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OPTIMIZATION OF HYDROGEN AND METHANE PRODUCTION IN TWO-PHASE ANAEROBIC DIGESTION*

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Abstract

Bio2Energy project aims at improving the efficiency of public utility facilities such as wastewater treatment plants and increasing the production of renewable energy in Tuscany. The production of biohydrogen and biomethane from the co-digestion of Organic Fraction of Municipal Solid Waste (Food Waste, FW) and sewage waste sludge (WS) and the use of digestate as agricultural fertilizer are the main objectives that position the project within the Circular Economy Strategy. Anaerobic digestion is a proven technology for the production of renewable energy but this technology is even more interesting if it is exploited also for hydrogen production. Therefore, the production of biohydrogen occurring in the acidogenic phase could be associated with anaerobic digestion (AD) so as to generate two gas streams (H_2 and CH_4), useful for different stakeholders, separately or mixed together. Our results revealed that the anaerobic co-digestion of sludge and food waste is an effective process able to produce energy self-sufficiency of wastewater treatment plants. Two-phase anaerobic digestion tests demonstrate the possibility to assess the production of hydrogen and methane in specific cases (sludge and food waste) and estimate the primary energy produced by different biogas users.

Keywords: anaerobic digestion; biohydrogen; biomethane; dark fermentation

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1. Introduction

Transitioning to a more circular economy is an essential contribution to the EU's long-term efforts to advance a competitive, sustainable, low-carbon and resource-efficient economy (European Commission, 2017). Two-phase anaerobic digestion process obtained by the union of biohydrogen production during the acidogenic phase (named Dark Fermentation - DF) and biomethane production in AD, can be considered the new borders of AD process optimization (Ghimire et al., 2015).

Most of the conventional wastewater treatment plants (WWTPs) use AD for the treatment of the produced sludge by using digesters with spare capacity to face variation in wastewater flow and future population growth (Nghiem et al., 2017). Due to low organic loading and low biogas yields of sludge, energy recovery via anaerobic digestion in WWTPs is typically not sufficient to cover its energy consumption. Co-digestion of bio-waste with municipal sewage sludge is nowadays consider one of the most strategic approach in waste and wastewater management thus increasing the energy production, reduce costs and facilitating nutrient recycling (Cavinato et al., 2013; Da Ros et al., 2014; Cavinato et al., 2014; Nghiem et al., 2017). Among the available substrates that have been tested for co-digestion, Food Waste (FW) is an optimum co-substrate in order to improve digestion efficiency of sewage sludge because of its readily biodegradability nature (Iacovidou et al., 2012). Furthermore, the co-digestion of these two substrates could be potentially suitable also for biohydrogen production from dark-fermentation. Indeed, due to their considerable alkalinity, sludge could be used to control pH in the optimal range for biohydrogen production avoiding drops that can bring to the failure of the process when using only FW (Jung et al., 2011; Zhu et al., 2011).

In this study, the production of methane and hydrogen of food waste (FW) and wastewater sludge (WS) were experimentally determined in order to compare possible upgrading solutions for a WWTP in Tuscany. Two possible layouts of FW and WS co-digestion were compared with the current WWTP (reference scenario). In these two co-digestion scenarios was evaluated the possibility to produce hydrogen by adding a new digester to perform dark-fermentation. For each scenario the mass balance, the energy budget and the greenhouse gas account were estimated.

The data used in the inventory were collected from several sources. The production of methane and hydrogen were determined by performing Biochemical Methane Potential (BMP) and Biochemical Hydrogen Potential (BHP) assays, that are well recognized among the scientific community as valuable, simply and low cost tools to assess the potential, adequacy and viability of the fermentative and methanogenic process (Cappai et al., 2014; Holliger et al., 2016; Labatut et al., 2011; Pecorini et al., 2016). The experimental data will be used as preliminary results to develop the two-phase co-digestion process in pilot and pre-industrial scale. Other data were obtained by direct management data of the WWTP, calculations and esteems.

2. Materials and methods

2.1. Food waste and wastewater sludge characterization

The WS and the FW used in the BMP and BHP assays were sampled at two treatment facilities in Tuscany. The WS was collected from the aerobic unit of the municipal wastewater treatment plant of Viareggio (LU, Italy) while the FW were sampled form the Organic Fraction of Municipal Solid Waste (OFMSW) delivered to a mechanical-biological treatment facility in the province of Florence. In particular, to obtain the FW samples, approximately 10 tons OFMSW were investigated by means of a picking analysis

(Laegerkvist et al., 2011). This sample was manually sorted in the following fractions: FW (45.8% w/w), garden waste (44.7% w/w), textiles (0.5% w/w), paper and cardboard (2.2% w/w), metals (0.1% w/w), wood (1.1% w/w), plastics (1.7% w/w), glass (1.2% w/w) and other (2.6% w/w). The sorted FW was then used for biohydrogen and biomethane production owing to its recognized potential (Holliger et al., 2016; Xiao et al., 2013). In order to homogenize the sample and to make it suitable for a wet fermentation technology, FW was grinded by blender and diluted with tap water until it reached a total solids (TS) content of approximately 15% w/w. In both BMP and BHP tests, sludge from an anaerobic reactor treating OFMSW was used as inoculum.

FW, WS and the inoculum were characterized through physical, chemical and bromatological analyses (Table 1). TS, Total Volatile Solids (TVS) and pH were determined according to standard methods (APHA, 2006). According to Angelidaki et al. cited by Holliger et al. (2016), TS determination was performed at 90°C instead of 105°C until constant weight in order to avoid the volatilization of volatile fatty acids. Proteins, lipids, cellulose, Total Kjeldhal Nitrogen contents were measured in accordance with the European Commission (2009). Carbohydrates were then calculated by subtracting to the total amount, the contents of humidity, ashes, proteins, lipids and fibers. Lignin was measured according to (MP 0424, 2010). Concerning the elementary composition C, H, N were obtained following (EN 15407, 2011) while S and P where measured using (EPA 6010 D, 2014) and (EN 13657, 2004) respectively. The oxygen content was estimated by subtracting the sum of C, H, N, S and P from the total. Ammonia was measured according to (APHA, 2012) while Total Organic Carbon (TOC) was measured thanks to (Ministero dell'Agricoltura e delle Foreste, 1989). Volatile Fatty Acids (VFAs: acetic and propionic acids) were measured according to (MP 0224 2012).

2.2. Hydrogen and methane production tests

2.2.1. Biochemical Hydrogen Potential (BHP) tests

The production of hydrogen from FW and SW was experimentally determined by performing biochemical hydrogen potential (BHP) assays. The analyses were conducted based upon the method described by Alibardi and Cossu (2015). The test was performed in triplicate using 1 L stainless steel batch reactors (Pecorini et al., 2016). The vessels were incubated in a water bath at 37°C for 7 days. After set-up the bottles were flushed with N_2 for few minutes to ensure anaerobic conditions in the headspace of the batches. The bottles were daily shaken to guarantee homogeneous conditions in the assay vessels. Each vessel was loaded with a Food/Microorganism ratio of 1/4 (w/w). The working volume of the bottle was approximately 0.5-l and consisted of inoculum, substrate, MES (2-N-Morpholino-EthaneSulfonic acid, VWR, Italy) buffer solution and HCl 2.5M to set initial pH at a value of 5.5. After set-up, the vessels were flushed with N_2 for few minutes to ensure anaerobic conditions. The inoculum, was previously heat-treated at 80°C for 30 minutes with the aim to select only hydrogen producing bacteria and inhibit hydrogenotrophic methanogens (Alibardi and Cossu, 2015; Cappai et al., 2014; Li and Fang, 2007). Biogas production was daily estimated by measuring the pressure in the headspace of each reactor and then converting to volume by the application of the ideal gas law. Pressure was measured using a membrane pressure gauge (Model HD2304.0, Delta Ohm S.r.L., Italy). The measured values of pressure were converted into biogas volume by Eq. (1).

$$V_{biogas} = \frac{P_{measured}\, T_{NTP}}{P_{NTP}\, T_r} V_r \qquad (1)$$

where: V_{biogas} - volume of daily biogas production, expressed in Normal liter (NL); $P_{measured}$ -

headspace pressure before the gas sampling (atm); T_r and V_r - temperature (K) and volume (L) of the reactor's headspace; T_{NTP} and P_{NTP} - normal temperature and pressure (273.15 K and 1 atm respectively).

Table 1. Food waste (FW), wastewater sludge (WS) and inoculum characterization

	FW	***WS***	***Inoculum***
TS (% w/w)	17.5 ± 0.8	1.9 ± 0.0	2.7 ± 0.1
TVS/TS (% w/w)	73.0 ± 1.5	79.9 ± 0.5	48.7 ± 0.5
pH	4.0 ± 0.2	4.4 ± 0.0	7.4 ± 0.3
TKN (%N w/w)	5.4 ± 0.4	0.2 ± 0.0	0.2 ± 0.0
TOC (%C w/w)	10.6 ± 1.9	1.2 ± 0.2	1.3 ± 0.2
Ammonia (mgN/kg)	856 ± 72	341 ± 47	1,040 ± 82
Acetic acid (mg/L)	5,200 ± 1,050	830 ± 120	< 40
Propionic acid (mg/L)	85 ± 26	390 ± 71	< 40
C (% TS)	53.8 ± 4.0	58.9 ± 4.3	50.8 ± 3.7
H (% TS)	5.7 ± 0.5	6.4 ± 0.5	3.9 ± 0.3
N (% TS)	3.4 ± 0.5	7.5 ± 0.8	8.0 ± 0.9
S (% TS)	0.2 ± 0.0	0.2 ± 0.0	0.2 ± 0.0
P (% TS)	0.6 ± 0.1	0.4 ± 0.1	0.4 ± 0.1
O (% TS)	36.3	26.6	36.7
Proteins (% w/w)	3.8 ± 0.2	0.9 ± 0.1	1.2 ± 0.1
Lipids (% w/w)	2.2 ± 0.2	< 0.3	< 0.3
Carbohydrates (% w/w)	6.9	0.1	0.1
Cellulose (% w/w)	3.5 ± 0.4	0.3 ± 0.3	0.4 ± 0.3
Lignin (% w/w)	1.3 ± 0.2	0.3 ± 0.0	0.3 ± 0.0

The BHP was determined as the cumulated hydrogen production divided by the TVS content contained in each batch. In order to determine the hydrogen production, the hydrogen content of the gas was measured by using gas cromatography (3000 Micro GC, INFICON, Switzerland).

2.2.2 Biochemical Methane Potential (BMP) tests

Biochemical Methane Potential (BMP) assays were performed for 21 days in order to determine the methane production of FW and WS. The analysis were conducted in triplicate based upon previous researches (Pecorini et al, 2016) and following the basic guidelines and advices included in Holliger et al. (2016). Each reactor was loaded with different amounts of substrate to achieve a concentration of substrate of about 2 gTVS/100 mL solution in each batch. This concentration is a compromise of, one hand, the need to use a large sample to have a good representativeness and to get a high easy-to-measure gas production, and, on the other hand, to avoid too large and impractical volumes of reactors and gas production and keep the solution dilute to avoid inhibition from accumulation of VFA and ammonia (Hansen et al., 2004). The inoculum to sample ratio was about 1.5:1 TVS basis and kept under 10:1 weight basis according to Pecorini et al. (2016) for fresh feed-in substrate (the amount of inoculum should be enough to prevent the accumulation of VFA and acid conditions). To determine the background methane production a blank assay with only the inoculum was done in triplicate. The inoculum was degassed for 5 days in order to deplete the residual biodegradable organic matter until the achievement of an endogenous metabolism phase. The test was performed at mesophilic conditions using the same equipment previously presented for BHP tests.

2.3. Co-digestion scenarios, inventory analysis

Two possible layouts of FW and WS co-digestion were compared with the current WWTP (reference scenario). In these two co-digestion scenarios, with reference to Fig. 1, Scenario H_2 (1) and Scenario H_2 (2), was evaluated the possibility to produce hydrogen by adding a new digester for the dark-fermentation to the current plant. For each scenario the mass balance, the energy budget and the greenhouse gas account were estimated. Beside the experimental data that will be presented in the next paragraph, the data reported in Table 2 were assumed in the inventory analysis.

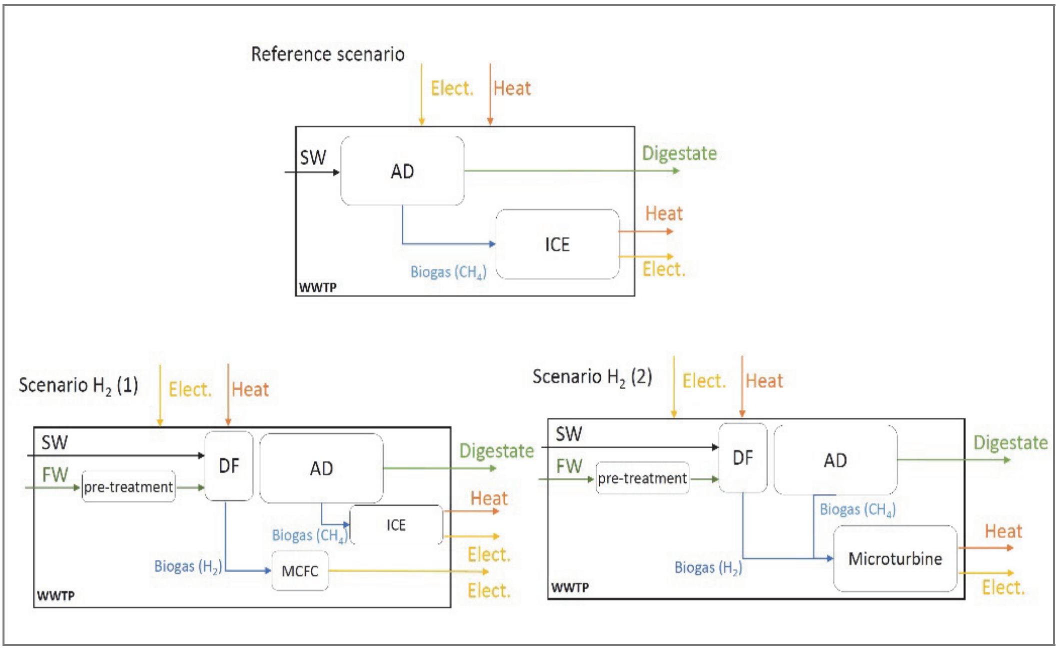

Fig. 1. Co-digestion scenarios layout

Table 2. Mass balance data inventory

	Reference Scenario	***Scenario H_2 (1)***	***Scenario H_2 (2)***
FW in (t/d)	0	54	54
TS (%)	-	28	28
TVS/TS (%)	-	73	73
WS in (t/d)	160	214	214
TS (% w/w)	0.7	0.7	0.7
TVS (% w/dw)	70	70	70
Digester volume	4500	818 (H_2) 4500 (CH_4)	818 (H_2) 4500 (CH_4)
SRT (d)	28	3 (H_2) 17 (CH_4)	3 (H_2) 17 (CH_4)
OLR (kgTVS/m^{3}d)	0.17	14.7 (H_2) 2.3 (CH_4)	14.7 (H_2) 2.3 (CH_4)

Table 3 reports the main inventory data concerning energy flows. In particular, the electricity consumptions of the reference scenario were provided by the owner of the WWTP while, for the co-digestion scenario the use of a screw-press to pre-treat the OFMSW prior to AD was considered. In all the scenarios, thermal energy consumptions were calculated accounting the heat needed to warm the digesters (working at mesophilic conditions) and the heat losses. Concerning the energy production, different choices were done. In the reference scenario it was consider to recover the biogas produced by an ICE. The use of a micro turbine was considered in scenario H_2 (2) in which, beside CH_4, also the H_2 is produced. In scenario H_2 (1) the ICE that recover biogas from AD was integrated by a MCFC for electricity production by the H_2 from DF.

Table 3. Energy data inventory

	Reference Scenario	***Scenario H_2 (1)***	***Scenario H_2(2)***
Electricity consumption (MWh/y)	730	1824	2145
Heat consumption (MWh/y)	1959	3755	3755
Bio-fuel utilization	ICE µel = 0.391 µt = 0.445 Functioning = 7500 h/y	MCFC (H2) µel = 0.45 Functioning = 7000 h/y ICE (CH4) µel = 0.391 µt = 0.445 Functioning = 7500 h/y	Micro turbine µel = 0.33 Exhaust gas flow = 4 kg/s Exhaust gas temp.=280°C Functioning = 8000 h/y

3. Results and discussion

3.1. Hydrogen and methane productions

BMP results for FW were in agreement with previous researches. In particular, the review work of Campuzano and González-Martínez (2016) highlighted an average value for methane production of 415 ± 138 NL CH_4/kgTVS$_{sub}$. as biogas production was found lower than FW due to its lower content of readily biodegradable component such as carbohydrates (Alibardi and Cossu, 2016). Concerning hydrogen production, the average value of 55.0 NL H_2/kgTVSsub is in the range of 25-85 NL H_2/kgTVSsub found by Alibardi and Cossu (2015) for FW mixtures. Table 4 reports BMP and BHP tests outcomes in terms of averages and standard deviations.

Table 4. BMP and BHP tests results. Values are expressed as averages and standard deviations

	FW	***WS***
BMP (NlCH_4/kgTVS$_{sub}$, %CH_4)	440.5 ± 8.7, 65.0 ± 2.3	159.3 ± 11.3, 55.0 ± 1.9
BHP (NlH_2/kgTVS$_{sub}$, %H_2)	55.0 ± 3.6, 45.0 ± 2.4	0.1 ± 0.0, 0.30 ± 0.02

3.2. Co-digestion scenarios performance

Table 5 shows the results of the mass balance, energy budget and greenhouse gas account estimated for each scenarios. In Fig. 2 the scenarios are compared in terms of energy and environmental performance. In order to assess the benefit gained with co-digestion in terms of energy savings, the net primary energy was calculated according to Eq. (2) (Pecorini et al., 2017), where: E_{el} is the net electricity produced in each scenario; Q_{th} is the net thermal energy recovered in each scenario; $\eta_{el,rif}$ is the reference efficiency for electricity, assumed equal to 0.525; p_g is the coefficient of distribution losses, assumed equal to 0.936, $\eta_{th,rif}$ is the reference efficiency for thermal energy, assumed equal to 0.90.

$$Primary\,Energy = \frac{E_{el}}{\eta_{el,rif} \cdot p_g} + \frac{Q_{th}}{\eta_{el,rif} \cdot p_g} \tag{2}$$

Concerning the calculation of CO_2 equivalent saved the conversion factor of 0.551 $kgCO_2/kWh_{el}$.

Table 5. Co-digestion scenarios mass balance, energy budget and GHG emissions comparison

	Reference Scenario	***Scenario H_2 (1)***	***Scenario H_2 (2)***
Biofuel produced			
Biogas (Nm^3/y)	82,000	535,619 (H_2) 2,662,206 (CH_4)	535,619 (H_2) 2,662,206 (CH_4)
CH_4 (Nm^3/y)	53,827	1,730,434	1,730,434
H_2 (Nm^3/y)	-	241,028	241,028
Heat (MWh/y)			
In	1,959	3,755	3,755
Out	219	7,049	6,716
Net	-1,740	3,295	2,962
Electricity (MWh/y)			
In	730	1,824	2,145
Out	193	6,501	5,833
Net	-537	4,677	3,689
GHG emissions (t CO_2eq)			
Produced	163	5,226	5,226
Saved	296	-2,577	-2,032
Net	459	2,649	3,194

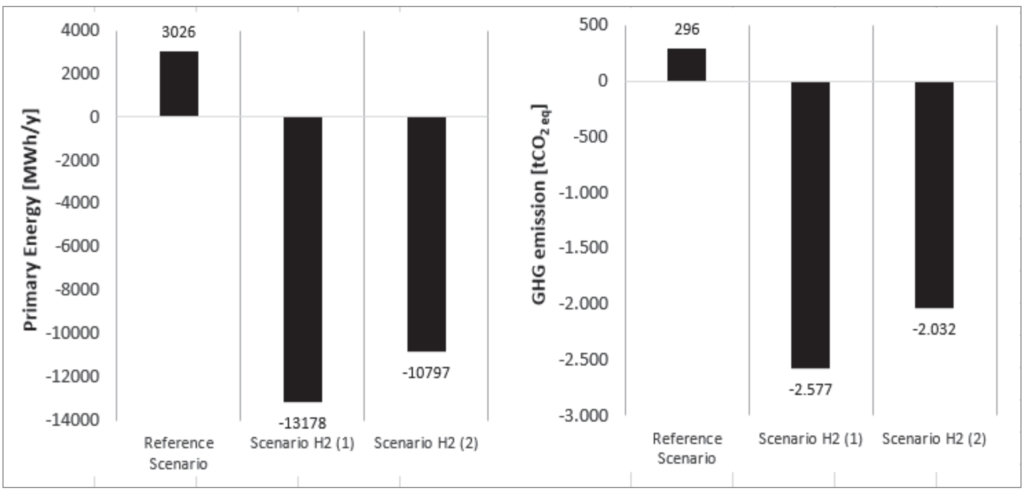

Fig. 2. Comparison of the perfcormance of co-digestion scenarios

4. Conclusions

The results demonstrated that dark-fermentation, performed in a dedicated reactor prior to co-digestion, increases the treatment capacity and the biofuel production (both in terms of hydrogen and methane).

In all the scenarios, the savings achieved by energy recovery from biogas produced were estimated by comparing the use of an ICE, a microturbine and an ICE integrated by a MCFC. The assessment shows that the scenario in which fuel cells and ICE were considered for energy production is the most virtuous in terms of both primary energy saved and avoided emissions of carbon dioxide.

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