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Biotic and abiotic insights into the storage of food waste and its effect on biohydrogen and methane production potential

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ABSTRACT

The present study investigated the physicochemical and microbiological changes occurring during the storage of simulated restaurant food waste (FW) and how such changes affected its biohydrogen and biogas production potential. FW was stored for 72 h in a closed atmosphere under two different scenarios: i) without and ii) with inoculation of a mixed microbial culture harboring lactic acid bacteria (LAB). Both storage scenarios resulted in similar biotic and abiotic changes in FW. Particularly, FW was pre-acidified and pre-hydrolyzed to some extent during the storage, resulting in a feedstock enriched in LAB (≈ 95 % total relative abundance) and lactate (10.5–12.3 g/L, 87.0–90.5 % selectivity). Biochemical hydrogen potential tests revealed that the use of stored FW resulted in similar or even higher hydrogen production efficiencies compared to that of non-stored FW, achieving up to 60 NmL H_2/g VS added and a maximum volumetric hydrogen production rate of 9.7 NL H_2/L-d. Metabolically, the conversion of lactate into hydrogen was crucial regardless of the use of non-stored or stored FW, albeit the presence of fermentable carbohydrates in the substrate was also essential either to produce lactate or to co-produce extra hydrogen. On the contrary, biochemical methane potential tests showed that the biogas production potential of FW was not affected by storage, yielding on average 400 NmL CH_4/g VS added and revealing that lactate oxidation to methane precursors represented an important step in FW biomethanization.

1. Introduction

The valorization of organic waste into renewable bioenergy such as biohydrogen and biogas has been adopted as a global strategy to solve food waste (FW) and fossil energy related problems. Circular FW management has taken on an important role as a sustainable model to bring economic and environmental welfare [1]. The most recent FW index report stated that the amount of FW generated worldwide in 2019 amounted to 931 million tonnes, of which 61 % was derived from households, 26 % from food service, and 13 % from retail [2]. In Europe, that figure accounted for 88 million tonnes [3], while about 168, 127, 232, and 465 million tonnes were estimated for the North America, Latin America and the Caribbean, sub-Saharan Africa, and Asia-Pacific regions, respectively [4–7]. Regardless of its geographical origin, source and season of collection, FW is an effective source of macro- and micronutrients needed to support biological processes [8]. Thus, due to its huge availability, inexpensive nature, and high nutrients (e.g., 36 ± 21 % carbohydrates, 21 ± 13 % proteins, and 15 ± 8 % w/w lipids) and moisture content (e.g., 22.8 ± 10 % w/w dry matter), FW is a potential feedstock to produce several biobased products such as biochemicals, biomaterials and biofuels [8,9].

Dark fermentation (DF) is a promising biotechnology for the development of FW-based biorefineries, which allows to transform such a feedstock into a clean and renewable fuel (i.e., biohydrogen), and high-value organic acids such as butyrate, acetate, lactate, etc. These acids can be employed as building blocks for producing other marketable products, such as bioplastics to mention an example [10]. Meanwhile, anaerobic digestion (AD) is a mature biotechnology able to treat various types of organic wastes and wastewaters, while recovering renewable bioenergy in the form of biogas and producing a nutrient-rich digestate, which has the potential to be used as a soil biofertilizer [11,12]. Both DF and AD are well aligned with the goals of circular economy. Additionally, such biotechnologies are regarded as sustainable FW alternatives that can help divert FW from landfill and incineration, which are

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nowadays the most applied but least sustainable and environmentally friendly disposal methods for FW [13].

While low and unstable hydrogen production yields and rates are the main bottlenecks in the DF of FW [14], the major limitations that constrain the deployment of the AD of FW are the very long retention times and process instability due to the accumulation of volatile fatty acids (VFAs) caused by the high complexity and low buffer capacity of FW [15]. Both DF and AD are sensitive to changes in substrate composition and operational process parameters. It is worth mentioning that FW is commonly subjected to storage prior treatment, which may last from few hours up to several days [16–18]. Such a storage step also occurs in decentralized FW management systems for supermarkets and restaurants [19,20]. Thus, the temporal storage of FW might influence not only its physical-chemical properties but also its microbial composition [21–24], which in turn might affect its valorization via DF and AD. For instance, stored FW can be pre-hydrolyzed/acidified to different extents depending on the storage conditions and FW composition, commonly leading to lactic fermentation [17,24] but the accumulation of fermentation products such as acetate, propionate, butyrate and ethanol may also exist [19,24,25].

Organic acids and solvents could have a negative effect on DF depending on their type and concentration [26,27]. Besides, it is well-known that the type and concentration of intermediate acidogenic products influence the AD performance [13,28–30]. Hence, it is of paramount importance to understand in detail how FW characteristics would change with storage conditions and how such latent changes would influence the yields and kinetics of biohydrogen and biogas production, in order to achieve an enhanced FW-to-bioenergy conversion process. In this context, great efforts have been made to investigate the storage of FW and its impact on bioenergy recovery [16,17–19,21,23,24,31–33]. It can be inferred from those studies that the maximum achievable bioenergy production from a given type of FW will greatly depend on the biotic and abiotic changes that the feedstock experiences from the time it is generated until its final valorization. Such changes are in turn dependent not only on the features of FW but also on the storage time and conditions such as temperature and atmosphere. Hence, it could be expected that FW storage has the largest impact on its subsequent transformation through DF and AD, but correlation of transformation efficiency with storage conditions and microbiological/physical-chemical changes in FW is yet limited.

Im and co-workers evaluated the effect of storage time on the performance of DF of FW [23]. The study assessed the effect of storage time and temperature on hydrogen production from cafeteria FW. It was found that FW stored for 1–2 days at 20 and 35 °C decreased hydrogen yields by 20–30 % (compared to DF of fresh FW) due to the enhanced growth of indigenous lactic acid bacteria (LAB) such as *Lactobacillus* and *Weissella* [23]. Here it should be noted that hydrogen can be produced directly from carbohydrates but also from lactate commonly hand-in-hand with acetate [34], which renders difficult to draw a general conclusion on the effect of FW characteristics on the performance of the DF of FW, particularly using stored FW rich in lactate. Evidence available to understand how FW storage might affect lactate-driven fermentative hydrogen production (herein referred to as LD-DF) is quite limited [20,24]. One of the main open research questions that the present study aims to address is the following: *Is LD-DF able to deal with a FW enriched in lactate and LAB?* Hence, the present study investigated the physical-chemical and microbiological changes occurring during the storage of FW under a closed atmosphere and the impact of such a storage on biohydrogen and biogas production potential. Two different storage methods were investigated: FW storage with the addition of an inoculum containing LAB, and FW storage driven by indigenous microflora. Biohydrogen production tests with fresh and stored FW were performed using an inoculant of hydrogenogenic consortium able to perform LD-DF, whereas methane production was investigated through biochemical methane potential (BMP) assays using an anaerobic sludge.

2. Materials and methods

2.1. Substrate

Simulated FW was used as the substrate in order to ensure substrate homogeneity and allow for comparison among the experiments. The synthetic FW mimicking restaurant FW was prepared using (on wet basis) 78 % potato, 14 % chicken, 4 % lard, and 4 % cabbage [35]. Potato and chicken were previously heated in autoclave at 120 °C for 30 min to mimic cooked waste. Subsequently, the mixture was grinded with the aid of an industrial kitchen blender to generate a homogeneous substrate with a low particle size, providing a larger surface area for microorganisms to degrade organic matter. No additional water was added during the grinding step. The substrate was stored in resealable plastic bags at −20 °C to inhibit microbial degradation and prevent any change in its composition before use. The proximate and ultimate composition of the grinded FW is shown in Table 1.

2.2. Inocula

A mixed hydrolytic-acidogenic culture enriched in LAB and hydrogen-producing bacteria (HPB) was used in both the storage experiment and the batch DF tests [13]. It was originally obtained from an anaerobic digester treating restaurant FW at mesophilic conditions (35 °C) under an organic loading rate of 0.86 g VS/L-d and a hydraulic retention time (HRT) of 60 days [36]. The digestate, which was collected under pseudo-steady state, was pretreated by heat-shock at 90 °C for 20 min to inhibit its methanogenic activity fully and irreversibly [13]. In order to enrich LAB and HPB, the pretreated inoculum was subjected to successive culture passages, following the procedure described previously by García-Depraect et al. [37]. Likely, the methanogenic inoculum used for the BMP tests was obtained from the anaerobic mixed sludge digester of the municipal wastewater treatment plant of Valladolid, Spain. Prior to use, it was preincubated for 7 days at 37 °C under anoxic conditions in the absence of substrate. The resulting anaerobic sludge exhibited a pH of 7.5 ± 0.1 and total solids (TS) and volatile solids (VS) contents of 23.9 ± 0.06 g/L and 12.6 ± 0.04 g/L, respectively.

2.3. FW storage

FW was stored in 2.1-L borosilicate glass roller bottles with screw caps. The amount of FW was 0.5 kg (wet weight) per container. The bottles were closed with rubber septa and aluminum caps, and then incubated in a roller shaker (Wheaton Scientific Products, USA) with a constant gently rotation (4.5 rpm). Gas-tight bottles were intentionally used to ensure natural anoxic conditions during storage [15]. The headspace was initially composed of air (i.e., ≈21 % O_2, ≈79 N_2), while the storage temperature was set at 36 ± 1 °C (by using a thermostatically controlled chamber) to promote changes in the composition of waste over time [24]. FW was stored for 18, 30, 48 and 72 h without inoculum addition, which are common storage times in FW management

Table 1
Characteristics of the synthetic FW.

Feature	Mean value
Total solids	21.9 g/kg
Volatile solids	21.0 g/kg
Carbohydrates	47.0 %[a]
Proteins	33.0 %[a]
Lipids	17.2 %[a]
Ash	4.9 %[a]
C	50.3 %[a]
H	7.7 %[a]
O	37.1 %[a]
N	4.7 %[a]

[a] Percentage (w/w) on dry matter basis.

[22,23,32]. A total number of 15 bottles was used to assess each and every storage time in triplicate. Accordingly, three random bottles were taken for each pre-determined storage time. Additionally, the effect of adding an inoculum containing hydrolytic/acidogenic bacteria (including LAB) on the storage of FW was also investigated (in triplicate) under identical storage conditions for the sake of comparison (see Section 2.1). The storage time for FW with inoculum addition was set at 72 h. The inoculum size was 5 % (v/v) with a volatile suspended solids (VSS) content of 2.2 g/L. For all experiments, stored FW was immediately frozen at −20 °C to be further characterized for pH, solids, total carbohydrates, soluble chemical oxygen demand (COD), and organic acids. The composition of the headspace was also determined after recording and releasing overpressure caused by the production of gas during storage. FW stored for 72 h with and without inoculum addition was used in the biohydrogen and biogas potential tests.

2.4. Biohydrogen potential tests

DF of FW was carried out in triplicate in three bench-scale gas-tight polypropylene-made reactors, each with a total volume of 1.4 L. The reactors had two individual ports for collecting gaseous and liquid samples, a pH control unit (EVopHP5, BSV Electronic, Spain), a magnetic stirrer set at ≈300 rpm, and a custom-made gas flow meter based on the liquid displacement method. The DF assays were carried out at constant temperature (37 °C) with a water jacket. The setpoint for the automatic pH control was set at 6.5 using a 6 N NaOH solution [35]. The fermentations were carried out at a TS concentration of 50 g/L [38]. Thus, the amount of substrate added depended on its TS content, while the inoculum size comprised 10 % v/v of the working volume. The hydrogenogenic inoculum had a VSS concentration of 0.5 g/L. Tap water was added to substrate and inoculum to achieve the reactor working volume of 800 mL. Three types of substrates were evaluated in the DF tests: fresh FW (0_S), FW with 72 h of storage without inoculum (72_S), and FW stored for 72 h with LAB-HPB inoculum (72_{SI}). All the DF experiments had a duration of 30 h, which was selected based on the observed plateau in the cumulative hydrogen production.

During the fermentations, the cumulative volume and composition of the produced acidogenic-off gas were recorded periodically. The volume of produced hydrogen was normalized to NmL by considering standard temperature and pressure conditions (0 °C and 1 atm). Liquid samples of the culture broth were also collected throughout the fermentation for further analysis. The performance of the LD-DF process was assessed based on the hydrogen production yield (YH_2, NmL H_2/g VS added), maximum volumetric hydrogen production rate (VHPR, NmL H_2/L-h), peak H_2 content in the acidogenic-off gas (% v/v), lag phase (λ, h), ultimate hydrogen production potential (P_{max}, NmL H_2), maximum hydrogen production rate (R_{max}, NmL H_2/h), total carbohydrates removal (%), and organic acids spectrum. Hydrogen production kinetics parameters, i.e., λ, P_{max}, and R_{max}, were estimated using the modified Gompertz model (Eq. (1); where H is the cumulative hydrogen/methane production (NmL)) [39]. The root mean square error (RMSE) was used as the objective function, while the coefficient of determination (R^2) and error percentage were used to score the prediction. VHPR was calculated by dividing R_{max} by the reactor working volume. Finally, the degree of acidification was computed as the relation between the net concentration (final – initial) of acidogenic products (i.e., VFAs, lactate, ethanol) expressed as COD equivalent and the total initial COD concentration of the substrate [40].

$$H = Pmax \bullet exp\left\{ -exp\left[\frac{Rmax \bullet e}{Pmax}(\lambda - t) + 1\right]\right\} \quad (1)$$

2.5. BMP tests

BMP tests were carried out to evaluate the effect of FW storage on the yields and kinetics of biogas production. The same types of FW used in the DF tests, i.e., 0_S, 72_S, and 72_{SI}, were also tested in the BMP assays in triplicate. Additionally, a blank test with only inoculum and a positive test with microcrystalline cellulose as the sole carbon source were performed. The BMP tests were carried out in 120 mL gas-tight serum bottles with a working volume of 50 mL. The F/M (food-to-microorganisms) ratio was kept at 0.25 on a VS basis [35]. The methanogenic sludge was previously supplemented with 5 g $NaHCO_3$/L to ensure buffer capacity. Once the substrate and inoculum were added, the bottles were sealed with butyl rubber stoppers. Subsequently, the residual oxygen in the headspace was removed by purging with helium gas (Abello Linde, Barcelona, Spain) at approximately 0.6 bar for 1 min. Following headspace flushing, the serum bottles were placed in an orbital shaker (120 rpm) for incubation at 36 ± 1 °C. After 1 h of incubation, the gas pressure in the headspace was quantified manometrically (IFM electronic PN7097, Germany) and the excess gas was vented to reach ambient atmospheric pressure. The pressure in the headspace and biogas composition were measured daily until the end of the experiment, which was ceased when the net daily biogas production recorded for three consecutive days was lower than 1 % of the total net cumulative biogas production. The data recorded was used to calculate the cumulative methane production per gram of VS added (NmL CH_4/g VS added), as previously described elsewhere [41]. Methane production kinetics parameters, i.e., λ (lag phase), P_{max} (methane production potential), and R_{max} (maximum methane production rate), were also estimated using the modified Gompertz model (Eq. (1)), which showed a better description of the experimental data than a first-order kinetic model (data not shown). The methane yield obtained experimentally was thus compared to that theoretically achievable from the FW used, which was estimated based on Buswell's eq. [42]. At the end of the BMP tests, digestate samples were collected and analyzed for pH and VFAs.

2.6. Analytical methods

Total carbohydrates, total Kjeldahl nitrogen (TKN), lipids, pH, COD, and solids were determined according to Martínez-Mendoza et al. [38]. Soluble COD was determined in filtered samples (0.45 μm). Biogas composition was analyzed by gas chromatography coupled with a thermal conductivity detector (GC-TCD) according to García-Depraect et al. [43]. Headspace pressure was measured by using a pressure transducer (IFM electronic PN7097, Germany). Due to their expected low concentration (in the order of few mg/L), the final concentration of VFAs in the digestate was measured by gas-chromatography coupled with flame-ionization-detection (GC-FID), following the procedure previously reported elsewhere [44]. Likewise, the quantitative analysis of organic acids (including lactate) and ethanol during DF was performed by high-performance liquid chromatography (HPLC), according to Martínez-Mendoza et al. [38]. Sample preparation for both GC-FID and HPLC analyses included centrifugation at 10000 rpm for 10 min, followed by filtration through a 0.22 μm nylon syringe filter and acidification using concentrated sulfuric acid.

The microbial community analysis was carried out in frozen/thawed FW and in composite samples of triplicates of FW stored for 72 h without (FW_S) and with (FW_{SI}) inoculum addition. DNA was isolated with the QIAsymphony PowerFecal Pro DNA Kit (Qiagen, Germany) in accordance with the manufacturer's instructions. The V3-V4 region of the 16S rRNA gene was thus amplified using the bacterial universal primer set 341F-805R and amplicon libraries (300 bp paired-end reads) were prepared by ADM-Biopolis (Valencia, Spain) following the 16S Metagenomic Sequencing Library Illumina 15,044,223 B protocol (Illumina 1.9) [45]. Raw sequences were merged and trimmed using the BBMerge package of BBMap V.38 software with "Cutadapt v 1.8.1" using default parameters. Quality checked (Q20 threshold) reads were afterward processed using the DADA2 denoise-single command [46]. Error rates were learned from a set of subsampled reads using "learnErrors" and sample inference algorithm was applied with the "dada" function. The chimeric amplicon sequence variants (ASVs) were removed using

"removeChimeraDenovo". Those clean ASVs were annotated against NCBI 16S rRNA database version 2022 using blast version 2.2.29 at 97 % similarity threshold [47]. The taxonomy of the ASVs with a lower percentage identity than 97 % was reassigned using NBAYES algorithm [48]. NBAYES classifier was trained on V3-V4 of 16S rRNA gene from SILVA v.138 database [49]. Finally, data was normalized using the rarefaction technique from Phyloseq R package to perform alpha diversity analysis [50].

2.7. Data analysis

Data from the storage and bioenergy potential tests were collected and further processed in the Excel software, using the 'Solver' function to perform kinetic estimations. Data reported in Figures and Tables is the mean and standard deviation values of triplicate measurements.

3. Results and discussion

3.1. Physical-chemical and biotic changes occurred during FW storage

Prior to DF or AD tests, FW was stored at 36 ± 1 °C for 72 h with and without addition of an inoculum harboring LAB among other hydrolytic-acidogenic bacteria. As shown in Fig. 1, the pH of the FW stored without inoculum sharply dropped from 6.2 to 4.5 after 18 h of storage and then slightly decreased down to 4.2 at the end of the test. The time-course of the pH for the inoculum-supplemented FW during storage was not monitored but decreased from 6.2 for the fresh FW to 4.3 by the end of the storage. Such a rapid decrease in pH, regardless of the storage conditions, was attributed to the accumulation of organic acids. Indeed, the total concentration of organic acids in the FW stored without inoculum increased within the first 18 h from 1.9 to 10.8 g COD equiv./L (Fig. 2). After 72 h of storage, the total concentration of organic acids was 12.9 ± 0.6 and 14.5 ± 0.3 g COD equiv./L for stored FW with and without inoculum, respectively, which corresponded to an acidification degree of ≈3 % for both storage conditions. Regardless of the storage conditions, lactate was the major organic acid produced throughout FW storage. The average titer of lactate and its corresponding selectivity at the end of storage, were 10.5 and 12.3 g/L, and 87.0 and 90.5 %, for FW storage with and without inoculum, respectively. Under both assessed FW storage conditions, acetate was the second most dominant organic acid with titers ranging from 1.1 to 1.3 g/L. In this context, previous studies in literature have shown a similar dominance of a primary lactate-type fermentation corresponding well with the tendency of the pH of FW to decrease over storage time [9,16,17,21,22,24].

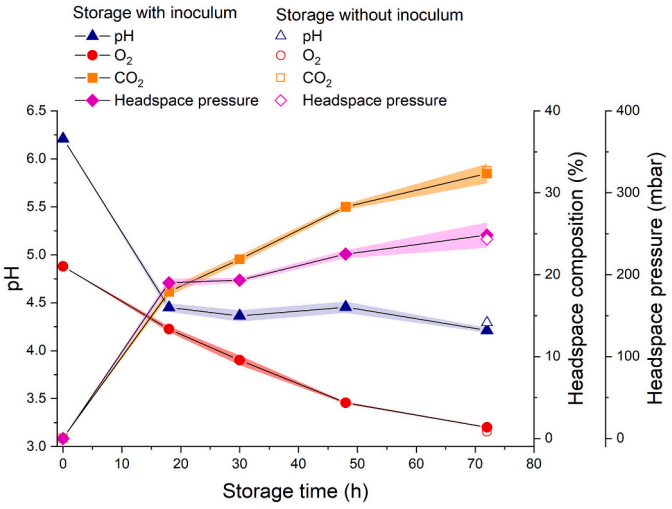

Fig. 1. Time evolution of pH and headspace gas pressure/composition during FW storage with and without addition of inoculum. Data from the inoculum-supplemented FW were only recorded at the beginning and at the end of storage. The markers are the mean and standard deviation (error lines are indicated with colored shade areas) values of triplicate experiments.

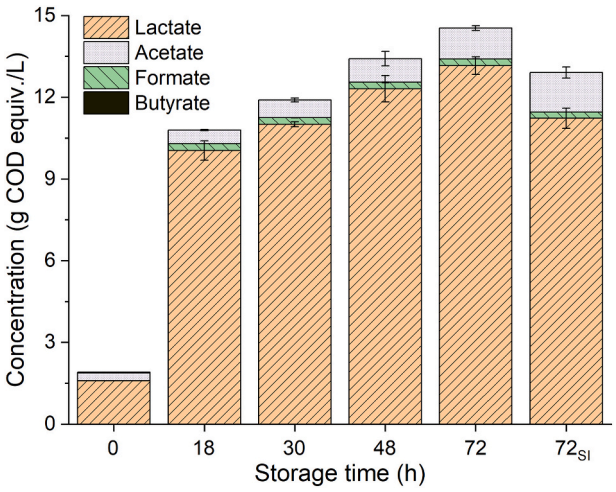

Fig. 2. Type and concentration of organic acids measured during the storage of FW under closed atmosphere without inoculum addition. The code "72_{SI}" stands for FW stored for 72 h with inoculation of a LAB-enriched bacterial mixed culture.

Furthermore, it has been reported that the type of atmosphere during the storage can influence the ultimate features of FW and therefore its energy recovery potential, FW stored anoxically exhibiting higher energy content than that stored under aerobic conditions [18,33]. It is expected that high-scale FW storage leads to lower levels of aerobic degradation [21]. In the present study, the O_2 content in the headspace decreased due to the activity of facultative anaerobes from ≈21 to 0.8 and 1.3 % (v/v) during the 72 h of FW storage with and without inoculum addition, respectively. In contrast, the initial headspace presented a CO_2 content of 0.04 %, which increased up to 32 % by the end of the 72-h storage period regardless of the condition tested. The headspace pressure recorded in the gas-tight bottles without inoculum gradually increased as the storage time progressed. For both storage conditions, the average gauge headspace pressure was recorded at 240 mbar following the 72 h of storage (Fig. 1). Interestingly, inoculation during FW storage did not cause a difference in the final carbohydrate removal efficiency, which accounted for 9.5 ± 4.0 % and 9.5 ± 5.9 % for storage with and without inoculum, respectively, reflecting that during storage only a small fraction of carbohydrates was assimilated by microorganisms.

Regarding microbial taxonomy, as shown in Fig. 3, the microbiota of non-stored FW was found to be more diverse than that of stored FW regardless of the type of storage tested. At a genus level, Richness, Shannon and Simpson indexes of 126, 2.76 and 0.87, 17, 1.51 and 0.74, and 21, 1.64 and 0.76 were estimated for 0_S, 72_S and 72_{SI}, respectively. The microbial communities of frozen/thawed FW included *Fastidiosipila* (19 %), *Gallicola* (8 %), *Gleimia* (6 %), *Lactobacillus* (6 %), among others. Some of the satellite bacteria detected in the non-stored FW were, for instance, *Leuconostoc*, *Alkaliphilus*, *Anaerococcus*, *Acholeplasma*, *Caldicoprobacter*, *Thermovirga* or *Caldicoprobacter*. On the other hand, bacteria belonging to the LAB group comprised about 95 % of the total relative abundances of the ASVs detected at the end of storage, regardless of the FW storage condition, indicating that storage strongly shaped microbial communities (Fig. 3). The dominance of LAB explains the recorded fermentation product spectra and suggests that inoculation had little to no influence on shaping the structure of microbial community during the storage. More specifically, the dominant genera were *Lactococcus*, *Latilactobacillus*, *Enterococcus* and *Leuconostoc*, with corresponding relative abundances of 37.2 and 33.9 %, 21.9 and 22.1 %, 21.2 and 18.8

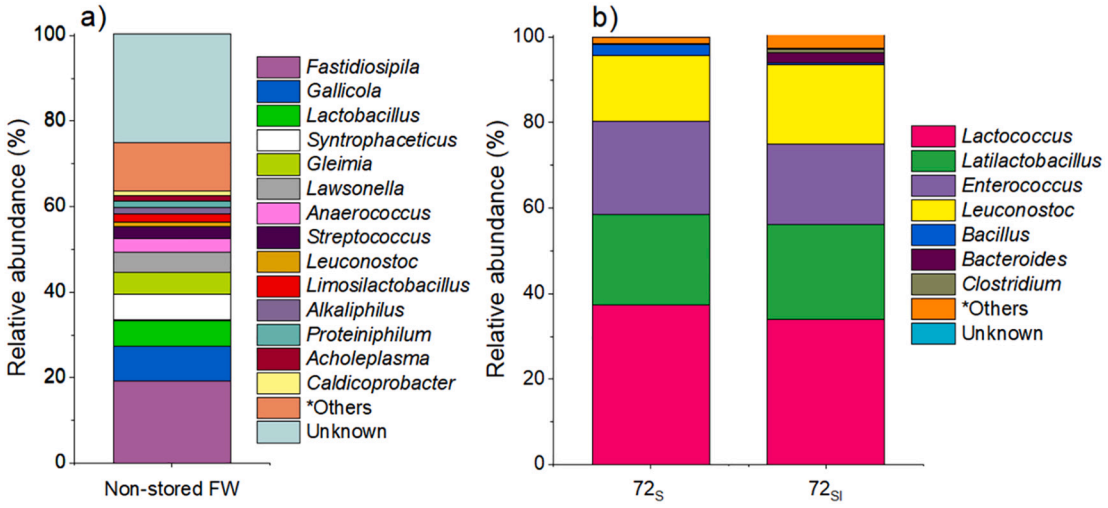

Fig. 3. Microbial community structure at genus level for a) non-stored FW and b) FW stored after 72 h under a closed atmosphere a) without (72_S) and b) with (72_{SI}) inoculation of a bacterial mixed culture enriched in LAB. *The term "Others" includes genera with a relative abundance lower than 1 %.

%, and 15.4 and 18.5 % for 72_S and 72_{SI}, respectively (Fig. 3). In the literature, LAB (the lactate producers par excellence) have been found to be the most dominant bacterial group in the spontaneous fermentation that may take place either during the storage [22,24] or anaerobic fermentation [13] of FW [22]. Bacteria belonging to *Bacillus*, *Bacteroides* and *Clostridium* genera were also detected in the FW under both storage scenarios investigated, but at minor proportions (Fig. 3). Despite presence of these three bacterial genera, secondary lactate fermentation did not take place, likely due to the too low pH imposed during storage [51,52].

The final VS removal efficiencies computed for the FW stored with and without inoculum were 4 ± 2 % and 7 ± 2 %, respectively. Such low losses in VS seem reasonable given the short storage time of 72 h tested herein. In addition, the soluble COD concentration increased from 90.7 ± 5.3 to 109.1 ± 3.9 after 72 h of storage in the assays without inoculum, which was found to be comparable to that of the inoculum-supplemented condition (112.7 ± 5.6 g/L). Overall, it can be pointed out, based on such limited increase in the concentration of soluble COD recorded and the observed productions of organic acids, that FW was pre-hydrolyzed and pre-acidified to some extent during the storage, which is in agreement with previous reports [21,25]. However, in the present study, the addition of inoculum to FW in the storage stage did not cause a strong effect in both the biotic and the abiotic features studied, as compared to those observations made for FW stored without inoculum. The following two aspects can be therefore highlighted: i) FW itself can be not only a good source of LAB but also of the nutrients needed for their thriving, and ii) the biotic and abiotic changes occurring during the storage of FW may converge to a LAB-dominated microbial community along with a lactate-dominated product spectrum, and it seems that such a trend can be boosted by an acidic pH and self-generated anaerobic conditions.

3.2. Effect of FW storage on biohydrogen production via LD-DF

The trends observed in the experimental cumulative hydrogen production and hydrogen production rates are depicted in Fig. 4. The use of fresh FW (0_S) resulted in a slightly lower production of hydrogen of 1960.1 ± 50.4 NmL, which corresponded to a YH_2 of 51.0 ± 5.9 NmL H_2/g VS added. In contrast, the cumulative hydrogen production achieved after 30 h of fermentation was 2320.6 ± 325.5 NmL (60.6 ± 8.5 NmL H_2/g VS added) and 2199.3 ± 275.7 NmL (57.5 ± 7.2 NmL H_2/g VS added) for 72_S, and 72_{SI}, respectively. Overall, the recorded hydrogen production yields are comparable to those previously reported using FW of a similar composition [35]. The content of hydrogen in the

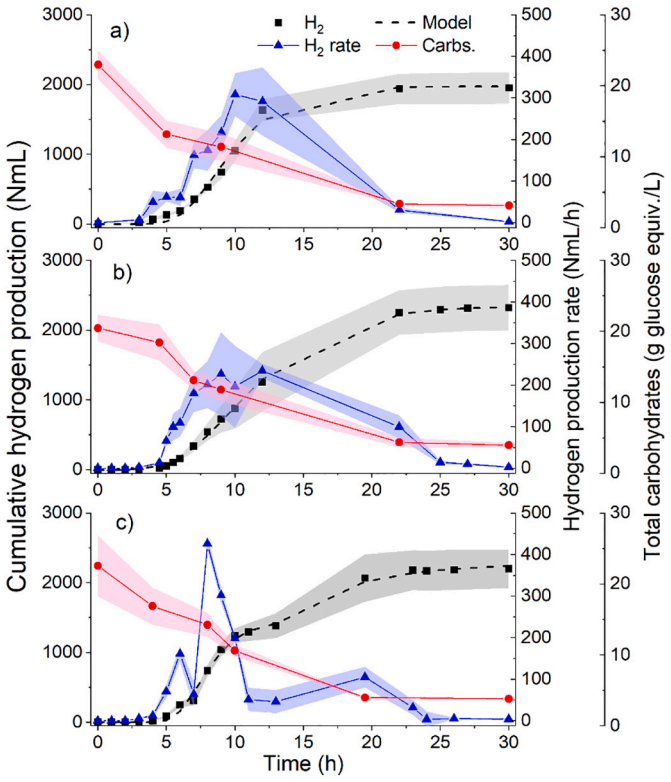

Fig. 4. Time evolution of cumulative hydrogen production, hydrogen production rate and the concentration of total carbohydrates in the fermentation broth for the three substrates tested: a) non-stored FW (0_S), b) 72-h FW stored without inoculum addition (72_S), and c) 72-h FW stored with inoculation (72_{SI}). The markers are the mean and standard deviation (shaded) values of triplicate experiments.

produced acidogenic off-gas was 43.9. ± 4.1 %, 44.5 ± 1.1 % and 38.2 ± 1.4 % (v/v) for 0_S, 72_S and 72_{SI}, respectively. The modified Gompertz model satisfactorily described the cumulative production of hydrogen, with R^2 values above 0.9950 and percentage errors between 3.8 and 9.5 % (Table 2). A visual inspection of the fitting can be seen in Fig. 4. More specifically, there was no difference in the length of lag phase between the different types of FW tested, with an average λ of 5.8 h, which is in accordance with previous observations [24]. This pointed out that the

Table 2
Kinetic parameters of the modified Gompertz model estimated from the evolution of cumulative hydrogen production recorded over culture time for the different feedstocks.

Feedstock	λ (h)	*Rmax* (NmL H_2/h)	*Pmax* (NmL H_2)	R^2	Error (%)[b]
0_S	5.9 ± 0.2	271.5 ± 33.6	1976.2 ± 222.7	0.9958	9.5
72_S	5.8 ± 0.4	216.9 ± 67.5	2390.8 ± 271.8	0.9990	4.5
72_{SI}[a]	5.7 ± 0.2	322.6 ± 3.9	1467.7 ± 199.8	0.9952	3.8
	3.0 ± 1.4	160.7 ± 47.7	2284.8 ± 266.3		

Note: 0_S: non-stored FW; **72_S:** 72-h FW storage without inoculum addition; **72_{SI}:** 72-h FW storage with inoculation.
[a] Modified Gompertz model with two sequential steps.
[b] Percentage error as the ratio of root mean square error to the mean target value.

pre-hydrolysis and pre-acidification that took place during anoxic storage did not impact the adaptation period. The hydrogen production rate peaked at 271.5 ± 33.6 NmL H_2/h for 0_S and 216.9 ± 67.5 NmL H_2/h for 72_S, while in 72_{SI} the rate accounted for 322.6 ± 3.9 NmL H_2/h and 160.7 ± 47.7 NmL H_2/h due to a diauxic hydrogen production trend. This diauxic hydrogen production observed in 72_{SI} assays could be attributed to a sequential substrate utilization phenomenon, e.g., carbohydrates, lactate (as discussed below) by the bacteria in the supplemented inoculum [53]. Based on the ratio of P_{max} to R_{max}, the estimated hydraulic retention time (HRT) ranged from 7 to 11 h, which lies within the common HRT values for DF systems [34]. The associated VHPR values were 8.2 ± 1.0, 6.5 ± 2.0, and 9.7 ± 0.1 NL H_2/L-d for 0_S, 72_S, and 72_{SI}, respectively, which are within the upper range of reported fermentative hydrogen productivity datasets using FW [35]. In general, no clear effect of FW storage on expected HRT was observed. However, the use of FW previously subjected to a storage stage resulted in numerically similar or higher extents and rates of potential biochemical hydrogen production compared to the control (0_S). This observation is contrary to the findings disclosed by Im et al. (2019) [23], but consistent with the results reported by Roslan et al. (2023) [24]. While Im and co-workers (2019) reported hydrogen inhibition (20–30 %) by the growth of indigenous LAB when FW was stored for 2 days at 20 and 30 °C, Roslan and co-workers (2023) observed that storage of FW (for 15 days at temperatures ranging from 4 to 45 °C) did not impact on the hydrogen potential but significantly improved the maximum hydrogen production rate (R_{max}). Noblecourt et al. (2018) reported the successful LD-DF of depackaging wastes, a feedstock that was pre-fermented mainly to lactate during its storage and transport [20]. Here, it is therefore hypothesized that the ability of the microbiota involved to metabolize lactate into hydrogen can make the difference between success and failure when using stored FW rich in lactate and dominated by LAB, as discussed with some details in the following paragraph.

Hydrogen can be produced fermentatively from the metabolism of carbohydrates via acetic- and butyric-type pathway, but also from the metabolism of lactate (LD-DF) [20,34]. As for the carbohydrates, their concentration in the fermentation broth showed a steep falling pattern (Fig. 4), reaching a final removal efficiency, on average, of 86.5, 80.8 and 83.1 % for 0_S, 72_S, and 72_{SI}, respectively. The organic acids spectra recorded for the three types of FW tested were very similar to each other (Fig. 5). Interestingly, lactate and acetate accumulated during the early stage of fermentation (during the first 9 h), but lactate was almost depleted after 20 h of fermentation, while acetate remained roughly unchanged (close to 4.5 g/L) till the end of fermentation. The concentration of formate also increased (but at a lower extent; 1.6–2.2 g/L) during the early stage of fermentation and then slightly decreased down to 0.7–1.2 g/L at the end, regardless of the storage condition tested (Fig. 5). The depletion of lactate was found to be highly correlated with

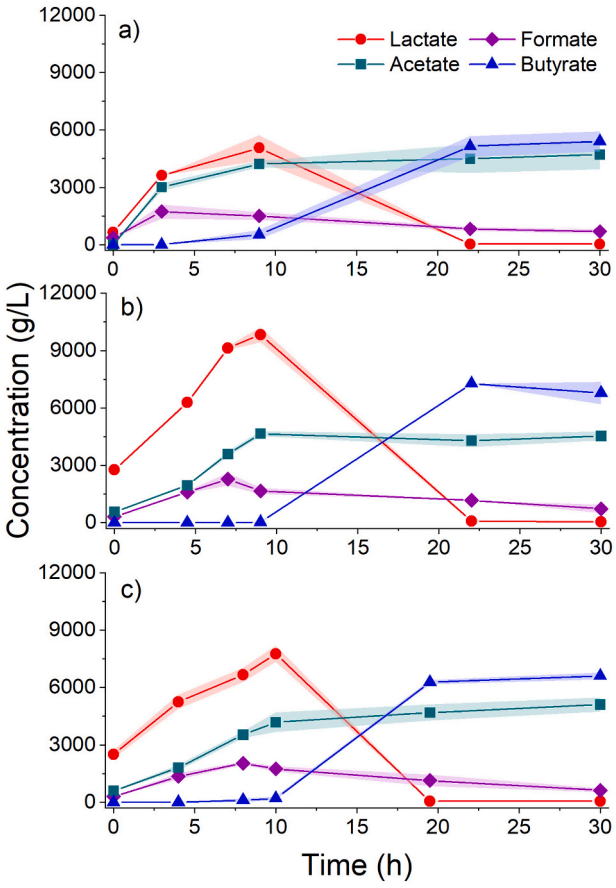

Fig. 5. Time evolution of organic acid concentration during the LD-DF of the three substrates tested: a) non-stored FW (0_S), b) 72-h FW stored without inoculum addition (72_S), and c) 72-h FW stored with inoculation (72_{SI}). The markers are the mean and standard deviation (error lines are indicated with colored shade areas) values of triplicate experiments.

the accumulation of butyrate (5.4–6.7 g/L) in the culture broth. Based on the metabolic patterns observed, it is highly probable that lactate was used as the main carbon source in the DF of both non-stored and stored FW. The LD-DF has been reported for FW [20,35,38], but also for some other complex substrates, e.g., vinasses [37,39]. It is widely recognized that the microorganisms involved in the LD-DF process can govern the fate of lactate during fermentation [34]. It is thus reasonable to argue that the hydrogenogenic inoculum herein used, which had a proven potential to perform LD-DF [35,38], was of utmost importance to allow a dual lactate fermentation: the fermentation of carbohydrates to lactate and the fermentation of lactate to hydrogen. It should be noted that besides lactate, fermentable carbohydrates are also hydrogen precursors, and therefore, part of the evolved hydrogen might have been produced directly from them. The bioconversion of formate into hydrogen and carbon dioxide might have also resulted in additional hydrogen. Nonetheless, the LD-DF was found to be crucial to deal with a FW enriched in lactate and LAB, which otherwise would lead to lactate accumulation accompanied by a poor hydrogen production performance [23,27]. Here it should be noted that the present study simulated a specific storage condition and used synthetic FW as the feedstock. Thus, future studies should investigate the biotic and abiotic variations of real FW that might occur during transportation and storage under the real conditions of urban FW management, and test how such changes might affect the outcome of the LD-DF process. Recently, lactic acid fermentation of FW was successfully proven to be a suitable storage method prior to produce fermentative hydrogen in batch mode [24]. In this context, it would be interesting to study the integration of lactic acid

pre-fermentation storage to continuous LD-DF.

3.3. Effect of FW storage on biogas production

The storage of FW did not markedly affect its BMP. The methane yields recorded after 29 days of biomethanization were 394.0 ± 10.5, 382.1 ± 6.1 and 416.9 ± 11.9 NmL CH_4/g VS added for 0_S, 72_S and 72_{SI}, respectively (Fig. 6), which agreed well with typical BMP data reported for FW [13,16,17]. Such recorded methane yields corresponded to biodegradability values in the range of 73 to 79 %, based on the theoretical BMP of the FW utilized. Kinetics of the time-course behavior of BMP was adequately described by the modified Gompertz model, with R^2 values close to 1 and percentage errors between 3.7 and 8.0 % (Fig. 6 and Table 3). The specific rate of methane production supported by stored FW was similar or even slightly higher than that of fresh FW, reaching up to 40.8 ± 2.4 NmL CH_4/g VS added-day in the case of 72_{SI} (Table 3). Considering the ratio P_{max}/R_{max}, the expected HRT was between 10 and 16 days. In the literature, there are discrepancies concerning the effect of FW storage on methane potential. Some studies have reported a negative effect on methane potential when storing FW. In this vein, Nie et al. (2022) reported that FW was hydrolyzed (about 35 %) and acidified (about 16 %) during transportation, generating up to 18.1 g/L of lactic acid (equivalent to 20 % of dissolved organic carbon) [16]. The authors recorded 25 % lower methane yields in transported and stored FW compared to fresh FW. Parra-Orobio and co-workers also found that storage (25 °C) shorter than 7 days negatively impacted the mesophilic methane production behavior of FW, mainly the methane production yield and rate rather than the lag phase. The increase in VFAs and the formation of humic substances during storage were the reasons identified for biogas inhibition. In another study, Degueurce et al. (2020) estimated a carbon loss value of around 10 % after a 4-days FW storage and a slightly methane inhibition associated to high levels of lactic acid [21]. However, in the same study, the authors reported that 1 week of storage increased the methane yield while compensating the loss of organic matter. Finally, Sailer et al. (2022) studied the impact of storage duration (20 to 40 days) and temperature (5 and 20 °C) under aerobic and anoxic conditions on the methane production potential of recipe-based FW. The authors found that the degree of preservation of methane potential depended on the physico-chemical changes occurring during storage, which in turn were dependent on whether storing was either performed under oxic or anoxic conditions. Compared to a control, aerobic storage led to 31 % (20 °C) and 16 % (5 °C) lower methane yields. In contrast, anaerobic storage preserved the methane potential of FW regardless of the storage time and temperature.

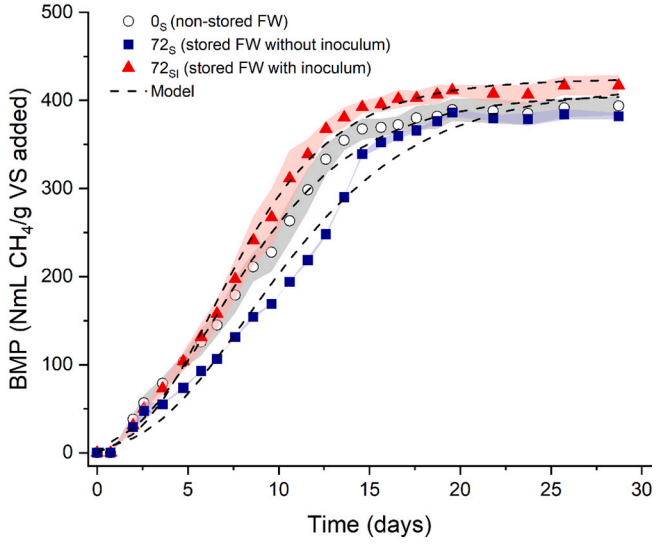

Fig. 6. Cumulative methane production from the BMP test on the three different types of FW. The markers are the mean and standard deviation (error lines are indicated with colored shade areas) values of triplicate experiments.

Table 3
Kinetic parameters estimated from the modified Gompertz model for the methane evolution recorded for 0_S (non-stored FW), 72_S (72-h FW storage without inoculum addition), and 72_{SI} (72-h FW storage with inoculation).

Feedstock	λ (days)	*Rmax* (NmL CH_4/g VS added-day)	*Pmax* (NmL CH_4/g VS added)	R^2	Error (%)[a]
0_S	1.9 ± 0.3	33.6 ± 1.8	407.0 ± 13.6	0.9960	4.8
72_S	3.1 ± 0.4	26.6 ± 3.4	416.8 ± 5.6	0.9907	8.0
72_{SI}	2.4 ± 0.3	40.8 ± 2.4	424.2 ± 8.2	0.9971	3.7

[a] Percentage error as the ratio of root mean square error to the mean target value.

Enhanced BMPs have been also reported with the use of FW previously subjected to storage compared to the fresh FW [17,31]. For instance, Lü el al. (2016) observed BMP values ranging from 285 to 308 NmL CH_4/g VS added using fresh or 1-day stored FW, while BMP values of 418–530 NmL CH_4/g VS added and 618–696 NmL CH_4/g VS added were recorded when using FW stored for 2–4 and 5–12 days, respectively [17]. The authors found that the methane yield was positively correlated with the acidification efficiency but not with the total hydrolysis efficiency. Påledal et al. (2018) also reported that the storage of FW in plastic bags for up to 21 days at 22 °C did not change the methane potential but led to faster methanization in comparison with fresh FW. The faster methane production rate was attributed to the preservation of volatile organic compounds inside the plastic bag and the promotion of the pre-hydrolysis of FW rather than its aerobic degradation [18]. Pangallo et al. (2021) investigated the effect of bioplastic bag storing (2, 4 and 6 days at 22 °C) on the BMP of the organic fraction of municipal solid waste (OFMSW) and found that methane yields slightly increased with the increase of the storage time, achieving yields between 500 and 650 NmL CH_4/g VS added.

Avoiding the loss of VS during FW storage is desirable for preserving the methane potential [18,33]. In this regard, the intentional addition of LAB to the storage of artificial kitchen waste has indeed been proposed as a measure to suppress the growth of putrefactive bacteria, while preserving its energy content for further biomethanization [52]. Daly and co-workers (2020) stated that the storage of FW followed classical ensilage dynamics with homofermentation to lactate together with low pH, thus preventing the subsequent breakdown (fermentation) of lactate into other carboxylic acids and hydrogen gas [19]. Lactate is a key intermediate in the AD process, which can allocate more energy for biogas production than other acidogenic end products (e.g., ethanol, acetate, butyrate, propionate) [13,29,30]. However, it is worth noting that the methane potential of lactate-rich stored FW may be impaired due to a reactor acidification and accumulation of propionic acid [16]. Herein, no overwhelming accumulation of VFAs was detected at the end of BMP, with a total concentration of 35.5 ± 8.7, 27.1 ± 4.0, and 23.1 ± 2.1 mg COD equiv./L for 0_S, 72_S and 72_{SI}, respectively. The average concentration of propionic acid at the end of the BMP test stayed 6 mg/L regardless of the substrate tested. Indeed, the final pH of the digestates averaged 7.6 ± 0.07 (data not shown). Such low organic acids levels measured along with the acceptable BMP recorded (even with lactate-rich FW) strongly suggested that lactate and its degradation products (such as acetate) were successfully biomethanized. The successful syntrophic oxidation of lactate without propionate accumulation could explain the high BMPs values observed for stored FW. As stated earlier, it would be interesting to study the integration of lactic acid pre-fermentation storage to continuous biogas production.

4. Conclusions

This study examined the biotic and abiotic changes of FW during its storage under a closed atmosphere without and with inoculation of a mixed culture enriched in LAB. This study also investigated how such changes in the stored FW impact its biochemical hydrogen and methane production potential. The addition of inoculum did not exert an important effect on the storage of FW, which following 3-day of storage was mainly characterized by a LAB-dominant microbial community. The accumulation of lactate as a result of the hydrolysis-acidogenesis of FW was favored by the self-generated anoxic-and-low pH conditions that occurred during the storage. Lactate-based hydrogen production was found to be of outermost importance in the DF of both the non-stored and stored FW. Compared to the non-stored FW, the use of stored FW resulted in a similar, or even, an improved hydrogen production performance. Likewise, the methane potential of FW was not impaired by its storage, which was supported by the successful conversion of lactate-rich stored FW to biogas. Finally, the use of FW stored for 3 days with inoculation shorten the foreseen fermentation times for both the hydrogen and methane production, an interesting finding that should be investigated further.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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References

[1] European Commission, Closing the loop - An EU action plan for the circular economy, Available at, https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=CELEX:52015DC0614, 2015. accessed on 22/08/2022.
[2] UN Environment Programme, Food waste index report 2021, Available at, https://wedocs.unep.org/handle/20.500.11822/35280, 2021. accessed on 22/08/2022.
[3] Fusions, Estimates of European food waste levels, Available at, https://cordis.europa.eu/project/id/311972/reporting, 2016. accessed on 22/08/2022.
[4] CEC, Characterization and Management of Food Loss and Waste in North America, Commission for Environmental Cooperation, Montreal, Canada, 2017, 48 pp.
[5] European Commission, Food waste, Available at, https://food.ec.europa.eu/safety/food-waste_en, 2022. accessed on 22/08/2022.
[6] Inter-American Development Bank, Available at, https://www.iadb.org/en/news/idb-and-partners-launch-platform-fight-food-loss-and-waste-0, 2018. accessed on 05/05/2023.
[7] World Food Program USA, Available at, https://www.wfpusa.org/articles/8-facts-to-know-about-food-waste-and-hunger/, 2021. accessed on 05/05/2023.
[8] H. Fisgativa, A. Tremier, P. Dabert, Characterizing the variability of food waste quality: a need for efficient valorisation through anaerobic digestion, Waste Manag. 50 (2016) 264–274, https://doi.org/10.1016/j.wasman.2016.01.041.
[9] S. Dahiya, A.N. Kumar, J.S. Sravan, S. Chatterjee, O. Sarkar, S.V. Mohan, Food waste biorefinery: sustainable strategy for circular bioeconomy, Bioresour. Technol. 248 (2018) 2–12, https://doi.org/10.1016/j.biortech.2017.07.176.
[10] S. Venkata Mohan, G.N. Nikhil, P. Chiranjeevi, C. Nagendranatha Reddy, M. V. Rohit, A.N. Kumar, et al., Waste biorefinery models towards sustainable circular bioeconomy: critical review and future perspectives, Bioresour. Technol. 215 (2016) 2–12, https://doi.org/10.1016/j.biortech.2016.03.130.
[11] G. Srisowmeya, M. Chakravarthy, N.G. Devi, Critical considerations in two-stage anaerobic digestion of food waste – a review, Renew. Sust. Energ. Rev. 119 (2020), https://doi.org/10.1016/j.rser.2019.109587.
[12] T.I. Assis, R.F. Gonçalves, Valorization of food waste by anaerobic digestion: a bibliometric and systematic review focusing on optimization, J. Environ. Manag. 320 (2022) 115763, https://doi.org/10.1016/j.jenvman.2022.115763.
[13] O. García-Depraect, L.J. Martínez-Mendoza, I. Diaz, R. Muñoz, Two-stage anaerobic digestion of food waste: enhanced bioenergy production rate by steering lactate-type fermentation during hydrolysis-acidogenesis, Bioresour. Technol. 358 (2022) 127358, https://doi.org/10.1016/j.biortech.2022.127358.
[14] Y.-M. Yun, M.-K. Lee, S.-W. Im, A. Marone, E. Trably, S.-S. Shin, et al., Biohydrogen production from food waste: current status, limitations, and future perspectives, Bioresour. Technol. 248 (2018) 79–87, https://doi.org/10.1016/j.biortech.2017.06.107.
[15] F. Xu, Y. Li, X. Ge, L. Yang, Y. Li, Anaerobic digestion of food waste – challenges and opportunities, Bioresour. Technol. 247 (2018) 1047–1058, https://doi.org/10.1016/j.biortech.2017.09.020.
[16] E. Nie, P. He, J. Zou, H. Zhang, F. Lü, Neglected effect of transportation on the property of municipal biowaste and the subsequent biomethane potential, J. Clean. Prod. 352 (2022) 131603, https://doi.org/10.1016/j.jclepro.2022.131603.
[17] F. Lü, X. Xu, L. Shao, P. He, Importance of storage time in mesophilic anaerobic digestion of food waste, J. Environ. Sci. 45 (2016) 76–83, https://doi.org/10.1016/j.jes.2015.11.019.
[18] S.N. Påledal, E. Hellman, J. Moestedt, The effect of temperature, storage time and collection method on biomethane potential of source separated household food waste, Waste Manag. 71 (2018) 636–643, https://doi.org/10.1016/j.wasman.2017.05.034.
[19] S.E. Daly, J.G. Usack, L.A. Harroff, J.G. Booth, M.P. Keleman, L.T. Angenent, Systematic analysis of factors that affect food-waste storage: Toward maximizing lactate accumulation for resource recovery, ACS Sustain. Chem. Eng. (2020) 13934–13944, https://doi.org/10.1021/acssuschemeng.0c03161.
[20] A. Noblecourt, G. Christophe, C. Larroche, P. Fontanille, Hydrogen production by dark fermentation from pre-fermented depackaging food wastes, Bioresour. Technol. 247 (2018) 864–870, https://doi.org/10.1016/j.biortech.2017.09.199.
[21] A. Degueurce, S. Picard, P. Peu, A. Trémier, Storage of food waste: variations of physical–chemical characteristics and consequences on biomethane potential, Waste Biomass Valori. (2020) 2441–2454, https://doi.org/10.1007/s12649-018-00570-0.
[22] S. Wu, S. Xu, X. Chen, H. Sun, M. Hu, Z. Bai, et al., Bacterial communities changes during food waste spoilage, Sci. Rep. 8 (2018) 1–9, https://doi.org/10.1038/s41598-018-26494-2.
[23] S. Im, M.-K. Lee, Y.-M. Yun, S.-K. Cho, D.-H. Kim, Effect of storage time and temperature on hydrogen fermentation of food waste, Int. J. Hydrog. Energy 45 (2019) 3769–3775, https://doi.org/10.1016/j.ijhydene.2019.06.215.
[24] E. Roslan, J.A. Magdalena, H. Mohamed, A. Akhiar, A.H. Shamsuddin, H. Carrere, E. Trably, Lactic acid fermentation of food waste as storage method prior to biohydrogen production: effect of storage temperature on biohydrogen potential and microbial communities, Bioresour. Technol. 378 (2023) 128985, https://doi.org/10.1016/j.biortech.2023.128985.
[25] Z. Zhang, P. Tsapekos, M. Alvarado-Morales, I. Angelidaki, Impact of storage duration and micro-aerobic conditions on lactic acid production from food waste, Bioresour. Technol. 323 (2021) 124618, https://doi.org/10.1016/j.biortech.2020.124618.
[26] M.C. Noguer, R. Escudié, N. Bernet, T. Eric, Populational and metabolic shifts induced by acetate, butyrate and lactate in dark fermentation, Int. J. Hydrog. Energy 47 (2022) 28385–28398, https://doi.org/10.1016/j.ijhydene.2022.06.163.
[27] M.A.Z. Bundhoo, R. Mohee, Inhibition of dark fermentative bio-hydrogen production: a review, Int. J. Hydrog. Energy 41 (2016) 6713–6733, https://doi.org/10.1016/j.ijhydene.2016.03.057.
[28] A. Detman, M. Bucha, L. Treu, A. Chojnacka, Ł. Pleśniak, A. Salamon, et al., Evaluation of acidogenesis products' effect on biogas production performed with metagenomics and isotopic approaches, Biotechnol. Biofuels (2021) 1–25, https://doi.org/10.1186/s13068-021-01968-0.
[29] P. Pipyn, W. Verstraete, Lactate and ethanol as intermediates in two-phase anaerobic digestion, Biotechnol. Bioeng. 23 (1981) 1145–1154, https://doi.org/10.1002/bit.260230521.
[30] Y. Wu, C. Wang, X. Liu, H. Ma, J. Wu, J. Zuo, et al., A new method of two-phase anaerobic digestion for fruit and vegetable waste treatment, Bioresour. Technol. 211 (2016) 16–23, https://doi.org/10.1016/j.biortech.2016.03.050.
[31] D. Pangallo, A. Pedullá, D.A. Zema, P.S. Calabró, Influence of the preliminary storage on methane yield of solid waste, Processes 9 (2021) 2017, https://doi.org/10.3390/pr9112017.
[32] B.A. Parra-Orobio, M.A. Correa-Betancourt, M.A. Lozano-Messa, Y.F. Foronda-Zapata, L.F. Marmolejo-Rebellón, P. Torres-Lozada, Influence of storage time of food waste on the substrate characteristics and energetic potential through

anaerobic digestion, Water Air Soil Pollut. 233 (2022) 190, https://doi.org/10.1007/s11270-022-05657-y.
[33] G. Sailer, J. Eichermüller, F. Empl, J. Poetsch, S. Pelz, D. Kuptz, et al., Improving the energetic utilization of household food waste: impact of temperature and atmosphere during storage, Waste Manag. 144 (2022) 366–375, https://doi.org/10.1016/j.wasman.2022.04.012.
[34] O. García-Depraect, R. Castro-Muñoz, R. Muñoz, E.R. Rene, E. León-Becerril, I. Valdez-Vazquez, et al., A review on the factors influencing biohydrogen production from lactate: the key to unlocking enhanced dark fermentative processes, Bioresour. Technol. 324 (2021) 124595, https://doi.org/10.1016/j.biortech.2020.124595.
[35] L. Regueira-Marcos, O. García-Depraect, R. Muñoz, Elucidating the role of pH and total solids content in the co-production of biohydrogen and carboxylic acids from food waste via lactate-driven dark fermentation 338 (2023) 127238, https://doi.org/10.1016/j.fuel.2022.127238.
[36] D. Marín, M. Lara, I. Suero, I. Díaz, S. Blanco, M. Fdz-Polanco, et al., Anaerobic digestion of food waste coupled with biogas upgrading in an outdoors algal-bacterial photobioreactor at pilot scale, Fuel 324 (2022) 30–40, https://doi.org/10.1016/j.fuel.2022.124554.
[37] O. García-Depraect, I. Valdez-Vázquez, E.R. Rene, J. Gómez-Romero, A. López-López, E. León-Becerril, Lactate- and acetate-based biohydrogen production through dark co-fermentation of tequila vinasse and nixtamalization wastewater: Metabolic and microbial community dynamics, Bioresour. Technol. 282 (2019) 236–244, https://doi.org/10.1016/j.biortech.2019.02.100.
[38] L.J. Martínez-Mendoza, O. García-Depraect, R. Lebrero, R. Muñoz, Influence of key operational parameters on biohydrogen production from fruit and vegetable waste via lactate-driven dark fermentation, Bioresour. Technol. 364 (2022) 128070, https://doi.org/10.1016/j.biortech.2022.128070.
[39] V.F. Diaz-Cruces, O. García-Depraect, E. León-Becerril, Effect of lactate fermentation type on the biochemical methane potential of tequila vinasse, BioEnergy Res. 13 (2020) 571–580, https://doi.org/10.1007/s12155-020-10093-z.
[40] X. Chen, H. Yuan, D. Zou, Y. Liu, B. Zhu, A. Chufo, et al., Improving biomethane yield by controlling fermentation type of acidogenic phase in two-phase anaerobic co-digestion of food waste and rice straw, Chem. Eng. J. 273 (2015) 254–260, https://doi.org/10.1016/j.cej.2015.03.067.
[41] S. Strömberg, M. Nistor, J. Liu, Towards eliminating systematic errors caused by the experimental conditions in biochemical methane potential (BMP) tests, Waste Manag. 34 (2014) 1939–1948, https://doi.org/10.1016/j.wasman.2014.07.018.
[42] S. Achinas, G. Jan, W. Euverink, Theoretical analysis of biogas potential prediction from agricultural waste, Resour. Technol. 2 (2016) 143–147, https://doi.org/10.1016/j.reffit.2016.08.001.
[43] O. García-Depraect, R. Lebrero, S. Rodriguez-Vega, S. Bordel, F. Santos-Beneit, L. J. Martínez-Mendoza, et al., Biodegradation of bioplastics under aerobic and anaerobic aqueous conditions: kinetics, carbon fate and particle size effect, Bioresour. Technol. 126265 (2022), https://doi.org/10.1016/j.biortech.2021.126265.
[44] O. García-Depraect, R. Lebrero, S. Rodriguez-Vega, R.A. Börner, T. Börner, R. Muñoz, Production of volatile fatty acids (VFAs) from five commercial bioplastics via acidogenic fermentation, Bioresour. Technol. 360 (2022) 127655, https://doi.org/10.1016/j.biortech.2022.127655.
[45] A. Klindworth, E. Pruesse, T. Schweer, J. Peplies, C. Quast, M. Horn, et al., Evaluation of general 16S ribosomal RNA gene PCR primers for classical and next-generation sequencing-based diversity studies, Nucleic Acids Res. 41 (2013), https://doi.org/10.1093/nar/gks808.
[46] B.J. Callahan, P.J. McMurdie, M.J. Rosen, A.W. Han, A.J.A. Johnson, S.P. Holmes, DADA2: high-resolution sample inference from Illumina amplicon data, Nat. Methods 13 (2016) 581–583, https://doi.org/10.1038/nmeth.3869.
[47] S.F. Altschul, W. Gish, W. Miller, E.W. Myers, D.J. Lipman, Basic local alignment search tool, J. Mol. Biol. 215 (1990) 403–410, https://doi.org/10.1016/S0022-2836(05)80360-2.
[48] N.A. Bokulich, B.D. Kaehler, J.R. Rideout, M. Dillon, E. Boylen, R. Knight, et al., Optimizing taxonomic classification of marker-gene amplicon sequences with QIIME 2's q2-feature-classifier plugin, Microbiome 6 (2018) 90, https://doi.org/10.1186/s40168-018-0470-z.
[49] C. Quast, E. Pruesse, P. Yilmaz, J. Gerken, T. Schweer, P. Yarza, et al., The SILVA ribosomal RNA gene database project: improved data processing and web-based tools, Nucleic Acids Res. 41 (2013), https://doi.org/10.1093/nar/gks1219.
[50] S. Weiss, Z.Z. Xu, S. Peddada, A. Amir, K. Bittinger, A. Gonzalez, et al., Normalization and microbial differential abundance strategies depend upon data characteristics, Microbiome 5 (2017) 27, https://doi.org/10.1186/s40168-017-0237-y.
[51] Q. Wang, K. Yamabe, J. Narita, M. Morishita, Y. Ohsumi, K. Kusano, Y. Shirai, H. I. Ogawa, Suppression of growth of putrefactive and food poisoning bacteria by lactic acid fermentation of kitchen waste, Process Biochem. 37 (2001) 351–357, https://doi.org/10.1016/S0032-9592(01)00217-5.
[52] S. Tamaki, T. Hidaka, F. Nishimura, Effects of using lactic acid bacteria in the storage and subsequent anaerobic co-digestion of crushed kitchen garbage, Bioresour. Technol. Reports 13 (2021) 100640, https://doi.org/10.1016/j.biteb.2021.100640.
[53] O. García-Depraect, V. Mena-Navarro, R. Muñoz, E.R. Rene, E. León-Becerril, Effect of nitrogen and iron supplementation on the process performance and microbial community structure of a hydrogen-producing reactor continuously fed with tequila vinasse, Fuel 334 (2023) 126736, https://doi.org/10.1016/j.fuel.2022.126736.