Analysis of mesophilic and thermophilic anaerobic digestion process applied to pressed biowaste during transient/stressed conditions and co-digestion with waste active sludge

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Abstract

The anaerobic digestion and co-digestion with waste activated sludge of liquid pressed biowaste from kerbside collection was studied both in mesophilic and thermophilic conditions. The research focused on the aspects related to a comparison between mesophilic and thermophilic conditions, also considering the processes stability during “non steady state” conditions and in specific perturbation in loading rate. The second part of the research was specifically dedicated to the study of the co-digestion with waste activated sludge in mesophilic and thermophilic conditions. Continuous operation results indicated that anaerobic digestion of biowaste juice was viable at all operating conditions tested, with the greatest specific gas production of 0.94 $m^3/kg\ TVS_{fed}$ at an organic load rate of 4.7 kg $TVS/m^3_r d$ in thermophilic conditions while it was around 0.79 $m^3/kg\ TVS_{fed}$ in mesophilic ones. No significant unbalanced and critical conditions were observed during perturbed conditions, especially when higher OLRs were applied, suggesting the possibility of either increase the OLR in existing anaerobic reactors or reducing the design volumes of new reactors. The co-digestion experimentation was designed in terms of OLR referring to the daily specific production of sludge and biowaste per inhabitant equivalent. The OLR was lowered to 1.7 kgTVS/ $m^3_r d$ (half due to biowaste and half due to sludge). Observed biogas yields were 0.63 $m^3/kg\ TVS_{fed}$, in thermophilic conditions, and 0.56 $m^3/kg\ TVS_{fed}$ in mesophilic conditions, methane being 60% of biogas in both cases. The obtained results were generally consistent with the highest values reported in literature because of the high biodegradability of the pressed semi-liquid biowaste. Finally, the contents of heavy metals and pathogens of fed substrate and both effluent digestates, in the two experimentations, were analyzed to verify the quality of the resulting materials. These showed low levels for both the parameters thus indicating the good quality of digestate and its possible use for agronomic purposes.

Key words Anaerobic co-digestion, Anaerobic digestion, Food waste, mesophilic, thermophilic, pressed biowaste, transient conditions, wastewater sludge

Introduction

Anaerobic digestion (AD) is widely applied in Europe for treating organic wastes while recovering biogas, a flexible biofuel. At present there are some 14,000 AD plants working in Europe, ca. 25% dedicated to the treatment of municipal organic waste, like biowaste, food waste or wastewater sludge (www.european-biogas.eu). Mattheeuws and De Baere (2010) reported that at that time some 7 million tons of biowaste were anaerobically digested within EU Countries. Biowaste is normally pre-treated and prepared for the AD process by means of steps dedicated to the removal of inert material and size reduction. These steps are time and energy consuming and generally are not able to achieve high removal yields for inert materials like small pieces of plastics and heavy materials like crashed glass, see food or similar. Because of this situation, in recent years, also another option came in operation: biowaste pressing for the production of two streams, one semi-liquid to be digested and a second one solid to be composted (Hansen et al., 2007). Satoto Nayono et al. (2009) studied AD of pressed off leachate from Organic Fraction of Municipal Solid Waste and studied (2010) the co-digestion of press water and food waste for improvement of biogas production. The co-digestion of press water and food residues with defibred kitchen wastes (food waste), operated at 14-21 kgCOD/m^{3}d of OLR, reported higher biogas production rates then sole biowaste. An increment of the OLR of biowaste by 10.6% with press water increased the biogas production as much as 18%, with a biogas production rate of 4.2 m^3/m^{3}d at OLR of 13.6 kgCOD/m^{3}d.

According to this scenario, the present study considered the anaerobic digestion and co-digestion of the semi-liquid fraction deriving from a press specifically designed for separately collected (kerbside) biowaste treatment. Both mesophilic and thermophilic conditions were considered for both the AD and

Co-AD processes. The co-digestion experimentation was designed considering the average production of sludge and biowaste per inhabitant equivalent.

Biowaste and sewage sludge play an important role in the process of co-digestion: the nutrients (N, P, micro-elements) content in the biological sludge can fill a possible nutrient deficiency of the other co-substrate (biowaste in this case) as already demonstrated by the authors (Facchin et al., 2013).

Beside the operational parameters and yields also the digestate characteristics were considered in detail in order to respond to the requirements defined in the “End of Waste Criteria” technical proposal by the Joint Research Center of Sevilla of January 2014. Based on suggested criteria, pathogens and metals were analyzed in the digestates in order to identify the possibility to further treat anaerobic digestate in a co-composting process.

Materials and Methods

A specifically designed press was applied to pre-treat separately collected biowaste and split it into two streams, one liquid to be anaerobically digested and a second one solid to be composted. This experimentation allowed for the assessing of the improvement of performance in terms of biogas yields with the use of the extremely biodegradable liquid stream intended for anaerobic digestion, both in mesophilic and thermophilic conditions. Treated biowaste was previously sieved and metallic residues removed. The material was shredded into a knife mill, then in a under-sink food waste disposer, and finally treated in a press for the separation and the maximum recovery of the more easily biodegradable fraction. Only the "juicy" fraction was then sent to the anaerobic process while the semi-solid part characterized by a higher content of dry matter was suitable for a subsequent stabilization process as composting. The semi-liquid stream was then sent to two CSTR anaerobic digesters, one mesophilic (37°C) and the other thermophilic (55°C), working with an organic loading rate in the range 3 - 6 kgTVS/m^3_r per day and a hydraulic retention time of 20 days to simulate the best operating conditions expected at a full-scale treatment plant. The research was carried out using two pilot scale reactors completely equal in terms of: electro-mechanics, working volume (0.23 m^3) and heating system. The reactors are made of stainless steel AISI-304, the mixing is ensured by mechanical arms-still agitators in order to maximize the uniformity material degree inside the reactor, thus avoiding the classical stratification of floating materials and heavy in the head and on the bottom of the reactor. The temperature of 37 °C (mesophilic thermal regime) and 55 °C (thermophilic thermal regime) of the reactors is maintained constant by an external jacket, in which heated water by a boiler is recirculated, and proper structure insulation. The biogas produced is sent to a hydraulic guard with the purpose of maintaining an operating pressure of 0.1 m water column in the gas line. The biogas production is monitored by a flow meter by Ritter Company™. The percentages of methane, carbon dioxide and oxygen are determined through a portable infrared gas analyzer GA2000™ (Geotechnical Instruments™). The content of heavy metals and pathogens of fed substrates and digestates, in the two experimentations (AD and Co-AD), were analyzed.

Analytical methods

The biowaste originated by a door-to-door collection scheme. It was analyzed at commodity class in accordance with the procedure reported by MODECOM™ (1998). The reactor effluents were monitored 3 times per week in terms of TS, TVS, COD, TKN and P total. The process stability parameters, namely pH, volatile fatty acid (VFA) content and distribution, conductivity, total and partial alkalinity and ammonia (NH_4^+–N), were checked daily. All the analyses performed according to the Standard Methods for Water and Wastewater Analysis (1998).

The anaerobic digestion test has provided, after inoculation of the reactor, a start-up phase of the reactor characterized by a gradual increase of the organic loading rate (OLR). In steady state conditions, the OLR applied was in the range 3.0 and 3.5 kgTVS/m^3_rd and the hydraulic retention time of 20 days, afterward an OLR period to about 4.5 $kgTVS/m^3{}_r d$, testing systems on tolerance to transient conditions of OLR from 3 to 6 $kgTVS/m^3{}_r d$.

The co-digestion option was evaluated considering the typical per capita production of activated sludge and the organic fraction of municipal solid waste, exactly 40 grams of dry activated sludge per inhabitant / day and 100 g of organic waste per inhabitant / day.

Results and discussion

The biowaste collected in Treviso area and used in this experimentation showed an average dry matter content of 298 gTS/kg, 90% volatile solids. The potential high biodegradability of the substrate is expressed through the COD values, typically greater than 1000 g/kg.

The biowaste composition showed that the content of the organic wastes were higher than 82% (on wet weight) while the remaining parts were paper (11%) and inert materials (7%) like glass and metals or textiles. The different fractions for each type of material in terms of wet and dry weight, and volatile fraction are shown in Table 1.

Table 1. Commodity characteristics of biowaste

Fractions	**Wet weight**	**Dry weight**	**Volatile**
	%	%	%
paper	10.75	11.25	12.87
plastic	0.85	1.20	1.31
inert	6.88	11.33	8.16
organic	81.52	76.22	77.66

The typical composition of the organic fraction is shown in Table 2: fruit and vegetable waste are typically half of the waste material, a result in line with previous studies on this topic (Valorgas D2.1 http://www.valorgas.soton.ac.uk/deliverables.htm) while pasta/bread and meat/seafood represented another 25% of the wasted food.

The semi-liquid biowaste obtained after pressing showed the characteristics reported in table 3, thus this substrate is particularly suitable for biological treatment processes.

Table 2. Organic fraction characterization

Organic commodity class	% WW	% DW
Fruits & Vegetables	46-58	42-52
Other kitchen waste *	16-25	15-22
Paper & paperboard	9-14	7-12
Not classifiable	8-14	6-12

* Putrescible material non-vegetable (eg pasta, cakes, meat, etc..). WW = wet weight. DW = dry weight

Table 3. Semi-liquid biowaste characterization

Parameter	m.u.	Average	Max	Min
Total Solids	gTS/kg	188	241	128
Total Volatile Solids	gTVS/kg	164	211	112
TVS/TS	%	88	93	84
COD	mgO_2/gTS	1061	1137	998
TKN	mgN gTS	27	36	19
TP	mgP gTS	3.8	5.0	3.0

Mesophilic digestion and co-digestion

The inoculum used for the mesophilic trials was a digestate originated from a full-scale process of anaerobic digestion. The reactor was maintained at the operating temperature of 37°C, feeding with a low loading rate and intermittent for a week, in order to acclimatize the biomass to the organic matrix fed, the latter being characterized by a high biodegradability.

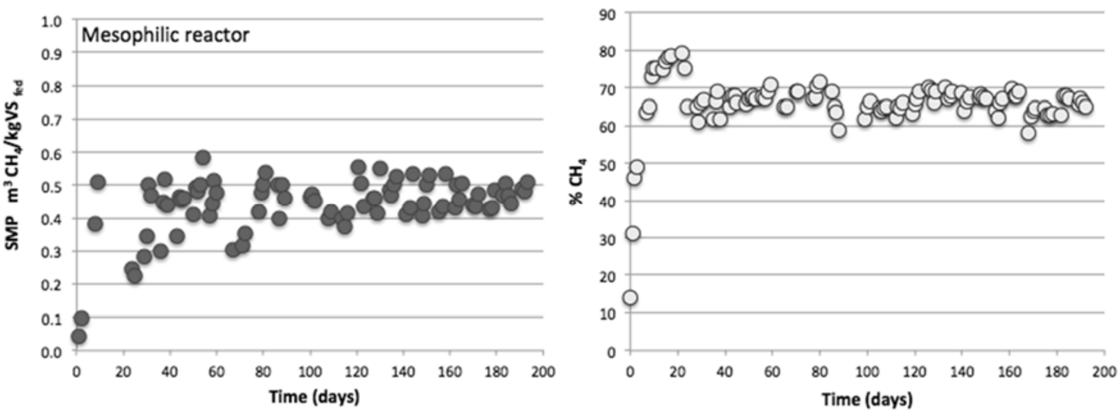
Once the methanogenic biomass was active and responding appropriately in terms of biogas quality, the reactor was fed daily, increasing the organic loading rate from 1 kgTVS/m^3_rd to 3.5 kgTVS/m^3_rd in 2 HRT. When the steady state condition was reached, VFA concentration were less than 2 g/l, with an average value of 912 gVFA/l. COD soluble (sCOD) showed an average value of 2294 mgCOD/l. The use of a real substrate and the heterogeneity of the MSW organic fraction determined inevitable variations in the solid content (standard deviation ± 60 gTS/kg); accordingly, the OLR fluctuated between 3 and 6 kgTVS/m^3_rd, thus imposing a transient condition to the system. The system didn't show any upset to its stability, thus indicating good robustness of the process also in transient conditions, a relevant aspect for the full-scale implementation of the process. As for pH, this remained constant, particularly in steady state conditions, with an average value around 7.7 because of the high buffer capacity of the system: this is highlighted by an average total alkalinity value of 5177 mg$CaCO_3$/l. The ratio between VFA concentration and alkalinity was also evaluated. The concentration of volatile fatty acids and alkalinity are the two parameters that show a more rapid variation when the system trends to withdraw from steady state, since in case of problems, the concentration of fatty acids verges to increase while the alkalinity tends to decrease. A useful parameter to consider is the relationship between these two amounts (Cecchi et al., 2005). Ratio values around 0.3 indicate a stable operation of the digester, while higher values may indicate the inception of instability.

In the period in which the organic load increased, the ratio was around 0.22, thus the system achieved a perfect steady state even with the transient increasing of the organic loading rate.

The monitoring of the total ammonia evinced an average value of 878 mgN-NH_4^+/l and 64 mgN-NH_3/l as free ammonia, well below the typical critical level for inhibition (Chen et al., 2008). The average content of total solids remained almost constant with an average value of 22.7 gTS/kg and an average volatile solids content of 16 gTVS/kg. The ratio between total solids and volatile shows an average value of 70.4% (TVS/TS), it's thus highlighted the capability of the system of converting the organic matter into biogas, leaving a residual dry matter content lower than 3% in digestate.

The biogas composition in terms of average percentage of methane detected in steady state condition (SSC) was 66% CH_4 and the remaining part CO_2. The specific methane production (SMP) average was 0.47 m^3CH_4/kgTVS. Specific methane production and percentage is shown in figure 1.

Figure 1. SMP and CH_4% trends.



Referring to biowaste input to the digester, it can be estimated a biogas production of about 160-180 m^3 per ton of treated biowaste and an energy yield around 350 kWh of electrical energy per tonne treated. In steady state condition the process showed great strength and resilience with reference to the process parameters (pH, alkalinity and VFA concentration, and biogas composition).

The average specific gas production (SGP) value and the gas production rate (GPR) were: 0.79 m^3biogas/kgTVS and 2.3 m^3biogas/m^3_rd, interesting with maximum values reached in SGP 0.82 m^3biogas/kgTVS in a period of increased OLR back to 4.5 kgTVS/m^3_rd, with a duration of 4 HRT. Interesting values in the velocity of biogas production were achieved in the latter period, precisely average value of 3.1 m^3biogas/m^3_rd.

During SSC of the mesophilic reactor, with OLR between 3 and 3.5 kgTVS/m^3_rd, the mass balance around the system was calculated: the influent and effluent (as both digestate and biogas) quantity of volatile solids accounted for 790, 184 and 512 g/d, respectively. The balance was therefore 88%, with a 12% error. Similar results were found for dry matter and COD thus confirming the quality of the calculation.

Also the mass balances for nutrients (nitrogen and phosphorus) closed well.

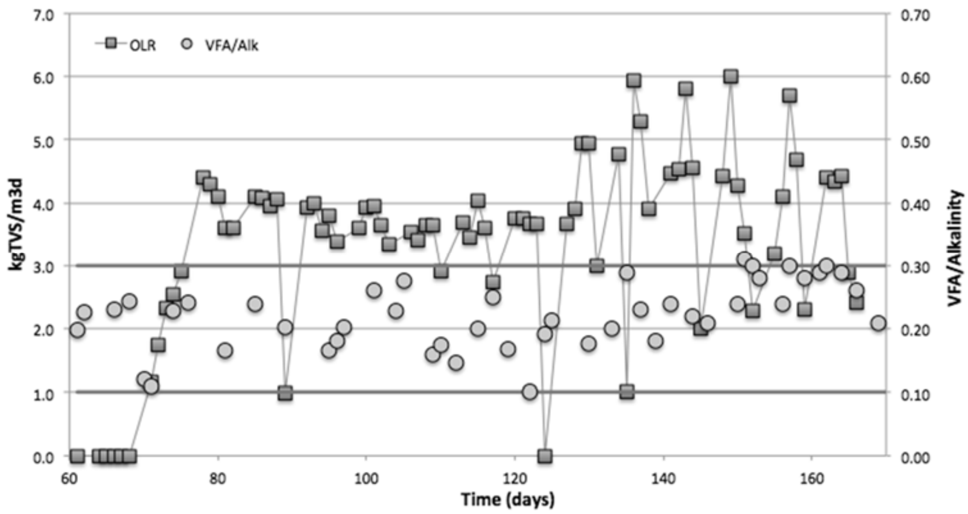
The co-digestion option was evaluated considering the typical per capita productions of WAS and biowaste, namely 40 g of dry matter for sludge per inhabitant day and 100 g of biowaste per inhabitant per day. The results obtained with an average OLR of 1.7 kgTVS/ m^3_rd. The observed yield was in values of SGP 0.56 m^3/kg TVS_{fed} in mesophilic conditions, consistent with literature data.

Thermophilic digestion and co-digestion

The inoculum used for starting up the thermophilic reactor originated from the same full-scale reactor used for the mesophilic reactor. After the first days of operation in mesophilic conditions the temperature reactor was turned up form 37°C to 55°C in one step, stopping the feeding. The thermophilic conditions were reached in a couple of days. The reactor was then maintained at a constant temperature of 55°C. Feeding was then started again, with a low organic loading rate (1 kgTVS/m^3_rd) for a week, with the aim of acclimate the biomass to the organic material fed. Then OLR was then increased to 3.5 kgTVS/m^3_rd in 2 HRT.

The concentration of the short chain volatile fatty acids remained constantly below 1 g/l, with an average value of 489 mgVFA/l, acetate being the main compound found. The average pH of digestate was 8.1. The buffer capacity of the system has maintained the pH compatible with the methanogenic thermophilic conditions. Average total alkalinity (determined at pH 4) in steady-state conditions was 5380 mg $CaCO_3$/l. Partial alkalinity (determined at pH 5.75) showed a profile in line with the trend of the volatile fatty acids, and consequently the difference between partial and total alkalinity, which is directly proportional to the concentration of VFA, remained constant. The values of partial and total alkalinity stood at 5200 and 3900 $CaCO_3$/l corresponding to a ratio between the difference of the two alkalinities constantly equal to 0.23. Even in the thermophilic reactor this relationship was stable thus this assessment justifies the fact of the increasing OLR period to 4 - 4.5 kgTVS/m^3_rd testing also transient OLR conditions from 3 to 6 kgTVS/m^3_rd. The VFA/alkalinity ratio had an average value of 0.27 as shown in figure 2.

Figure 2. Trend of the ratio volatile fatty acids and alkalinity with OLR

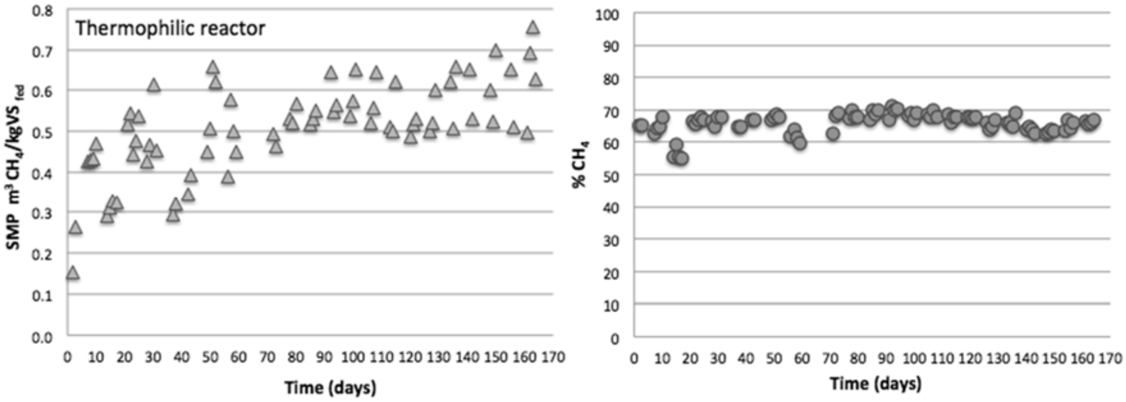


Therefore the system achieved a perfect steady state condition also during increasing OLR conditions. The monitoring of the total and free ammonia showed values distant from a possible inhibition of the process. The average total ammonia concentration (as mgN-NH_4^+/l) was 1004 mgN-NH_4^+/l, up 1086 mgN-NH_4^+/l in the following period where OLR increased while the corresponding value of free ammonia was 374 mgN - NH_3/l, a value below the level of inhibition normally reported in literature: some 700 mgN/l and more (Angelidaki et al., 1994).

The average content of total solids remained almost constant with an average value of 16.3 gTS/kg and an average volatile solids content of 12.3 gTVS/kg. The ratio between total solids and volatile shows an average value of 76.5% (TVS/TS), it's thus highlighted the large capacity of the system to convert the organic matter into biogas, leaving a residue of dry matter lower than 2% in digestate.

The composition of the biogas in terms of methane average percentage was 68.8%. The specific methane production (SMP) was 0.55 m^3CH_4/kgTVS. Specific methane production and CH_4 percentage are shown in figure 3. The production of biogas was estimated at about 190 m^3 per tonne (14% greater than the corresponding mesophilic trial) for a production of electric energy greater than 400 kWh per ton. The GPR increased up to 3.4 m^3biogas/m^3_rd with an average value in SSC of 3 m^3biogas/m^3_rd; very interesting value from the point of view of the full-scale implementation of the process. SGP reached an average value as high as 0.94 m^3biogas/kgTVS at an OLR 4.5 kg TVS/m^3_rd period (4 HRTs), reporting an average increased value in biogas production of 0.9 m^3biogas/$kgTVS_{fed}$, absolutely high and considerable value from a scientific point of view, similar to the ultimate biogas value obtainable from food waste.

Figure 3. Specific methane production in thermophilic anaerobic digestion and methane percentage



During the steady state condition of the thermophilic reactor, with OLR between 3 and 3.5 kgTVS/$m^3{}_r$d, the mass balance around the system was calculated: the influent and effluent (as both digestate and biogas) quantity of volatile solids accounted for 790, 141 and 578 g/d, respectively. The balance was therefore 91%, with a 9% error. Similar results were found for dry matter and COD (errors of 11% and 11.5%, respectively) thus confirming the quality of the calculation. Also the mass balances for nutrients (nitrogen and phosphorus) closed well.

The results obtained during the co-digestion of WAS and the liquid fraction of biowaste in thermophilic conditions were interesting and the SGP was 0.63 m^3/kg TVS_{fed}. These obtained achievements were generally consistent and higher with literature data, acknowledgment to the high biodegradable pressed biowaste in co-digestion with WAS.

Digestate characteristics

Heavy metals concentrations of the mesophilic digestate and the substrate were evaluated. The concentration of heavy metals in both the input matrix (biowaste) and in mesophilic digestate reported values much lower than the limits of the technical proposals End-of-Waste criteria (EoW-2014) elaborated by the Joint Research Center of Sevilla. The test methods, used to analyze these concentrations, have been adopted were: EPA 3051A 2007 + EPA 6020A 2007.

EU Reg.organic agriculture	**EoW 2014**	**BIOWASTE**	**WAS**	**MESOPHILIC DIGESTATE**	**CO - DIGESTATE**
Cu mg/kg d.m.	200	47	249	68.1	138
Zn mg/kg d.m.	600	112	1015	155	452
Pb mg/kg d.m.	120	1.54	39	17.3	0.2
Ni mg/kg d.m.	50	43.7	30	42.1	17.4
Cr tot mg/kg d.m.	100	61.5	40	85.9	34.8
Cd mg/kg d.m.	1.5	0.4	0.2	0.23	0.1
Hg mg/kg d.m.	1	0.055	0.2	0.24	0.1
As mg/kg d.m.	10	0.24	9	0.25	0.2

Heavy metals concentrations of the thermophilic digestate and the substrate were evaluated as well. The concentration of heavy metals in both the input matrix and the digestate reported values much lower than the limits of the technical proposals End-of-Waste criteria (EoW-2014). The test methods, used to analyze these concentrations, have been adopted were EPA 3051A 2007 + EPA 6020A 2007.

EU Reg.organic agriculture	**EoW 2014**	**BIOWASTE**	**WAS**	**THERMOPHILIC DIGESTATE**	**CO - DIGESTATE**
Cu mg/kg d.m.	200	47	249	52.5	105.8
Zn mg/kg d.m.	600	112	1015	129	352

Pb mg/kg d.m.	120	1.54	39	7.81	0.1
Ni mg/kg d.m.	50	43.7	30	27	23.5
Cr tot mg/kg d.m.	100	61.5	40	51.5	29.4
Cd mg/kg d.m.	1.5	0.4	0.2	0.26	0.1
Hg mg/kg d.m.	1	0.055	0.2	0.08	0.1
As mg/kg d.m.	10	0.24	9	0.19	0.1

It is therefore evident that digestate presented characteristics in line with the end-of-waste criteria for heavy metals in all tested cases.

The analysis of heavy metals concentrations in input substrates (biowaste and sewage sludge) outlined how the organic waste is within the limits, whereas the activated sludge concentrations of copper and nickel were higher than the regulatory limits. Of particular interest is the result of the concentration of metals in the digestate co-digestion, it showed values much lower than the limits End-of-Waste (EoW2014), this to specify how, for the characteristics of these digestate, they can be reused.

Pathogens concentrations

The content of pathogens of fed substrate and both effluents digestates, in the two experimentations, were analyzed. While Salmonella spp was never found, the limit of 1000 CFU/g for E.coli proposed in the End of Waste Criteria technical report (2014) was reached only in some occasions. This suggests the opportunity to treat digestate in a post-composting process.

SAMPLE	TBC 37°C ISS A004A	TBC 22°C ISS A004A	*E.coli*	total Coliform	*Salmonella spp* ISS 011A

BIOWASTE	**$4 \cdot 10^8$ UFC/g**	**$8 \cdot 10^8$ UFC/g**	**$7 \cdot 10^5$ UFC/g**	**$6 \cdot 10^5$ UFC/g**	**absent**
WAS	**$3 \cdot 10^8$ UFC/g**	**$6 \cdot 10^8$ UFC/g**	**$1 \cdot 10^4$ UFC/g**	**$3 \cdot 10^6$ UFC/g**	**absent**
Thermophilic	**$1 \cdot 10^7$ UFC/g**	**$1 \cdot 10^7$ UFC/g**	**$4 \cdot 10^3$ UFC/g**	**$1 \cdot 10^3$ UFC/g**	**absent**
Mesophilic	**$3 \cdot 10^6$ UFC/g**	**$4 \cdot 10^6$ UFC/g**	**$3 \cdot 10^3$ UFC/g**	**$2 \cdot 10^4$ UFC/g**	**absent**

Conclusions

The use of a press for treating separately collected biowaste produced a semi-liquid stream characterized by a dry matter content of about 20% and a very high degree of biodegradability. The solid fraction determined by the press should be used for composting together with the dewatered anaerobic digestate and some bulking material.

The semi-liquid stream then underwent to AD. After 10 HRT the mesophilic digestion process showed a biogas production of 0.79 m^3/kgTVS, with 66% of methane. With an OLR of 4.5 kgTVS/m^3_rd it reached values of 0.82 m^3_{biogas}/kgTVS$_{fed}$.

The process maintained its stability conditions also subjected to the increase in organic load and transient conditions of OLR from 3 to 6 kgTVS/m^3_rd. The co-digestion option had an average OLR of 1.7 kgTVS/ m^3_rd, the observed yield was SGP 0.56 m^3/kg TVS$_{fed}$

In thermophilic conditions the biogas production showed an average value of 0.8 m^3/kgTVS, with an organic load of 3.5 kgTVS/m^3_rd and over 68% of methane. Significant and remarkable values of specific biogas production of 0.94 m^3_{biogas}/kgTVS$_{fed}$ and 0.7 m^3CH_4/kgTVS$_{fed}$ at 4.7 kgTVS/ m^3_rd. The system maintained its steady state even when subjected to the increasing organic load and subsequent transient OLR conditions. The co-digestion option had an average OLR of 1.7 kgTVS/ m^3_rd, the observed yield was SGP 0.63 m^3/kg TVS$_{fed}$.

In meso/thermo conditions and co-digestion the concentrations of the main heavy metals and pathogens were below the limits reported for the definition of the end-of-waste criteria for future legislative developments (End of Waste criteria 2014). The concentrations low levels for both the parameters thus indicating the good quality of digestate and its possible use for agronomic purposes.

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