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Mechanical pretreatment of municipal biowaste to produce an aqueous slurry dedicated to anaerobic digestion

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Abstract

This study investigated a wet mechanical pretreatment to improve methane production by anaerobic digestion from biowaste material by separating a biodegradable aqueous slurry fraction (ASF) from a more recalcitrant particulate fraction (PF). Four source-sorted municipal biowaste were studied, namely Household (HBW), Supermarket (SBW), Restaurant (RBW) and Green Biowaste (GBW). The treatment consisted in soaking the waste in water and then press the slurry through a grid with 3-mm openings to separate the 2 fractions. Methane production of ASF and PF obtained from the 4 biowastes were

measured using the BMP protocol and compared to the potential of the respective untreated biowaste.

Results were very different for GBW as compared to the other 3 BW. With GBW, which was the most lignocellulosic of the BW studied, only 17% of the initial methane potential was recovered in the ASF. The extraction was much better on the other biowastes and increased in the following order: HBW (58%) $\approx$ RBW (57%) < SBW (67%). The ASF from these biowastes exhibited low total solids contents and high BMPs (416, 408 and 423 NL_{CH₄}.g⁻¹_{vs} for HBW, RBW and SBW respectively).

The experimental results obtained in this study therefore showed that wet pressing separation was an efficient pretreatment to improve and facilitate methane production by anaerobic digestion of biowaste such as HBW, RBW and SBW.

Keywords: biomethane potential; municipal organic waste; wet press separation; energy recovery; biodegradable organic fraction; bioconversion

Graphical abstract

Mechanical pretreatment of municipal biowaste to produce an aqueous slurry dedicated to anaerobic digestion

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1 Introduction

Municipal solid waste production (MSW) is still growing in most European cities despite the efforts made to reduce it (Fisgativa et al. 2016; Braguglia et al. 2018). MSW comprises several categories of waste collected on urban territories, including biowaste (BW). According to the European Directive 2018/851, BW includes biodegradable garden and park waste, food waste from restaurants and supermarkets, and the biodegradable organic fraction of household waste (European Union 2008). The production of municipal biowaste in EU-28 was estimated to 86.10^6 Tons per year. Household BW and Garden BW represented 60% and 35% of this production respectively (European Environment Agency 2020). Due to their biological origins and specific characteristics, BW are potential resources of organic matter for the production of compost, nutrients, or energy (Escamilla-Alvarado et al. 2017).

Selective collection of BW is developing fast in several European urban areas (Bernstad et al. 2014; Sidaine and Gass 2013). European regulations will make it compulsory in 2023 (European Union 2018). This evolution should improve the quality of the recovered organic matter and thereby favor its reuse for agricultural purposes in the future (Hansen et al. 2007). However, the possible presence of undesirable materials such as glass, plastics, ceramics, etc and even hazardous substances such as heavy metals and other micropollutants, is likely to remain a significant risk in big cities where the quality of the selective collection may be degraded for various reasons (Weithmann et al. 2018). In this context, energy recovery appears more adapted in urban territories because it would be less sensitive to possible contaminations of the resource.

Among the different possible strategies to convert BW into energy, methane production is attractive in urban territories. Methane has a high energy content of 10 kWh per Nm³, several possible energy usages, and it is already distributed in many cities through existing grids. BW however contains different proportions of biodegradable matter and more recalcitrant constituents such as lignocellulosic compounds (St Joly et al. 2000; Físgativa et al. 2017). Several types of processes have therefore been used to optimize methane production from BW. Biological anaerobic digestion is best adapted to convert the humid and readily biodegradable organic constituents (Capson-Tojo et al. 2016). Thermochemical processes such as gasification would be more adapted for their less humid and more recalcitrant constituents (Vakalis et al. 2017). Pretreatments of BW are therefore needed to separate the 2 types of constituents and apply adapted treatments to each of them (Rodríguez-Valderrama et al. 2020).

Mechanical treatments have been used for decades in solid waste treatment applications, and still are. Sorting operations are used to remove undesirable materials or, on the contrary, recover valuable constituents from a waste flow (Ariunbaatar et al. 2014; Pognani et al. 2012). Several authors have used mechanical operations as pretreatments prior to anaerobic digestion (Hansen et al. 2007; Do Carmo Precci Lopes et al. 2019). Press-separation was investigated to remove undesirable impurities and recalcitrant particulate matter from BW (Jank et al. 2015) or to produce a biodegradable aqueous slurry (Micolucci et al. 2015a; Do Carmo Precci Lopes et al. 2019). However, most of published studies on the subject have concerned household BW mixed or not with green waste. Do Carmo Precci Lopes et al. (2019) reported that the design and implementation of mechanical pretreatments was still a challenging step to convert BW into methane

and/or compost. To our knowledge, no comparative study has been conducted on specific BW categories as reported in the present article.

The present study investigated the implementation of wet pressing as a mechanical pretreatment of 4 types of BW to optimize their anaerobic digestion. The treatment was meant to generate an aqueous slurry fraction (ASF) expected to extract as much as possible the soluble readily biodegradable constituents, and a particulate fraction (PF) expected to collect fibrous and particulate constituents along with undesirable materials that may be present. The aqueous slurry is to be oriented to anaerobic digestion to produce methane, whereas the particulate fraction would be treated by gasification followed by methanation of syngas.

2 Materials and methods

2.1 Selection and characterization of biowaste samples

The categories of BW material investigated in this study were selected in collaboration with the technical services of Lyon Metropolitan Area, France, considering the following criteria:

- Quantitative production on the considered territory;
- Availability (dispersion, accessibility), collection costs and possible difficulties;
- Territorial specificities, public policies, political strategies, other operational issues.

Four types of BW were selected, namely Garden BW (GBW) collected from domestic, municipal and private sources; BW from restaurants (RBW) collected from private and public collective catering sources including schools; Household kitchen BW (HBW) collected from a set 200 hundred people (faculty members, staff and students of our

institution) who separated at source their own food waste; BW from supermarkets (SBW) collected from a company specialized in depackaging of mixed supermarket food waste. Representative samples of about 200 kg of each BW were collected in September 2018 following standard sampling guidelines and protocols NF EN 14899 (2006). Each sample was homogenized by coarse shredding using a Blik BB350, and analyzed as reported in a previous work (Moretti et al. 2020). All analyses were duplicated and results were well reproducible. Table S1 (supplementary data) gathers the physico-chemical composition and biodegradation potential of each BW, which were determined as described in Moretti et al. (2020).

2.2 Biowaste pre-treatment by soaking and pressing

The protocol followed to treat the different BW samples is illustrated in Figure 1. Assays were done in triplicates. Each shredded sample was soaked in de-ionized water in a 1 L glass beaker. Two liquid to solid ratios were tested: 5 and 10 g of water per g of dry BW. 250 mL of the suspensions were mixed for 20 minutes at a room temperature of $23\pm 1^{\circ}\text{C}$, in a Heidolph REAX 20 tumbler agitator set at 10 rpm. The objective of this operation was to extract as much as possible the soluble and colloidal organic matter from the BW samples.

Lab-scale filtration-compression cells were then used to separate ASF from PF. The experimental setup is shown in Figure S1 (supplementary data). It was made of three 0.46L cylindrical stainless steel cells of 70 mm inner diameter and 120 mm height. A stainless-steel disk perforated with holes of 3 mm diameter was placed at the bottom of the cylinder. The BW suspensions obtained after soaking in water were introduced into the 3 cells in order to run triplicate assays for each BW sample. The filtrate (ASF) was collected from the bottom of the cells and its mass monitored online over time. After 20 minutes pressing, the system was stopped and PF was collected from the cells.

The experimental conditions tested are gathered in Table 1. Due to the high and regular annual production of HBW and the current evolution of the European regulations which will make it compulsory to collect this waste selectively and manage it specifically in 2023, HBW was selected as the resource to focus on in priority. The first two series of experiments (first 2 lines of Table 1) were carried out only with the HBW. These experiments compared 2 pressures (3 and 6 bars) and L/S ratios for the wet-pressing treatment. The treatment duration was fixed at 20 min. Preliminary studies had shown that this duration would guarantee no kinetic limitations that could bias the comparisons

between the waste samples. The last series of experiments was done to compare the results obtained with the different waste samples. These experiments were carried out at a given pressure (6 bars) and given L/S ratio (10). The four BW were studied for comparison.

2.3- Analysis of aqueous slurry (ASF) and particulate fractions (PF)

2.3.1 *Physical and chemical analyses*

ASF and PF obtained from the pretreatment of each BW were characterized in triplicates. Total solids (TS) were measured by drying for 24h at 105°C known masses of samples and weigh them dry (Baird and Bridgewater 2017). Volatile Solids (VS) were determined as the mass loss upon calcination at 550 °C for 4 h (Baird and Bridgewater 2017). Chemical oxygen demand (COD), Total Kjeldahl nitrogen (TKN), and ammonia nitrogen (NH₃-N), were analyzed according to standard methods (Baird and Bridgewater 2017) on samples dried at 70 °C for 3 days (until to constant weight) and crushed down to below 2 mm. Water soluble carbohydrates (WSC) and Volatile fatty acids (VFA) were analyzed only in the aqueous slurries by Ionic Chromatography Dionex ICS5000 Thermo Fisher after filtration at 0.45 µm.

2.3.2 *Biochemical methane potential (BMP)*

Biochemical methane potentials (BMP) were measured on each selected BW and on ASF and PF, following the guidelines reported by Holliger et al. (2016). For BW samples and PF, BMP assays were conducted at 35°C in 2 L glass vessels, for ASF, assays was performed in 0.5 L. The inoculum was a fresh digested sludge taken from the wastewater

treatment plant of La Feyssine, Lyon, France (TS 2.0-3.3%wt; VS 1.4-2.2%wt). The inoculum to substrate VS ratio was 2 g/g. Once filled, the glass vessels were purged with N₂/CO₂ (80/20% v/v) gas for about 2 minutes, sealed and equilibrated at 35°C. Blanks containing only the inoculum and water were systematically monitored along with each series of assays in order to correct the recorded BMP from residual methane production of the inoculum. Positive controls with cellulose were also monitored.

All blanks and assays were triplicated. Biogas production was followed by monitoring the pressure in the flasks using a Digitron precision manometer. Biogas was released from the flasks when the pressure exceeded 1200 hPa. Gas composition was analyzed using an Agilent 3000 micro gas chromatography equipped with a thermal conductivity detector (GC-TCD). Molsieve 5A (14 m length; pore size: 5 Å) and PoraPlot A (10 m length; 0.320 mm ID) columns were used as stationary phases for GC-TCD, using Argon as a carrier gas. According to the protocol described by Holliger et al. (2016), tests were stopped when the daily biogas production was less than 1% of the total volume of biogas produced.

The rate of methane production was determined from the net methane production (i.e., after subtracting the blank methane production) according to a first order kinetic model (equation 1).

$$V_{CH_4}(t) = V_{max}(1 - e^{-kt}) \quad (1)$$

where $V_{CH_4}(NL)$ is the cumulated volume of methane produced at time t ; t (d) is the time, V_{max} (NL) the maximum volume of methane produced, and the first order kinetic constant.

2.3.4 Balance of total solids, volatile solids, oxidizable matter (COD) and BMP between aqueous slurry and particulate fraction

Mass balance calculations were performed on total solids (TS), volatile solids (VS), COD and BMP using the results obtained from the analyses done on each BW before treatment and on the fractions ASF and PF obtained after separation.

Parameters $x_{i,ASF}$ and $x_{i,PF}$ were defined as the proportions of parameter i (TS, VS, COD and methane production) of each respective BW sample which were transferred into ASF and PF fractions during the pretreatment operations. They were expressed in % w/w and calculated according to equations (2), (2'), (3), (3'), (4) and (4').

$$x_{VS,ASF} = \frac{m_{VS,ASF}}{m_{VS,ASF} + m_{VS,PF}} \cdot 100 \quad (2) \quad x_{VS,PF} = \frac{m_{VS,PF}}{m_{VS,ASF} + m_{VS,PF}} \cdot 100 \quad (2')$$

$$x_{CH_4,ASF} = \frac{V_{CH_4,ASF}}{V_{CH_4,ASF} + V_{CH_4,PF}} \cdot 100 \quad (3) \quad x_{CH_4,PF} = \frac{V_{CH_4,PF}}{V_{CH_4,ASF} + V_{CH_4,PF}} \cdot 100 \quad (3')$$

$$x_{COD,ASF} = \frac{m_{cod,ASF}}{m_{cod,ASF} + m_{cod,PF}} \cdot 100 \quad (4) \quad x_{COD,PF} = \frac{m_{cod,PF}}{m_{cod,ASF} + m_{cod,PF}} \cdot 100 \quad (4')$$

Where:

$m_{s,ASF}$: Mass of total or volatile solids in ASF (g_{TS} or g_{VS})

$m_{s,PF}$: Mass of total or volatile solids in PF (g_{TS} or g_{VS})

$V_{CH_4,ASF}$: Biomethane production measured on ASF (NL of CH₄)

$V_{CH_4,PF}$: Biomethane production measured on PF (NL of CH₄)

$m_{cod,ASF}$: Mass of COD measured in ASF (g_{O2})

$m_{cod,PF}$: Mass of COD measured in PF (g_{O2})

217

218 **3 Results and discussion**

219

220 3.1 Effects of pressure and L/S ratio on household biowaste (HBW) pretreatment

221 The effects of the operational conditions were investigated using this BW sample.

222 From preliminary experiments (not shown), three sets of conditions were compared: (i)

223 20 minutes soaking at a liquid to solid (L/S) ratio of 5 g of water per g of HBW dry solids

224 followed by pressing under 3 bars or (ii) 6 bars, (iii) 20 minutes soaking at a L/S ratio of

225 10 followed by pressing under 6 bars (Table 1).

226

227 Figure 2 shows the dynamics of extraction of ASF recorded during the pressing operation

228 of soaked HBW under the different operational conditions. Time 0 on the graphs showed

229 the time when pressure was applied. However, small volumes of solutions dripped out of

230 the cell by gravity before, and were included in the monitoring. The curves revealed 3

231 successive phases which resulted from the two processes involved in the operation. In a

232 first phase of 10 to 20 seconds corresponding to the filtration phase, the extracted mass

233 increased very sharply and linearly. According to the conventional theory of Ruth et al.

234 (1933a), which was verified by Vesilind (1994) on sludge material, filtration involves the

235 mobilization of free water under the effect of pressure and the convective transfer of the

236 suspended solids smaller than the size of the perforations in the filter.

237 A filtration cake consisting of the PF of HBW was thereby rapidly formed at the surface

238 of the filter disk by accumulation of the suspended solids larger than the disk openings

239 (see figure S2). This phenomenon rapidly reduced the permeability at the surface of the

240 disk and therefore the rate of ASF extraction slowed down over the next 1-2 minutes.

241 This was the second phase, corresponding to the formation and consolidation of the filter

cake. Particles in the filter cake were compressed, increasing furthermore the resistance to mass flow. Finally, the last phase of the curves observed in the following 18-19 minutes corresponded to the dewatering of the filtration cake. The extraction rate slowed down progressively until no significant extraction was recorded, indicating that all free water had then been removed from PF. At the completion of the assays (20 min), 69% of the initial mass of soaked HBW was extracted at 6 bars and only 49 % at 3 bars for the ratio of 5 g water.g⁻¹TS.

Increasing the pressure from 3 to 6 bars at a L/S ratio of 5 g_{water}.g⁻¹ TS increased the mass extracted in the filtration phase from about 20% to about 35%. of the initial mass (Fig. 2). However, the curves over the last phase (dewatering of filter cake) were very similar at 3 and 6 bars. Increasing the pressure also increased logically the TS content of PF from 26 to 41 % of the mass of the fraction (Table 2) since more aqueous solution was extracted. TS content of ASF also increased slightly from 7.1 to 9.3 % probably due to the extraction of dissolved and colloidal compounds.

Increasing the L/S ratio from 5 to 10 g_{water}.g⁻¹TS in the soaking operation of HBW increased furthermore the extraction during the filtration phase under 6 bars from 69% at L/S of 5 to 88% of the initial mass at L/S of 10. This result was explained by the higher proportion of water in the initial soaked HBW, which was easily extracted in the filtration phase. TS content in ASF was logically smaller at L/S of 10 as compared to L/S of 5 (Table 2). It can be noted that TS contents obtained here in the aqueous fraction (from 5 to 9.3 %, Table 2) were lower than reported in similar studies done on urban biowaste by other authors where TS contents as high as 18 to 31% were reported (Jank et al. 2015; do Carmo Precci Lopes et al. 2019; Micolucci et al. 2015b). This parameter however is strongly modified by the experimental conditions, which vary from one study to another.

For example, do Carmo Precci Lopes et al. (2019) studied dry press separation with 12 mm openings, whereas Jank et al. (2015) used filters with 8 mm openings.

The mass proportions of each fraction produced from HBW were calculated by equations 1 and 1' (TS) and 2 and 2' (VS). Results are shown in Fig. 3.

Fig. 3a showed that the major part of the initial soaked waste mass was collected in ASF. This was logical since this fraction received all the free water present in the initial soaked waste. Increasing the pressure during the pressing operation allowed to extract more free water and thereby increased the mass proportion of ASF. Increasing the liquid to solid ratio in the soaking phase had the same effect for the same reason. Using a similar approach as in this study, Jank et al. (2015) reported almost 85% ww extraction in the aqueous fraction with a filtration disk perforated with 8 mm holes. Similar results were reported by Novarino and Zanetti (2012) with extruded HBW at 8 mm holes. Hansen et al. (2007) reported lower values with 55% to 64% ww extraction using an not-optimized industrial screw press process.

Figs. 3b and c however showed that the dry matter (total solids, TS) initially present in the soaked BW was collected mostly in PF. The organic matter (VS) followed the same pattern as the total solids. Increasing the pressure from 3 to 6 bars in the pressing operation or the L/S ratio in the soaking phase was found to increase the proportion of the VS extracted into PF. This result was probably due to a stronger extraction of particulate organic matter from the soaked waste into ASF by the increased amounts of free water extracted into ASF under these conditions.

3.2 Pretreatment of the four biowaste by soaking at 10 g_w.g_{TS}⁻¹ followed by pressing at 6 bars)

291
292 *3.2.1 Total mass, total solids and volatile solids distribution between aqueous slurry and*
293 *particulate fraction*
294

295 Figure 4 shows the dynamics of the extraction of ASF recorded in the operation of
296 pressing of the four soaked BW investigated.

297
298 The curves obtained with all waste samples exhibited a very sharp slope at the very
299 beginning of the treatment, corresponding to the first phase, or filtration phase, discussed
300 above, and a flat slope in the last phase corresponding to the filter cake dewatering
301 process. The second phase showed however two distinct patterns. With GBW and SBW
302 samples, phase 2 was almost not observed, whereas it occurred over 1 to 2 minutes with
303 RBW and HBW samples. This result was attributed at different characteristics in the
304 formation and consolidation of the filter cake. GBW probably generated a more porous
305 and less compressible filter cake than RBW, SBW and HBW, due to its higher contents in
306 lignocellulosic organic compounds. It was therefore probably more rapidly stable,
307 resulting in a very short phase 2. At the end of the treatment, 76% (GBW) to 90% (SBW)
308 of the initial mass of soaked waste was collected in ASF.

309
310 Figure 5 a, b and c shows the distribution of the overall mass, dry mass (TS) and organic
311 dry mass (VS) of the respective initial soaked waste between the two fractions produced.
312 TS concentrations in the fractions are given in Table 3.

313 Figure 5 revealed relatively similar results for SBW, HBW and RBW samples. For these
314 samples, 86 to 92 % of the initial wet mass of soaked waste was collected in ASF, and 39
315 to 52 % of TS or VS were collected in PF. GBW however exhibited a distinct behavior,

with more than 90 % of TS or VS collected in PF. This observation suggested that the biochemical composition of the waste played a strong role. In a previous article, Moretti et al. (2020) reported that GBW sample was composed of lignocellulosic materials at up to 82% of VS. These biopolymers are very stable, recalcitrant, and poorly soluble. They were therefore collected almost quantitatively in the PF. In contrast, the other BW studied here mostly contained food products, with a higher proportion of less recalcitrant, more soluble compounds. Part of the TS initially present in the BW were therefore degraded and leached during soaking, and therefore smaller proportions were collected in PF.

3.2.2 Distribution of methane potential in the 2 fractions obtained from each biowaste

Table 4 shows the results from the BMP determinations of the fractions produced from the pretreatment of each BW. Figure 6 illustrates the time course of methane production recorded during the BMP assays.

Biogas production from either fraction obtained from HBW, RBW and SBW pretreatment followed first order kinetics. No inhibition was observed. Methane productions from ASF of these 3 BW were found in the same order of magnitude, ranging from 408 to 423 $\text{NL}_{\text{CH}_4} \cdot \text{g}^{-1} \text{vs}$. These results were relatively close to previously published data. Micolucci et al. (2015b) obtained a production of 470 $\text{NL}_{\text{CH}_4} \cdot \text{g}^{-1} \text{vs}$ from the anaerobic digestion of pressed HBW using a mesophilic digester. Jank et al. (2015) reported slightly smaller methane potentials of 360 $\text{NL}_{\text{CH}_4} \cdot \text{g}^{-1} \text{vs}$ on pressed biowaste using the BMP protocol. The differences observed may be attributed to the different origins of the waste materials and the different experimental protocols used.

The kinetic constants determined from the curves of biogas production were also very close for the 3 biowaste HBW, RBW and SBW, ranging between 0.35 and 0.40 d^{-1} . These observations suggested that the aqueous slurry fractions from these BW contained readily biodegradable compounds in the forms of dissolved molecules, colloids and small particles.

RBW and SBW samples exhibited similar methane productions from their respective PF and ASF (between 408 and 455 $\text{NL}_{\text{CH}_4} \cdot \text{g}^{-1} \text{vs}$), indicating that the soaking operation was not efficient enough in extracting the readily biodegradable constituents from these BW into the ASF. The corresponding kinetic constants however were smaller in PF than in

ASF (0.26 and 0.28 d⁻¹ vs. 0.35 and 0.40 d⁻¹), indicating that the PF fractions contained more recalcitrant compounds than the corresponding ASF.

The efficiency of soaking operation was better with HBW, as shown by the lower BMP of PF ($269 \pm 21 \text{ NL}_{\text{CH}_4} \cdot \text{g}^{-1}_{\text{VS}}$) as compared to ASF ($416 \pm 13 \text{ NL}_{\text{CH}_4} \cdot \text{g}^{-1}_{\text{VS}}$). This result was attributed to the lignocellulosic contents, which was higher in HBW than in RBW and SBW samples as shown in Table S1, making PF of HBW less readily biodegradable than those of RBW and SBW.

Finally, GBW logically exhibited the smallest methane productions from each of its fractions ASF and PF (160 and $60 \text{ NL}_{\text{CH}_4} \cdot \text{g}^{-1}_{\text{VS}}$, respectively) as compared to all the other waste tested. The kinetic constant recorded on its PF was the smallest of all, confirming the recalcitrance of its ligno-cellulosic constituents. These observations were explained by the nature, origin and composition of this BW (see Table S1).

Figure 5d shows the proportion of the volume of methane produced by ASF and PF from each BW in the BMP assays, calculated by equations 3 and 3' (see materials & methods section). It also shows the distribution of COD between each fraction of each waste, calculated by eq. 4 and 4'.

The distributions of COD and methane production between the two fractions followed very similar patterns for each BW. The efficiency of the pretreatment operations in extracting as much as possible the methane potential of the BW materials into ASF was found to decrease in the following order: SBW (67%) > HBW (58%) $\approx$ RBW (57%) >> GBW (17%). This result was fairly well correlated to the distribution of volatile solids (VS, see Fig. 5c), and very correlated to the distribution of COD (Fig. 5d). Working on a

mixture of HBW and GBW, do Carmo Precci Lopes et al. (2019) reported a higher extraction of 82 % of methane potential in aqueous slurry.

In our study, the pretreatment operations allowed for SBW, HBW and RBW samples to concentrate in ASF between half (48%) and more than the 2/3rds (71%) of COD and methane productivity. In contrast, with GBW sample, the same operations conducted under the same conditions concentrated strongly these parameters in the PF (83-94%).

3.2.3 Composition of aqueous slurry fractions (ASF)

Table 5 gathers the results from the chemical analyses of ASF obtained from the pretreatment of each BW. It can be seen that the aqueous slurry fractions produced from HBW, RBW and SBW exhibited very similar chemical compositions, except for N-TKN concentration which was much smaller in ASF from HBW (177 mg/L) than those from RBW and SBW (around 1000 mg/L). This may be attributed to a higher proportion of meat products in RBW and SBW samples. The low pH (4.6 to 4.9) and relatively high VFA concentrations (from 3.3. to 5.1 g_{COD}.L⁻¹, mainly as lactic and acetic acids), suggested that some reactions of acidogenesis had already started within the biowaste during the collection time and the pretreatment of these BW, thereby confirming their high biodegradability.

The chemical composition of the ASF produced from GBW pretreatment was very different from that of the ASF obtained from the other waste. It confirmed that GBW was not adapted to anaerobic digestion.

3.2.4 Energy balance estimation

The results discussed above showed the possible added-value of implementing a wet-press pretreatment prior to anaerobic digestion of biowaste with low lignocellulosic content ($< 40\%$ of VS).

Further investigations at a larger scale and complementary economic considerations appear however necessary to define the optimal conditions of implementation. Although the nature and scale of the present study are actually not adapted to such estimations, efforts were made to propose a simplified evaluation of the energy balance of the system. Calculations are detailed in Table 6. The potential energy productions from anaerobic digestion of the aqueous slurry fractions were calculated for a unit mass of organic matter ($MJ.kg_{VS}^{-1}$) by multiplying the BMP values ($N_{LCH_4}.kg_{VS}^{-1}$) by the heat of combustion of methane (35.9 kJ / L). The energy needed for the wet-press pretreatment was evaluated in a simplified manner as the work developed to press the soaked waste under a constant pressure of 6 bars ($6 \cdot 10^5 \text{ Pa}$) over a volume variation ΔV equal to the difference between the initial and the final volume. In addition, an overall energy yield of 0.3 was considered between the electrical energy consumed and the work $-P \Delta V$ developed by the press. The energy consumed for soaking the waste was neglected.

Table 6 shows that the energy consumed to run the wet-press pretreatment was very small as compared to the energy potentially produced by anaerobic digestion ($>1\%$), with exception for GBW, where the recovered amount of VS in the slurry was very small and poorly degradable. In a first approach, it can therefore be considered that the implementation of the pre-treatment is energetically acceptable.

421

422 **4 Conclusions**

423 A wet mechanical pretreatment was designed to improve and facilitate methane
424 production from biowaste. The treatment consisted in separating a biodegradable aqueous
425 slurry fraction from a more recalcitrant particulate one. Pressing of soaked waste was
426 investigated and shown as a good approach according to the experimental results
427 obtained.

428 The extraction of the biodegradable organic matter into the aqueous slurry fractions ASF
429 was found better with the biowaste materials containing relatively little amounts of
430 lignocellulosic substances. The efficiency of the pretreatment operations in extracting
431 methane potential of the BW materials into ASF was found to decrease in the following
432 order: SBW (67%) > HBW (58%) $\approx$ RBW (57%) $\gg$ GBW (17%). The pretreatment
433 operations were therefore poorly efficient with respect to this criterion for waste material
434 with high lignocellulosic contents such as Green BW. Energy balance of the system was
435 highly positive considering energy production from biogas and energy demand for press-
436 filtration. Further investigations are in progress to scale up the results.

437 **Supplementary data**

438 E-supplementary data for this work can be found in e-version of this paper online

439

440 **Declarations**

441 Ethics approval and consent to participate

442 Not applicable

443 Consent for publication

444 Not applicable

445 Availability of data and materials

446 The datasets used and/or analysed during the current study are available from the
447 corresponding author on reasonable request.

448 Competing interests

449 The authors declare that they have no competing interests

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453

454 Authors' contributions

455 PM and MMO have conducted the experimental work, the analyses, most of data
456 interpretation and draft redaction of the article.

457 RB was the project coordinator, the major contributor to the design of experimental
458 work, and contributor to the redaction of the manuscript

459 PB and RG were major contributors in the research project and in the redaction of the
460 manuscript.

461 JMA and ABC contributed in redaction of the manuscript

462 All authors have read and approved the final manuscript.

463

464

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References

- Ariunbaatar J, Panico A, Esposito G, Pirozzi F, Lens PNL (2014) Pretreatment methods to enhance anaerobic digestion of organic solid waste. *Applied Energy* 123:143-156.
- Baird R, Bridgewater L (2017) Standard methods for the examination of water and wastewater. 23rd edition. Washington D.C. American Public Health Assoc.
- Bernstad A (2014) Household food waste separation behavior and the importance of convenience. *Waste Manage* 34:1317-1323.
- Braguglia C, Gallipoli A, Gianico A, Pagliaccia P (2018) Anaerobic bioconversion of food waste into energy : A critical review. *Bioresour Technol* 248:37-56
- Capson-Tojo G, Rouez M, Crest M, Steyer JM, Delgenes JP, Escudié R (2016) Food waste valorization via anaerobic processes: a review. *Environ Sci Biotechnol* 15:499-547.
- Do Carmo Precci Lopes A, Robra S, Müller W, Meirer M, Thumser F, Alessi A, Bockreis A (2019) Comparison of two mechanical pre-treatment systems for impurities reduction of source-separated BW. *Waste Manage* 100:66-74.
- Escamilla-Alvarado C, Poggi-Varaldo HM, Ponce-Noyola MT (2017) Bioenergy and bioproducts from municipal organic waste as alternative to landfilling: a comparative life cycle assessment with prospective application to Mexico. *Environ Sci Pollut Res* 24:25602–25617.
- European Environment Agency (2020) Bio-waste in Europe – turning challenges into opportunities. EEA Report 04/2020. ISSN 1977-8449.
- European Union (2008) Directive 2008/98/EC of the European Parliament and the Council of 19 November 2008 on Waste and Repealing Certain Directives. Official J of the European Union 22/11/2008.

499 European Union (2018) Directive 2018/851 of the European Parliament and the Council
 500 of 30 May 2018, amending Directive 2008/98/EC on waste. Official J of the European
 501 Union 14/06/2018.

502 Fisgativa H, Tremier A, Dabert P (2016) Characterizing the variability of food waste
 503 quality: A need for efficient valorisation through anaerobic digestion. *Waste Manage*
 504 50:264-274.

505 Fisgativa H, Tremier A, Le Roux S, Bureau C, Dabert P (2017) Understanding the
 506 anaerobic biodegradability of food waste: Relationship between the typological,
 507 biochemical and microbial characteristics. *J Environ Manage* 188:95-107.

508 Hansen TL, Jansen JLC, Davidsson A, Christensen TH (2007) Effects of pre-treatment
 509 technologies on quantity and quality of source-sorted municipal organic waste for
 510 biogas recovery. *Waste Manage* 27:398-405.

511 Holliger C, Alves M, Andrade D, Angelidaki I, Astals S, Baier U, Bougrier C, Buffière
 512 P, Carballa M, Wilde V, Ebertseder F, Fernández B, Ficara E, Fotidis I, Frigon JC ,
 513 de Laclos HF, Ghasimi DSM, Hack G, Hartel M, Heerenklage J, Horvath IS, Jenicek
 514 P, Koch K, Krautwald J, Lizasoain J, Liu J, Mosberger L, Nistor M, Oechsner H,
 515 Oliveira JV, Paterson M, Pauss A, Pommier S, Porqueddu I, Raposo F, Ribeiro T,
 516 Pfund FR, Strömberg S, Torrijos M, Eekert MV, Lier JV, Wedwitschka H, Wierinck
 517 I (2016) Towards a standardization of biomethane potential tests. *Water Science*
 518 *Technol* 74:2515-2522.

519 Jank A, Müller W, Schneider I, Gerke F, Bockreis A (2015) Waste Separation Press
 520 (WSP): A mechanical pretreatment option for organic waste from source separation.
 521 *Waste Manage* 39:71-77.

522 Micolucci F, Gottardo M, Malamis D, Bolzonella D, Pavan P, Cecchi F (2015a) Analysis
 523 of Meso/Thermo AD process applied to pressed biowaste. *Waste biomass valor*
 524 6:723–731.

525 Micolucci F, Gottardo M, Cavinato C, Pavan P, Bolzonella D (2015b) Mesophilic and
 526 thermophilic anaerobic digestion of the liquid fraction of pressed BW for high energy
 527 yields recovery. *Waste Manage* 48:227-235.

528 Moretti P, Morais de Araujo J, Borges de Castilhos A, Buffiere P, Gourdon R, Bayard R
 529 (2020) Characterization of municipal biowaste categories for their capacity to be
 530 converted into a feedstock aqueous slurry to produce methane by anaerobic digestion.
 531 *Sci Total Environ* 726:137084.

532 NF EN 14899 (2006) Characterization of waste - Sampling of waste materials -
 533 Framework for the preparation and application of a sampling plan.

534 Novarino D, Chiara Zanetti M (2012) Anaerobic digestion of extruded OFMSW.
 535 *Bioresour Technol* 104:44-50.

536 Pognani M, Barrena R, Font X, Sanchez A (2012) A complete mass balance of a complex
 537 combined anaerobic/aerobic municipal source-separated waste treatment plant. *Waste*
 538 *Manage* 32:799-805.

539 Rodríguez-Valderrama S, Escamilla-Alvarado C, Rivas-García P, Magnin JP, Alcalà-
 540 Rodríguez M, Garcia-Reyes RB (2020) Biorefinery concept comprising acid
 541 hydrolysis, dark fermentation, and anaerobic digestion for co-processing of fruit and
 542 vegetable wastes and corn stover. *Environ Sci Pollut Res* 27:28585–28596.

543 Ruth BF, Montillon GH, Motonna RE (1933a) Studies in filtration: I. Critical analysis of
 544 filtration theory. *Ind Eng Chem* 25:76-82.

545 Sidaine JM, Gass M (2013) État de l’art de la collecte séparée et de la gestion de proximité
 546 des biodéchets. *Awiplan ADEME*.

547 Vakalis S, Sotiropoulos K, Moustakas D, Malamis K, Vekkos M, Baratieri M (2017)
 548 Thermochemical valorization and characterization of household BW. J of Environ
 549 Manage 203:648-654.
 550 Vesilind PA (1994) The role of water in sludge dewatering. Water Environ Res 66:4-11.
 551 Weithmann N, Möller J, Löder M, Piehl S, Laforsch C, Freitag R (2018) Organic fertilizer
 552 as a vehicle for the entry of microplastic into the environment. Sci Advance
 553 4:eaap8060.

554 **Abbreviation List**

555	ASF	Aqueous Slurry Fraction
556	BMP	Biomethane Potential
557	BW	Biowaste
558	COD	Chemical Oxygen Demand
559	GBW	Green Biowaste
560	HBW	Household Biowaste
561	L/S	Liquid to Solid ratio
562	PF	Particulate Fraction
563	RBW	Restaurant Biowaste
564	SBW	Supermarket Biowaste
565	TS	Total Solid
566	VS	Volatile Solid
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Figures and Tables :

Figure 1 Flow chart of the pretreatment operations applied to each BW sample.

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576 **Figure 2** Time course of ASF extraction during the operation of pressing of soaked
 577 household BW (HBW) under different operational conditions: Soaking stage at L/S of 5
 578 $g_{\text{water}} \cdot g_{\text{TS}}^{-1}$ followed by pressing at 3 bars or 6 bars; soaking at L/S 10 followed by pressing
 579 under 6 bars.

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Figure 3 Total mass (a), total solids (b) and volatile solids (c) distributions between ASF and PF for HBW pretreatment under different experimental conditions. Calculations were done on the basis of 100 g ww / TS / VS of household BW (HBW) before pretreatment.

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588 **Figure 4** Time course of ASF extraction during the operation of pressing at 6 bars of the
 589 four BW previously soaked in water for 20 min at L/S ratio of 10 $\text{g}_{\text{water}} \cdot \text{g}_{\text{TS}}^{-1}$. HBW:
 590 Household BW; RBW: Restaurant BW; SBW: Supermarket BW; GBW: Green BW.

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595 **Figure 5** Distribution of Total mass (a), total solids (b), volatile solids (c), COD and
596 methane production (d) between ASF and PF produced from the operation of pressing at
597 6 bars of the 4 BW previously soaked in water for 20 min at a L/S ratio of $10 \text{ g}_{\text{water}} \cdot \text{g}_{\text{TS}}^{-1}$.
598 Supermarket BW (SBW), Household BW (HBW), Green BW (GBW) and Restaurant
599 BW (RBW).
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Figure 6 Production of methane of ASF and PF after pretreatment of 1 kgVS of GBW (a), HBW (b), SBW (c), RBW (d). Results are average values of BMP triplicate tests.

Table 1 Experimental conditions used for the pretreatment operations of the BW samples.

Test	BW	L/S Ratio	Pressure	Pressing time
reference	tested	$(g_{water}.g^{-1}_{TS})$	(bar)	(min)
3 bar 5 L/S	HBW	5	3	20
6 bar 5 L/S	HBW	5	6	20
	HBW			
6 bar 10 L/S	RBW	10	6	20
	SBW			
	GBW			

HBW: Household BW, RBW: Restaurant BW, SMBW: Supermarket BW, GBW: Green BW

Table 2 TS contents of ASF and PF obtained from household BW (HBW) pretreatment under different experimental conditions. Results are expressed in % of the dry mass of the respective fraction. The results are average values and standard deviation of triplicated analyses.

Experimental conditions			
<i>Fraction</i>	3 bar 5 L/S	6 bar 5 L/S	6 bar 10 L/S
Aqueous slurry (ASF)	7.1 ± 0.3	9.3 ± 0.6	5.0 ± 0.1
Particulate (PF)	26.0 ± 3.6	41.0 ± 3.5	32.2 ± 1.9

Table 3 TS contents of ASF and PF after the operation of pressing at 6 bars of the four BW previously soaked in water for 20 min at a L/S ratio of 10 g_{water} .g⁻¹_{TS} . Results are expressed in % of the mass of the respective fraction.

Biowaste				
<i>Fraction</i>	HBW	RBW	SBW	GBW
Aqueous slurry (ASF)	5.0 ± 0.1	6.0 ± 0.1	6.0 ± 0.1	0.9 ± 0.1
Particulate (PF)	32.2 ± 1.9	39.2 ± 0.8	42.8 ± 2.0	40.0 ± 0.6
<i>Supermarket BW (SBW), Household BW (HBW), Green BW (GBW) and Restaurant BW (RBW)</i>				

Table 4 BMP results (methane productions and kinetic constants) of ASF and PF produced from the operation of pressing at 6 bars of the four BW previously soaked in water for 20 min at a L/S ratio of 10 g_{water}.g_{TS}⁻¹. Results are average values of triplicated analyses.

	HBW	RBW	SBW	GBW
Aqueous slurry (ASF)				
BMP (NL _{CH₄} .g ⁻¹ _{VS})	416 ± 13	408 ± 12	423 ± 13	160 ± 10
k (d ⁻¹)	0.35 ± 0.07	0.40 ± 0.01	0.35 ± 0.04	0.38 ± 0.02
Particulate (PF)				
BMP (NL _{CH₄} .g ⁻¹ _{VS})	269 ± 21	455 ± 18	409 ± 28	60 ± 6
k (d ⁻¹)	0.32 ± 0.01	0.28 ± 0.09	0.26 ± 0.03	0.14 ± 0.03

Supermarket BW (SBW), Household BW (HBW), Green BW (GBW) and Restaurant BW (RBW)

**Calculated as the product of BMP value expressed in NL_{CH₄}.g⁻¹_{VS} by the heating value of methane 35.9 kJ/NL*

Table 5 Composition of the aqueous slurry fraction (ASF) produced from the operation of pressing at 6 bars of the four BW previously soaked in water for 20 min at a L/S ratio of 10 $\text{g}_{\text{water}} \cdot \text{g}_{\text{TS}}^{-1}$. The results are average values of triplicated analyses.

Parameter	Unit	HBW	RBW	SBW	GBW
pH	-	4.9 ± 0.1	4.6 ± 0.1	4.9 ± 0.2	6.3 ± 0.1
N-NH ₃	$\text{mg}_N \cdot \text{L}^{-1}$	69 ± 0.2	73 ± 0.7	93 ± 1.3	16 ± 0.1
N-TKN	$\text{mg}_N \cdot \text{L}^{-1}$	177 ± 21	977 ± 27	1012 ± 13	69 ± 2
COD	$\text{g}_{\text{COD}} \cdot \text{kg}_{\text{TS}}^{-1}$	1067 ± 3	1056 ± 3	1056 ± 2	610 ± 1
VFA	$\text{equivalent g}_{\text{COD}} \cdot \text{L}^{-1}$	4.5 ± 0.6	3.3 ± 0.4	5.1 ± 0.8	0.6 ± 0.1
	% of COD	8.5	5.3	8.1	9.4
WSC	$\text{equivalent g}_{\text{COD}} \cdot \text{L}^{-1}$	10.2 ± 1.2	3.3 ± 0.1	7.5 ± 0.8	>0.1
	% of COD	19.3	5.3	12.0	0.3

COD: Chemical Oxygen Demand; VFA: Volatile Fatty Acids; WSC: Water Soluble Carbohydrates; TKN: Total Kjeldahl Nitrogen. Supermarket BW (SBW), Household BW (HBW), Green BW (GBW) and Restaurant BW (RBW)

Table 6 Estimations of the energy balance of the wet pressing pretreatment in the process of anaerobic digestion of the 4 biowaste

	HBW	RBW	SBW	GBW
Potential energy production from aqueous slurry (ASF)				
BMP (NL _{CH4} .kg ⁻¹ _{VS})	416	408	423	160
Potential energy production* (kJ.kg ⁻¹ _{VS})	14930	14650	15150	5740
Estimated energy demand of wet pressing operation				
Wet pressing Pressure (in Pa)	6 10 ⁵	6 10 ⁵	6 10 ⁵	6 10 ⁵
Volume variation (in L)	0.43	0.45	0.45	0.41
Mass of sample in press (in g vs)	9.3	12.3	12.2	1.20
Energy yield of electrical press (dimensionless)	0.3	0.3	0.3	0.3
Estimated energy demand for pressing (kJ.kg ⁻¹ _{VS})	93.1	72.4	73.9	683
Estimated energy demand for pressing (% of energy produced)	0.6	0.5	0.5	12

*Calculated as the product of BMP value expressed in NL_{CH4}.g⁻¹_{VS} by the heating value of methane 35.9 kJ/NL