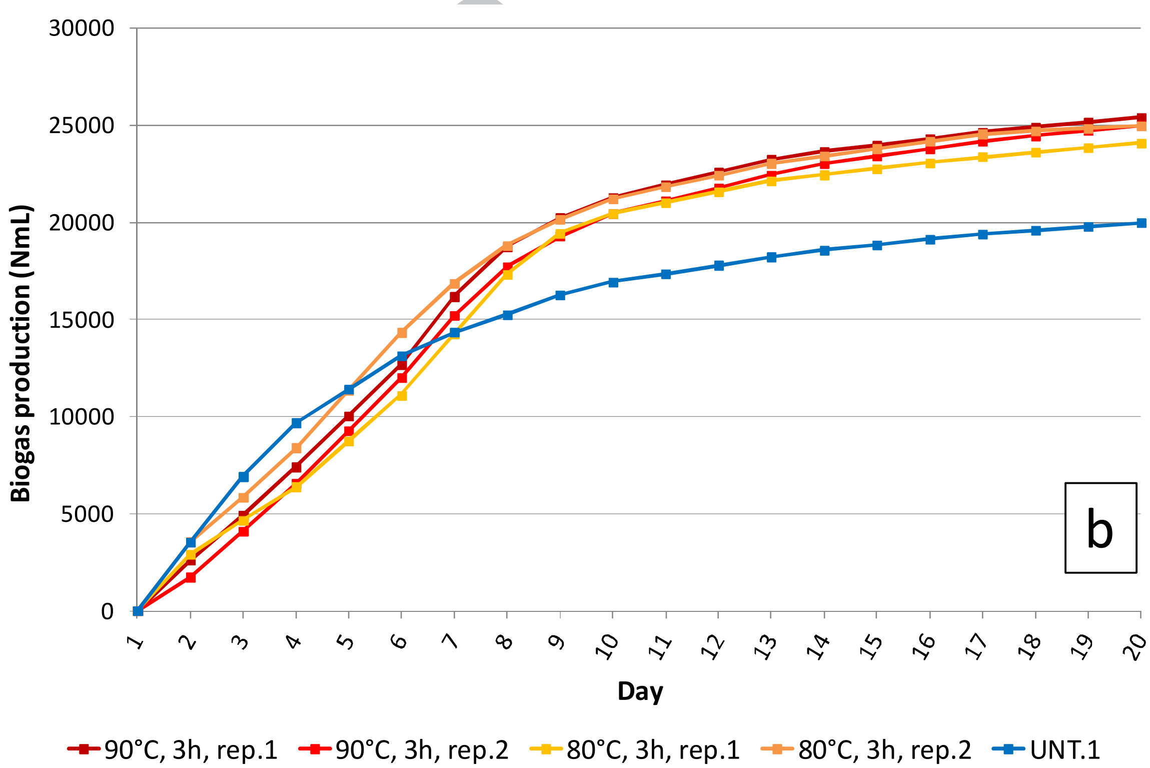

35%
30%
25%
20%
15%
10%
5%
0%
Disintegration rate (%)
0
2
4
6
8
10
12
14
16
Treatment time (h)
T = 70°C
T = 80°C
T = 90°C

Figure 2.

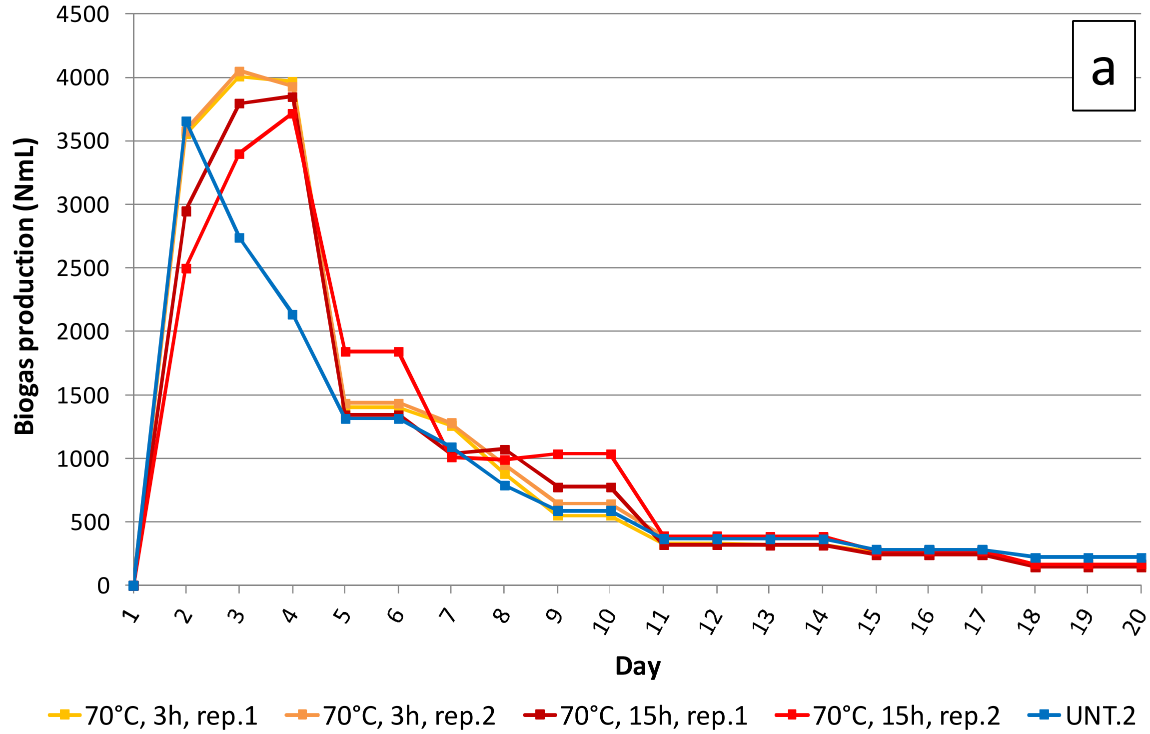


a
Biogas production (NmL)
Day
90°C, 3h, rep.1
90°C, 3h, rep.2
80°C, 3h, rep.1
80°C, 3h, rep.2
UNT.1

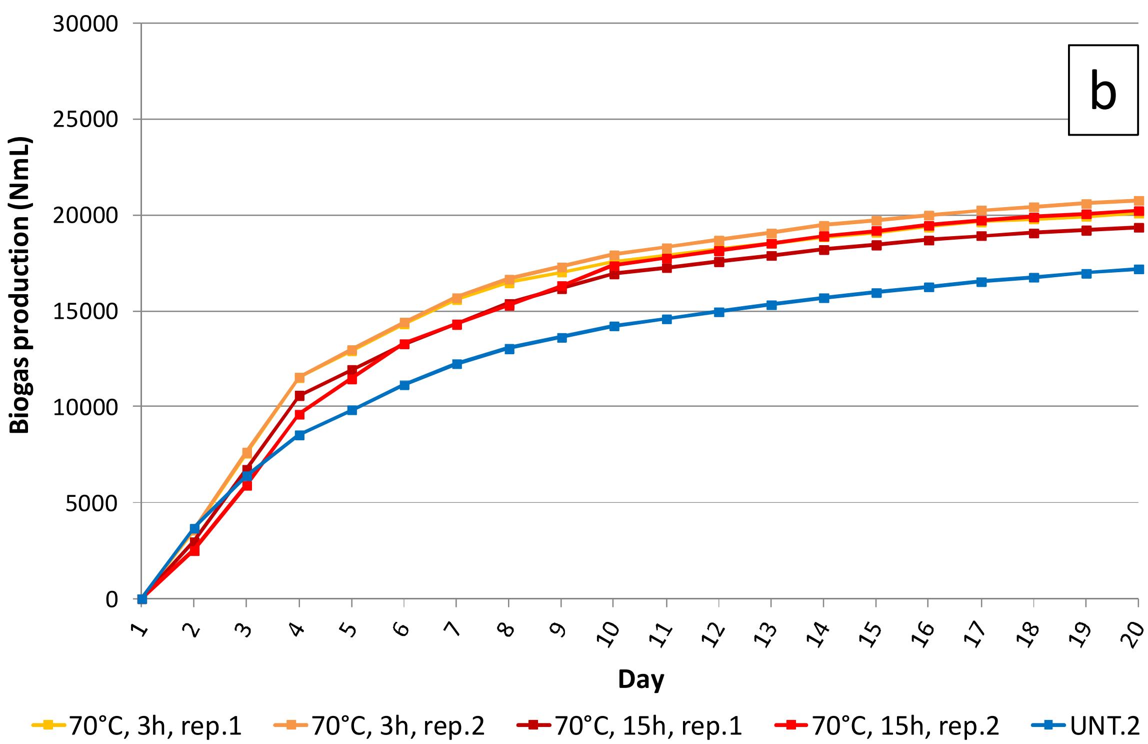


b
Biogas production (NmL)
Day
90°C, 3h, rep.1
90°C, 3h, rep.2
80°C, 3h, rep.1
80°C, 3h, rep.2
UNT.1

Figure 3.

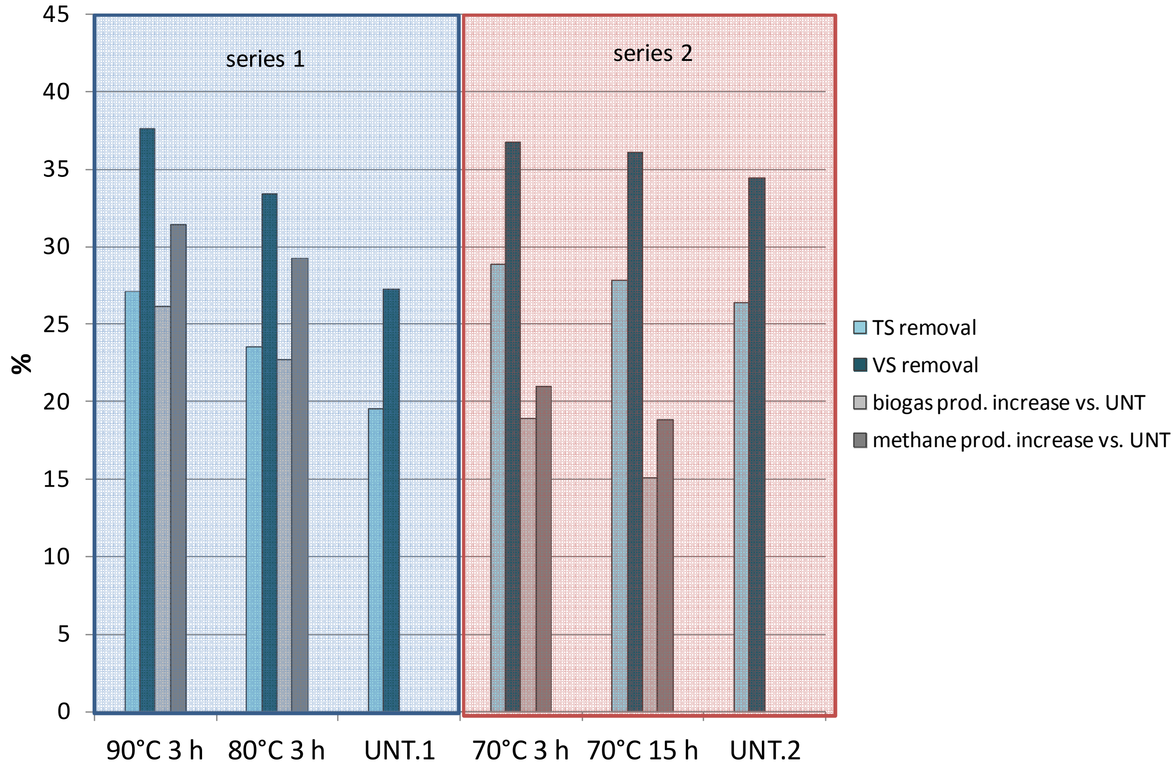


a
Biogas production (NmL)
4500
4000
3500
3000
2500
2000
1500
1000
500
0
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20
Day
70°C, 3h, rep.1
70°C, 3h, rep.2
70°C, 15h, rep.1
70°C, 15h, rep.2
UNT.2



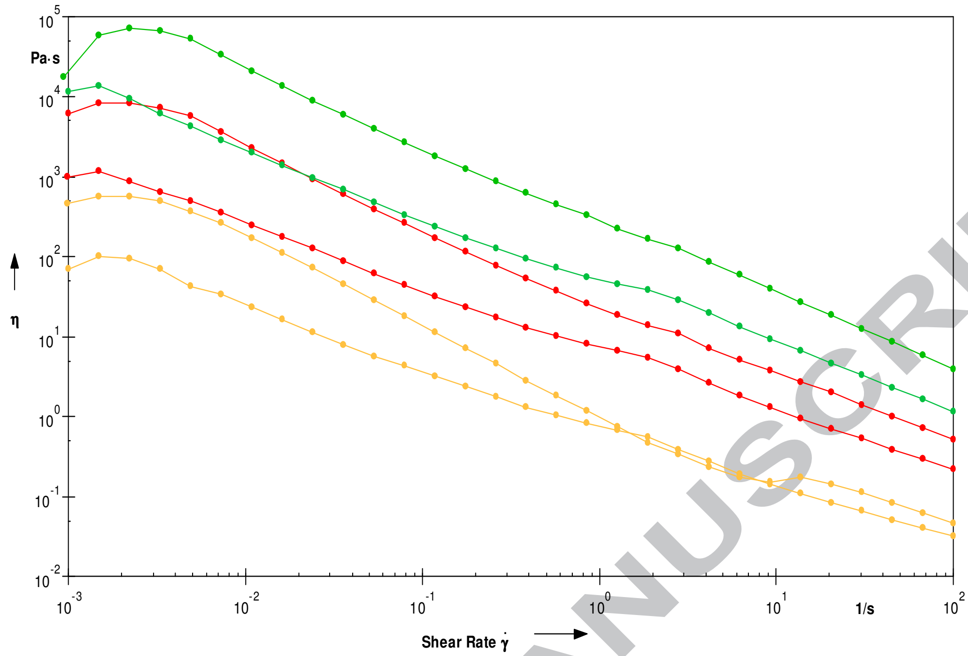
b
Biogas production (NmL)
30000
25000
20000
15000
10000
5000
0
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20
Day
70°C, 3h, rep.1
70°C, 3h, rep.2
70°C, 15h, rep.1
70°C, 15h, rep.2
UNT.2

Figure 4.



45
40
35
30
25
20
15
10
5
0
%
series 1
series 2
90°C 3 h
80°C 3 h
UNT.1
70°C 3 h
70°C 15 h
UNT.2
TS removal
VS removal
biogas prod. increase vs. UNT
methane prod. increase vs. UNT

Figure 5.



10^5
Pa·s
10^4
10^3
10^2
η
10^1
10^0
10^-1
10^-2
10^-3
10^-2
10^-1
10^0
10^1
1/s
10^2
Shear Rate γ̇

Highlights:

The efficiency of AD on WAS in the largest Italian WWTP has to be improved

Mechanical and low-temperature thermal pre-treatments were tested

Thermally treated samples produced from 20 to 30% more methane than untreated samples

Low-temperature thermal pre-treatments caused a significant decrease in WAS viscosity

Total revenues from electricity sale raised of 10% compared to the present scenario