



HYDROGEN PRODUCTION FROM FOOD WASTE USING BIOCHEMICAL HYDROGEN POTENTIAL TEST*

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Abstract

The bio-hydrogen production from food waste was evaluated by means of experimental analysis and kinetic model. Biochemical hydrogen potential tests and the application of the modified Gompertz equation were performed. Batch test results showed a production of 48.9 $\text{NIH}_2/\text{kgTVS}_{\text{sub}}$ while the kinetic model well fitted the experimental curve with a correlation coefficient of 0.998. Experimental and model data fell within the range reported by previous researches on bio-hydrogen production from food waste.

Keywords: biochemical hydrogen potential, food waste, Gompertz equation, hydrogen

1. Introduction

The renewed interest in anaerobic digestion (AD) of biodegradable residues has prompted the scientific community to a further development of the process. For instance, bio-hydrogen production during the acidogenic phase of AD is nowadays regarded as a key topic by many researchers due to its potential benefits on both energy and environmental balance (Ghimire et al., 2015, Khan et al., 2016). Hydrogen has gained interest because of its

*Selection and peer-review under responsibility of the ECOMONDO

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eco-friendly nature since it is a carbon-free clean fuel (Kotay and Das, 2008) and because of its versatility as it can be used either in combustion engines or converted to electricity (Alves et al., 2013). Several organic substrates have been tested for biohydrogen production (Ghimire et al., 2015) and food waste (FW) seems to be a valuable feedstock because of its biodegradability characteristics and availability (Cavinato et al., 2011, 2012; Chinellato et al., 2013; Han and Shin, 2004; Micolucci et al., 2014). Indeed, FW is a major fraction of municipal solid waste since it is largely produced in residential areas and its employment in conventional AD is already a well-established technology.

In order to have a rapid, low cost and valuable response of hydrogen production of a substrate, Biochemical Hydrogen Potential (BHP) tests are used in literature (Alibardi and Cossu, 2015; Alibardi and Cossu, 2016; Argun et al., 2008; Cappai et al., 2014; Chinellato et al., 2013; Giordano et al., 2011). BHP tests consist in batch reactors where a certain amount of substrate is incubated with an inoculum under anaerobic fermentative conditions. Batch tests are mostly preferred when time and costs are a constraint due to their simplicity and less time-consuming procedure in comparison with more complex and high-priced continuous reactor experiments. BHP assays evaluate the specific amount of hydrogen that can be potentially produced when a certain substrate or waste is biodegraded under fermentative conditions and it is usually expressed as $\text{NIH}_2/\text{kgTVS}_{\text{added}}$. In particular, BHP tests play a fundamental role as previous experimental tests to assess the potential, adequacy and viability of the dark fermentative treatment of such wastes of interest (Holliger et al., 2016; Wang and Wan, 2009; Zumar Bundhoo et al., 2015; Zumar Bundhoo and Mohee, 2016).

In this study, biohydrogen production from FW was evaluated through BHP assays using two types' set-ups.

2. Materials and methods

2.1. Food waste and inoculum characterization

FW was collected from the Organic Fraction of Municipal Solid Waste (OFMSW). In order to obtain a slurry with a total solid (TS) content suitable to wet fermentation, the sample was treated in a food processor, sift with a strainer (3 mm diameter) and mixed with tap water. Activated sludge (AS) from the aerobic unit of a municipal wastewater treatment plant was used as inoculum (Angeriz-Campoy et al., 2015; Favaro et al., 2013). A first characterization of FW and WS, taking into account TS, TVS and pH results is presented in Table 1.

Table 1. Food waste and inoculum characterization
(pH, TS and TVS/TS are expressed by mean and standard deviation)

<i>Material</i>	<i>TS (%)</i>	<i>TVS/TS (%)</i>	<i>pH</i>
FW	5.6 ± 0.1	91.6 ± 0.3	3.81 ± 0.01
AS	1.5 ± 0.1	78.6 ± 0.3	7.08 ± 0.01

2.2 Analytical parameters

FW and AS were studied through physico-chemical, bromatological and methane potential analysis. TS, TVS and pH were determined in order to characterize inoculums and FW according to standard methods (APHA, 2006). Due to the acidic condition of each substrate, TS determination was performed at 90°C instead of 105°C until constant weight in order to avoid the volatilization of VFA.

Proteins, lipids, cellulose, Total Kjeldahl Nitrogen (TKN) contents were measured in accordance with the European Commission Regulation 152 (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 were measured using EPA 6010 D (2014) and EN 13657 (2004). 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 Decreto Ministeriale 196 (1989). Volatile Fatty Acids (VFAs, including acetic, propionic, butyric, iso-butyric, valeric and iso-valeric acids) were measured according to MP 0224 (2012) while total alkalinity was obtained through MP 1635 (2013). FW was also characterized in terms of methane production by means of BMP tests following the procedure of Pecorini et al. (2016).

2.3 Experimental set-up

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 placed on a magnetic stirred and incubated in a water bath at 37°C for 2 days. The ratio between the volatile solids of the substrate to be degraded and volatile solids of the inoculum biomass (Food/Microorganism, F/M) was 0.5 gTVS/gTVS. 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 the initial pH at 5.5. After set-up, the bottles were flushed with N₂ for few minutes to ensure anaerobic conditions in the headspace of the batches. The inoculums were 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; Jung et al., 2011; Li and Fang, 2007).

Biogas production was periodically 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):

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

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).

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 chromatography (3000 Micro GC, INFICON, Switzerland).

2.4 Kinetic model

The mean cumulative hydrogen production curves were obtained over the course of the batch experiment and analysed using the modified Gompertz equation (Van Ginkel et al., 2005). Eq. (2) is used in many works to describe the kinetic of hydrogen production from

batch fermentation assays (Pan et al., 2008).

$$H(t) = H_{max} \exp \left\{ - \exp \left[\frac{R \cdot e}{H_{max}} (\lambda - t) \right] + 1 \right\} \quad (2)$$

where: $H(t)$ - hydrogen production at a time t (NL H₂/kgTVSsub); H_{max} - total amount of hydrogen produced (NL H₂/kgTVSsub); R - maximum hydrogen production rate (NL H₂/kgTVSsub h); λ - length of the lag phase (h).

The time needed to attain 95% of the maximum hydrogen yield (t_{95}), was obtained from the Gompertz equation as follows (Cappai et al., 2014) (Eq. 3):

$$t_{95} = \frac{H_{max}}{R \cdot e} (1 - \ln(-\ln 0.95)) + \lambda \quad (3)$$

Constants were estimated by minimizing the sum square of errors between the experimental data and results of the model. The estimations were carried out by using the solver function of Microsoft Excel version 2016.

3. Results and discussion

3.1. Analytical characterization of FW and inoculums

Table 2 presents the measured data of chemical, bromatological and methane potential analysis. Butyric, iso-butyric valeric and iso-valeric acids contents were not shown since they were found below the limit of detection (LOD = 40 mg/L). Acetic acid was the prevalent VFA for both AS and FW.

Table 2. Chemical, bromatological and methane potential data expressed by mean and standard deviation

<i>Parameter</i>	<i>AS</i>	<i>FW</i>
TOC (%C w/w)	1.2 ± 0.2	1.9 ± 0.2
TKN (%N w/w)	0.2 ± 0.0	0.2 ± 0.0
Ammonia (mgN/kg)	341 ± 47	191 ± 5
Acetic acid (mg/L)	830 ± 120	958 ± 30
Propionic acid (mg/L)	390 ± 71	< 40
C (%TS)	58.9 ± 4.3	36.0 ± 1.9
H (%TS)	6.4 ± 0.5	5.8 ± 0.2
N (%TS)	7.5 ± 0.9	2.9 ± 0.3
S (%TS)	0.9 ± 0.1	0.2 ± 0.0
P (%TS)	0.4 ± 0.1	0.4 ± 0.1
O (%TS)	27.9	54.6
Proteins (% w/w)	0.9 ± 0.1	1.0 ± 0.1
Lipids (% w/w)	< 0.3	0.5 ± 0.0
Carbohydrates (% w/w)	0.0	2.4
Cellulose (% w/w)	0.1 ± 0.0	0.8 ± 0.1
Lignin (% w/w)	0.3 ± 0.0	0.3 ± 0.1
BMP (NICH ₄ /kgTVSsub)	-	511.6 ± 38.2

With regard to the C:N ratio, FW showed a value of 12.4, slightly below other FW findings: Zhang et al. (2007) reported an average value of 14.8 while Pan et al. 2008 and Han

and Shin 2004 obtained C:N ratios of 17.1 and 14.7 respectively. The C:N result found for AS (7.9) is concurring with previous researches and it is explained by the high N content and the high ammonia concentration (Table 2). In general the C:N ratio of sludge ranges between 6-9 (Iacovidou et al., 2012). C:N ratios lower than 6 negatively affect the digestion process mostly due to the low carbon availability in combination with high ammonia concentration that can cause toxicity to anaerobic bacteria (Iacovidou et al., 2012; Salerno et al., 2006).

The methane yield obtained for FW (511.6 NL CH₄/kgTVS_{sub}) was higher than values reported by Zhang et al. (2007), who obtained 435 NL CH₄/kgTVS at 50°C and 28 days and Heo et al. (2004) who obtained 489 NL CH₄/kgTVS at 35°C and 40 days.

Among the macromolecules, carbohydrates were the main component for FW while AS highlighted a predominance of proteins (Wilson and Novak, 2008). FW proteins and carbohydrates were found slightly below previous works probably due to the dilution employed in the present study (Table 3).

Table 3. Comparison of proteins and carbohydrates results of FW with previous studies

<i>Reference</i>	<i>Substrate</i>	<i>Proteins (g/kg)</i>	<i>Carbohydrates (g/kg)</i>
Present study	FW	10	24
Chu et al., 2008	FW	41-49	60-72
Lee et al., 2010	FW	1	35
Yeshanew et al., 2016	FW	31	134

3.2. BHP tests

Fig. 1 and Table 4 present the cumulative hydrogen production over time and the kinetic parameters calculated using Gompertz Equation. Hydrogen production was observed until 47 h. After this period, the cumulative curve highlighted a decreasing trend owing to biological hydrogen consumption (De Gioannis et al., 2013, 2017). The inoculum heat pre-treatment prior to the DF process was effective since methane content in biogas was detected null along all the duration of the tests (Zumar Bundhoo et al., 2015; Zumar Bundhoo and Mohee, 2016). As such, hydrogen consumption is probably attributable to propionic fermentation (Dong et al., 2010) or homoacetogenesis (De Gioannis et al., 2017; Saady, 2013). The final production of 48.9 ± 4.3 NL H₂/kgTVS_{sub} fell within the range reported by previous works for FW. Alibardi and Cossu (2015) determined final results in the range of 25-85 NL H₂/kgTVS_{sub}, while Pecorini et al. (2017) and De Gioannis et al. (2017), reported hydrogen productions of 55.0 and 56.5 NL H₂/kgTVS_{sub} respectively.

Concerning the kinetic, the Gompertz model fitted well the experimental data with a high correlation coefficient (0.998). The kinetic parameters fell in the range of previous works (Table 4). The lag phase lasted few hours (3.4 h) while the time needed to attain 95% of the maximum hydrogen yield (*t*₉₅) was reached after approximately one day (29.3 h) (Fig. 3).

Table 4. Experimental and model results

<i>Reference</i>	<i>BHP (NL H₂/kgTVS_{sub})</i>	<i>R (NL H₂/kgTVS_{sub}h)</i>	<i>λ (h)</i>	<i>t₉₅ (h)</i>	<i>R²</i>
Present study	48.9 ± 4.3	2.8	3.4	29.3	0.998
Cappai et al., 2014	77.5	7.2	6.2	22.1	-
Cappai et al., 2014	56.7	7.8	13.3	23.9	-
Cappai et al., 2014	117.6	16.6	3.9	14.3	-
De Gioannis et al., 2017	56.5	3.8	4.1	26.4	0.988
Pan et al., 2008	39	-	4.4	-	0.988

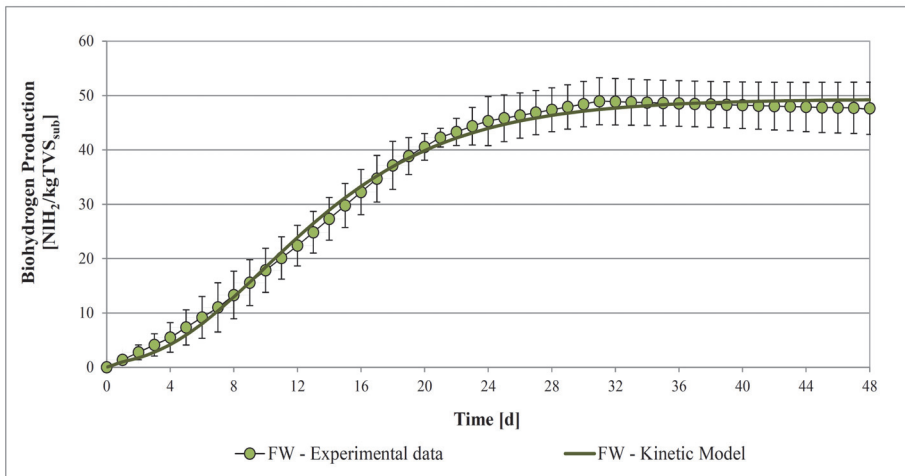


Fig. 1. Hydrogen production over time. Solid lines indicate Gompertz model curves. Y-error bars represents standard deviation.

4. Conclusions

The bio-hydrogen production from food waste was evaluated by means of experimental analysis and kinetic model. Biochemical hydrogen potential tests and the application of the modified Gompertz equation were performed. Batch test results showed a production of 48.9 $\text{NIH}_2/\text{kgTVS}_{\text{sub}}$ while the kinetic model well fitted the experimental curve with a correlation coefficient of 0.998. Experimental and model data fell within the range reported by previous researches on bio-hydrogen production from food waste.

Acknowledgements

This project has been funded with support from the MIUR-MISE-Regione Toscana DGRT 758/2013 PAR FAS 2007-2013. The authors want to thank the support of SEA Risorse and Alia S.p.A. for providing the waste samples.

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