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### ► To cite this version:

Mariana Moreira de Oliveira, Paul Moretti, Carina Malinowsky, Rémy Bayard, Pierre Buffière, et al.. Mechanical pre-treatment of source-collected municipal biowaste prior to energy recovery by anaerobic digestion. *Chemosphere*, 2022, 292, pp.133376. 10.1016/j.chemosphere.2021.133376 . hal-03506975

**HAL Id: hal-03506975**

**<https://hal.science/hal-03506975v1>**

Submitted on 8 Jan 2024

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## **Mechanical pre-treatment of source-collected municipal biowaste prior to energy recovery by anaerobic digestion**

Mariana Moreira de Oliveira<sup>a,b\*</sup>, Paul Moretti<sup>a</sup>, Carina Malinowsky<sup>c</sup>, Rémy Bayard<sup>a</sup>,  
Pierre Buffière<sup>a</sup>, Armando Borges de Castilhos Júnior<sup>c</sup>, Joacio de Araujo Morais Júnior<sup>b</sup>,  
Gilson Barbosa Athayde Júnior<sup>b</sup>, Rémy Gourdon<sup>a</sup>.

<sup>a</sup> University Lyon, INSA Lyon, DEEP, EA7429, 69621 Villeurbanne, France

<sup>b</sup> Federal University of Paraíba, Department of Civil and Environmental Engineering.  
João Pessoa, CEP 58051-900, Paraíba State, Brazil

<sup>c</sup> Federal University of Santa Catarina, Department of Sanitary and Environmental  
Engineering, Florianópolis, CEP 88040-970, Santa Catarina State, Brazil

\*Corresponding author at: Federal University of Paraíba, Department of Civil and  
Environmental Engineering. João Pessoa, 58051-900, Paraíba State, Brazil  
E-mail address: [mariana.moreira-de-oliveira@insa-lyon.fr](mailto:mariana.moreira-de-oliveira@insa-lyon.fr) (M. M. de Oliveira).

1 **Mechanical pre-treatment of source-collected municipal biowaste prior to energy**  
2 **recovery by anaerobic digestion**

3

4 **Abstract**

5 Pre-treatments are usually necessary to prepare biowaste to anaerobic digestion. The major  
6 objectives are (i) to remove undesirable materials such as plastics and metals, which may  
7 contaminate the biowaste even if separated source-collection systems are implemented, and (ii)  
8 to extract the most readily biodegradable organic fractions from the waste stream. In this study,  
9 two wet mechanical pre-treatments, namely air-compressed press and worm screw press, were  
10 investigated on urban household biowaste. Two liquid to solid ratios were tested in each pre-  
11 treatment. Anaerobic digestion of pre-treated biowaste was studied by measuring their  
12 biomethane potentials and by controlled experiments in a continuously stirred-tank reactor with  
13 a feed load of  $3.5 \text{ gVS.L}^{-1}.\text{d}^{-1}$ . It was observed that increasing liquid to solid ratio in the pre-  
14 treatments allowed to increase the proportion of biodegradable organic matter extracted from  
15 the biowaste, up to  $949 \text{ gCOD.kgTS}^{-1}$  from household biowaste. The biomethane potentials of  
16 pre-treated waste were very high (up  $525 \text{ LCH}_4.\text{kgVS}^{-1}$ ) and COD ( $949 \text{ gCOD.kg}^{-1}\text{TS}$ ) from  
17 household biowaste. Anaerobic digestion in continuously stirred-tank reactor showed a very  
18 strong conversion of COD load (81%) and a high methane production up to  $345 \text{ LCH}_4.\text{kgVS}^{-1}$ .

19

20 **Keywords:** pre-treatment, press, anaerobic digestion, biowaste, biomethane potential.

21

22 **Abbreviations**

23 ASF - Aqueous Slurry Fraction

24 BMP- Biochemical Methane Potential

25 GC-TCD - Gas Chromatography equipped with a Thermal Conductivity Detector

- 26 COD - Chemical Oxygen Demand
- 27 CSTR - Continuous Stirred-Tank Reactor
- 28 HBW- Household Biowaste
- 29 MSW - Municipal Solid Waste
- 30 NH<sub>4</sub>-N - ammonia nitrogen
- 31 OLR - Organic Loading Rates
- 32 PT#1 - Pre-treatments with air-compressed press
- 33 PT#2 - Pre-treatments with worm screw press
- 34 SF- Solid Fraction
- 35 SBW - Biowaste from Supermarkets
- 36 TKN - Total Kjeldahl Nitrogen
- 37 TS - Total Solids
- 38 VFA - Volatile Fatty Acids
- 39 VS - Volatile Solids
- 40 WSC - Water Soluble Carbohydrates

41

42 **1 Introduction**

43 Over the last decade, European and French legislations on waste management have  
44 been strongly pushing the prevention of waste production and the recovery of materials and /or  
45 energy from the waste stream, with a strong incentive to reduce drastically landfill disposal.  
46 The European Directive 2018/851/EC encourages European Union member states to implement  
47 systems for the specific collection of source-separated biowaste to facilitate the recovery of  
48 organic matter (European Union, 2018). In France, the energy transition for green growth act  
49 requires that municipalities take proper measures to achieve before 2025 specific collection and

50 resource recovery targets from municipal biowaste streams (LTECV, 2015). Despite the efforts  
51 to prevent waste generation, Municipal Solid Waste (MSW) production is still growing in most  
52 cities, following the growth of the population in urban areas (Fisgativa et al., 2016, Jank et al.,  
53 2017). MSW includes several categories of waste collected on the urban territories, including  
54 biowaste. Municipal biowaste consists of gardens and parks waste, food and kitchen waste from  
55 households, offices, restaurants, wholesalers, canteens, catering and retail units, and similar  
56 waste from food processing industry (European Union, 2018). Household waste represents the  
57 main category of biowaste, corresponding to more than 34% of the mass of MSW generated in  
58 Europe in 2017 (European Environmental Agency, 2020).

59       Among the processes available to recover materials and/or energy from biowaste,  
60 anaerobic digestion appears as a very attractive solution (Shahriari et al., 2012; Cesaro and  
61 Belgiorno, 2014). It has a low production of biosolids, low energy consumption, and high rates  
62 of methane production. Anaerobic digestion of biowaste is also considered a good solution to  
63 reduce landfill disposal while producing a renewable energy vector and an organic fertilizer  
64 (Novarino and Zanetti, 2012; Fisgativa et al., 2016).

65       The efficiency of anaerobic digestion depends, however, on the composition and  
66 characteristics of biowaste, since biochemical constituents are more biodegradable into  
67 methane than others (Bayard et al., 2016, Komilis et al., 2017). In most cities around the world,  
68 biowaste streams are currently collecting mixed with other non-biodegradable materials such  
69 as plastics, inorganic components, and ligno-cellulosic fractions which are rather recalcitrant  
70 (Saint-Joly et al., 2000; Fisgativa et al., 2017). The presence of these impurities in biowaste can  
71 cause technical problems in the anaerobic digestion process and depreciate the quality of the  
72 biogas and/or the residual digestate, which can lead to environmental impacts (Hartmann et al.,  
73 2004, Naroznova et al., 2016). Therefore, most of the organic waste substrates need to be pre-  
74 treated mechanically to remove impurities and make biodegradable matter more easily available  
75 (Jank et al., 2017, Lopes et al., 2019).

76 In a previous study, press separation was shown to generate a readily biodegradable  
77 aqueous slurry with a high biomethane potential (Micolucci et al. 2015, Lopes et al. 2019).  
78 Thus, higher values of biogas production rate in the aqueous slurry fraction are expected when  
79 compared to the particulate fraction due to a significantly higher content of ligno-cellulosic  
80 fibres and lower biodegradability (Hansen et al., 2007, Jank et al., 2015). The distribution of  
81 the waste between the two output fractions from the press process is affected by the pressure  
82 (Moretti et al., 2020, Moretti et al., 2021). Many works used press technologies to study specific  
83 fractions of interest in the anaerobic digestion process (Nayono et al., 2010a, Micolucci et al.,  
84 2015, Micolucci et al., 2016, Lopes et al., 2019).

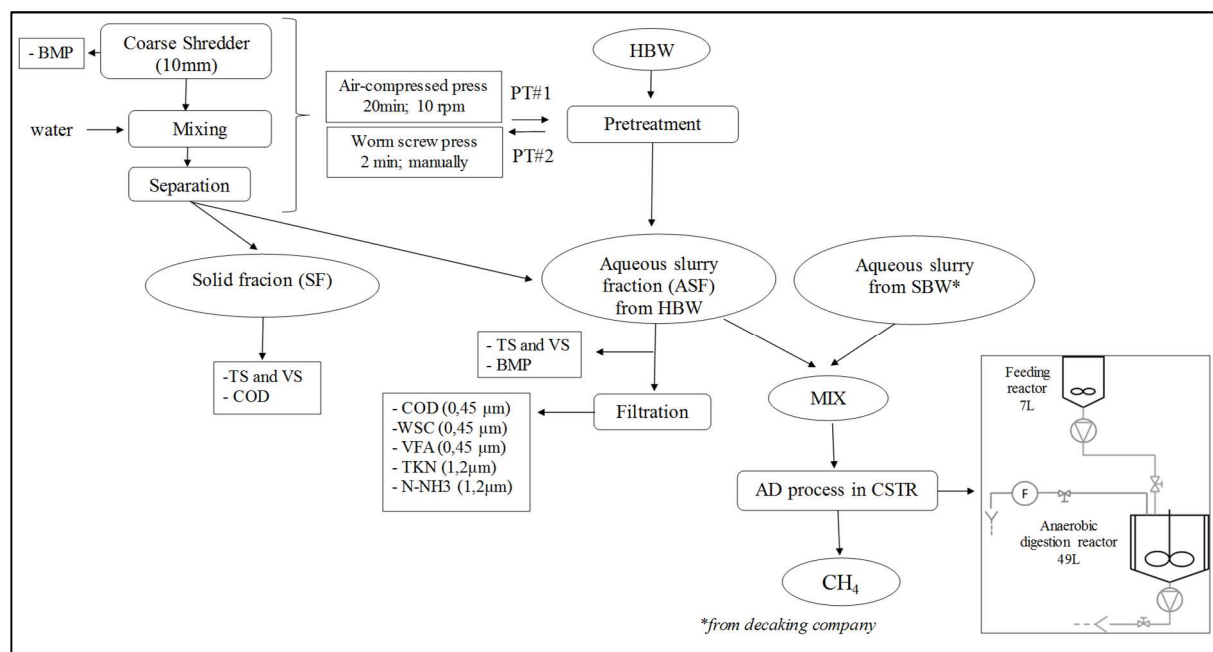
85 The objective of this work was to determine the conditions, in relation to dry matter  
86 content in the aqueous slurry fraction (ASF) as well as the percentage of dry matter extracted  
87 into this fraction, from the initial samples of biowaste. Two experimental devices of mechanical  
88 pressing, namely air-compressed press (PT#1) and worm screw press (PT#2), were compared.  
89 These two technologies were chosen because of their set up facilities and efficiency already  
90 proven in the literature. In a first step, two conditions of liquid/solid ratio were compared for  
91 the extraction of ASF from household biowaste samples (HBW). In a second step, the anaerobic  
92 digestion of a mixture of HBW ASF with a pulp extracted from supermarkets biowaste (SBW)  
93 samples was studied in a continuously stirred-tank reactor (CSTR), to assess the performance  
94 of methane production in dynamic conditions of treatment.

95

## 96 **2 Material and Methods**

97 The flowchart of the successive operations implemented on the biowaste samples is  
98 shown in Fig. 1.

99



**Fig. 1.** Flowchart of global scheme of pre-treatment and anaerobic digestion process

## 2.1 HBW pre-treatment

### 2.1.1 Pre-treatment procedures

Two pre-treatments technologies on HBW collected at source from a selective collection were studied, namely: air compressed press (PT#1) and worm screw press (PT#2). The experimental procedure shown in Fig. 1 was designed to extract as much as possible the soluble and colloidal organic matter from the biowaste in ASF (Moretti et al., 2020). The first operation of shredding was done to homogenize the samples in order to ensure the reproducibility and relevance of the results obtained. Each shredded sample was then soaked in de-ionized water. Two different liquid to solid ratios (L/S), 5 and 10 g of water per g of biowaste (dry mass), were tested. Then, liquid/solid separations were performed.

### 2.1.2 Air compressed press pre-treatment of HBW (PT#1)

Filtration-compression cells were used to produce ASF. Experiments were carried out in triplicates, using identical cylindrical stainless-steel cells of 0.46 L and 70 mm inner diameter.

117 The lower part of the cylinder cells was composed of a perforated stainless-steel disc with 3  
118 mm diameter holes. This technology allowed to work at high controlled pressure (up to 10 bars)  
119 but with a small load of biowaste. The biowaste suspension obtained after soaking was  
120 introduced into the cylinder cells and pressing 20 minutes at 6 bars.

121 Following the pressing operation, the ASF was collected from the bottom and the solid  
122 fraction (SF) part on the filter was removed from the cylinder cells.

123

### 124 *2.1.3 Worm screw press pre-treatment of HBW (PT#2)*

125 The biowaste suspension obtained after soaking in water was introduced into the worm  
126 screw press developed by the company Altantique industrie<sup>®</sup> to separate the liquid phase from  
127 the solid phase by the principle of separation by gravity and mechanical pressing. HBW were  
128 added at the feed inlet located at the beginning of the auger. The biowaste was carried by the  
129 auger to the outlet. Located at the outlet, a cork valve equipped with a hydraulic cylinder  
130 allowed compressing the biowaste as the material was added to the inlet. Under the pressure  
131 effect, the valve opened, allowing the exit of the solid material, constituting the remained SF.  
132 The pulp, resulting from the pressing, was collected through a cylindrical grid (2 mm diameter  
133 perforations) located along the entire length of the pressing chamber. Finally, with the use of a  
134 recovery tank, the ASF was collected at the level of the liquid outlet.

135 This technology has a high loading capacity with a batch of 50 L of soaked biowaste. A  
136 quantity of biowaste was set in the container, water was added to reach 25 kg of biowaste  
137 suspension, and then was mixed manually with a shovel for 2 min.

138

### 139 *2.1.4 Physico-chemical analyses of the ASF*

140 ASF obtained from HBW mechanical pre-treatments was analysed in triplicates. The total  
141 solid (TS) and volatile solid (VS) contents for all samples were determined through 105°C  
142 drying for 24h and calcination at 550°C during 2h (APHA, 2012). For mass balance, SF after



143 press was characterized, the samples were dried at 70 °C until constant weight (3 days) and  
144 crushed down to below 2 mm. Chemical oxygen demand (COD), total Kjeldahl nitrogen (TKN),  
145 and ammonia nitrogen (NH<sub>4</sub>-N) were analysed according to standard methods (Baird and  
146 Bridgewater 2017).

147 Volatile fatty acids (VFA) were measured by gas chromatography (Shimadzu Corp.)  
148 equipped with a HP-FFAP fused silica capillary column (30 m×0.25 mm Agilent  
149 Technologies), using H<sub>2</sub> as carrier gas and a flame ionization detector. Lactic acid, formic acid,  
150 and water soluble carbohydrates (WSC) were analysed by liquid chromatography (LC Module  
151 1 plus, Waters), equipped with a Supelcogel™ C-610 column (300 mm×7.8 mm, Sigma-  
152 Aldrich) with 0.1%v H<sub>3</sub>PO<sub>4</sub> solvent (flow rate of 0.5 mL.min<sup>-1</sup>). All liquid samples were filtered  
153 at 0.45 µm before analyses. All the analyses were performed in triplicates.

154

#### 155 *2.1.5 Biochemical methane potentials (BMP) analyses of ASF*

156 The BMP test was carried out following the guidelines reported by Holliger et al. (2016)  
157 thermostatically controlled at 35 °C. The BMP was performed on raw HBW, ASF, and ASF  
158 mix, destined for treatment in the reactor. For raw biowaste samples, 2 L glass vessels were  
159 used to guarantee the representativeness of the samples. For the ASF, 0.5 L glass vessels were  
160 used. The digested sludge from wastewater treatment plant in Lyon, France, was used as an  
161 inoculum, presenting TS 2.0-3.3%wt and VS 1.4-2.2%wt. The glass vessels were filled with  
162 the samples and inoculum, in the proportion of 2 g.g<sup>-1</sup>. Subsequently, the glass vessels were  
163 purged with N<sub>2</sub>/CO<sub>2</sub> (80/20% v/v) gas for about 2 minutes, sealed, and equilibrated at 35 °C. In  
164 addition, blanks composed of only inoculum and distilled water measured. This aimed to correct  
165 the recorded BMP from inoculum's residual methane production, and a positive which has  
166 already known BMPs for the validation of BMP test results. All the analyses were made in  
167 triplicates.

168 Biogas production was measured by monitoring the pressure in the flasks using a Digitron  
169 precision manometer. The gas composition was analysed using an Agilent 3000 micro gas  
170 chromatography equipped with a thermal conductivity detector (GC-TCD). Molsieve 5A (14 m  
171 length; pore size: 5 Å) and PoraPlot A (10 m length, 0.320 mm ID) columns were used as  
172 stationary phases for GC-TCD, using argon as a carrier gas. The tests were stopped when the  
173 biogas production in one day was less than 1% of the total volume of biogas produced (Holliger  
174 et al., 2016). The rate of methane production was determined from the net methane production  
175 and was used a first order kinetic model, as presented in Eq.1.

176

$$177 \quad V_{CH_4}(t) = V_{max}(1 - e^{-kt}) \quad (1)$$

178

179 Where:  $V_{CH_4}$  is the volume of methane produced at time  $t$ , in NL;  $t$  is the time, in days;  $V_{max}$  is  
180 the maximum volume of methane produced, in NL; and  $k$  is the first order kinetic constant.

181

#### 182 *2.1.6 Balance of TS, VS and COD between ASF and remaining SF*

183 Mass balance calculations were performed on the biowaste before treatment and on the  
184 ASF and SF obtained after separation. The content of TS, VS, and COD were analysed. The  
185 mass balance between the ASF and SF, of each respective biowaste sample, were calculated  
186 using the Eq.2 where  $i$  was defined as the proportions of TS, VS, and COD, and  $x$  corresponds  
187 the ASF and PF. It was expressed in % w/w.

188

$$189 \quad y_{i,x} = \frac{m_{i,x}}{m_{i,ASF} + m_{i,SF}} \cdot 100 \quad (2)$$

190

## 191 **2.2 Treatment of aqueous slurry mix by continuous anaerobic digestion process**

### 192 *2.2.1. Feedstock of aqueous slurry for anaerobic digestion: ASF mix.*

193 Anaerobic digestion assays were performed with a mix of aqueous slurry from HBW and  
194 SBW stream. HBW aqueous slurry was obtained from the worm screw press system PT#2 (see  
195 section 2.1.3). SBW pulp was collected from the company ECOVALIM dealing with the  
196 depackaging of mixed supermarket food waste in Rhône area (France). ASF mix was performed  
197 with 65% of HBW aqueous slurry and 35% of SBW aqueous slurry in humid mass. The choice  
198 of mixing HBW and SBW in that proportion was made considering the profile of production of  
199 biowastes of Grand Lyon urban territory: HBW and SBW were the main biowaste streams on  
200 territories, with green biowaste coming after, but this last being interesting for anaerobic  
201 digestion because of its high lignocellulosic contents (Moretti et al., 2020). The feedstock was  
202 packed in 2 L container and then freeze at -20 °C waiting for reactor feed.

203

#### 204 *2.2.2. Anaerobic digestion process and operational condition*

205 Anaerobic digestion process was performed in 49 L hermetic CSTR, mixed and  
206 thermostated at 35 °C. Fig. 1 illustrates the anaerobic digestion set up. Feedstock of ASF mix  
207 was introduced by feeding the digester of 7 L. Pump fed the anaerobic reactor each day at 0.10  
208 L.min<sup>-1</sup> during a feeding time depending on organic loading rate (OLR) (except weekend).  
209 Extraction was performed simultaneously to feeding to ensure constant reactor volume and  
210 continuous mode. Biogas output was on the top of the anaerobic reactor and a mass flowmeter  
211 recorded continuously biogas flow.

212 In the literature, OLR ranking is reported between 2 to 10 gVS.L<sup>-1</sup>.d<sup>-1</sup> with a hydraulic  
213 retention time up to 20 days (Nayono et al., 2010b, Micolucci et al., 2016, Komilis et al., 2017,  
214 Malinowsky et al., 2021). Considering these previous works, operational conditions were an  
215 OLR of 3.5 gVS.L<sup>-1</sup>.d<sup>-1</sup> associated with a hydraulic retention time of 38.6 days. Results herein  
216 presented were obtained during stabilized conditions of the process. The inoculum used for the  
217 mesophilic trials was digested sludge from the full-scale anaerobic digestion process of  
218 wastewater treatment plant, in Lyon, France. The pilot was maintained at an operating

219 temperature of 35 °C with OLR for two weeks to acclimatize the biomass to ASF mix. Once  
 220 the biomass was stabilized, the reactor was fed daily.

221

### 222 *2.2.3. Reactor monitoring*

223 The reactor was monitored to ensure correct digestion conditions and to determine reactor  
 224 efficiencies. Analysis methods are presented in section 2.1.4. TS, VS, COD soluble, and VFA  
 225 were determined three times a week on input and digestat. COD<sub>total</sub>, TKN, and WSC were  
 226 measured once a week. Biogas composition was also followed, with three samples a week  
 227 analysed by GC-TCD analysis. The pH, temperature, and gas flow were monitored by online  
 228 sensor measures connected to acquisition software.

229

## 230 **3 Results and Discussion**

### 231 *3.1 Extraction of digestible matter from HBW using different press systems*

232 Table 1 shows the TS and VS content for HBW pressed processed by the air-compressed  
 233 press (PT#1), or processed by the worm screw press (PT#2). For PT#1 system, two pulping L/S  
 234 ratios were tested to obtain a dry matter content of the ASF suitable for the anaerobic digestion  
 235 (between 4 and 10% of TS).

236

237 **Table 1.** TS and VS contents of aqueous slurry fraction (ASF) obtained from household  
 238 biowaste (HBW) pressed with air-compressed press PT#1 and pressed with worm screw press  
 239 PT#2. Results are expressed in % of the dry mass of the respective fraction

ASF	PT#1: HBW pressed with air-compressed Press		PT#2: HBW pressed with worm screw press	
	L/S 5	L/S 10	L/S 5	L/S 10
TS	7 ± 0.4%	5 ± 0.4%	8 ± 0.0%	4 ± 0.0%
VS	79 ± 1.0%	84 ± 1.0%	86 ± 0.0%	87 ± 1.0%

240

241 Increasing the L/S ratio decreased TS content in the ASF, for both press technologies.  
242 Few variations in VS concentration were observed. When analysing the performance of the two  
243 mechanical pre-treatments, it was observed that, comparing the same ratio in both pre-  
244 treatments, the TS values were similar. The VS values from worm screw press PT#2 were  
245 slightly higher than those found from air-compressed press (PT#1) (Table 1).

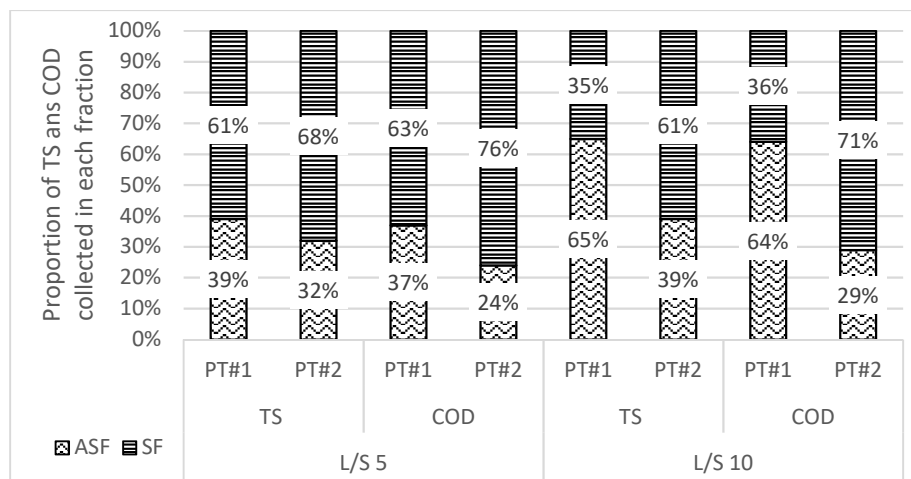
246 A wide range of TS value of ASF was found in literature, ranging from 10 to 42% (Hansen  
247 et al., 2007, Bernstad et al., 2013, Jank et al. 2015, Micolucci et al., 2015, Lopes et al., 2019).  
248 Similarly, Lopes et al. (2019) analysed, among other processes, the pressing of the mixture of  
249 food and garden waste from households with water (ratio of approximately 350 kg of water per  
250 1000 kg of biowaste) and obtained a value of 27.5% of TS for the aqueous fraction resulting  
251 from pressing. The highest TS value reported in the literature mentioned in this paragraph, when  
252 compared to the result of the present study, could be related to a higher cut-off point of the press  
253 of 12 mm compared to 3 and 2 mm in our case. This could mean that the cut-off point is a  
254 crucial parameter to promote TS concentration in ASF.

255 Fig. 2 presents the distribution of mass between ASF and SF, after the pre-treatment  
256 process for HBW in the two pre-treatments technologies at different L/S ratios.

257 TS initially present in the biowaste and water mixture was collected mostly in SF. With  
258 the water addition, *i.e.*, increasing the L/S ratio, the reduction on the proportion of the TS into  
259 SF was observed, and consequently an increase in the ASF, demonstrating a positive influence  
260 on TS extraction in this fraction. It has been observed that 32% of TS was extracted in liquid at  
261 L/S ratio of 5, while this proportion increased to 39% at L/S ratio of 10, for the worm screw  
262 press PT#2. The same phenomenon was also noted with air-compressed press PT #1 (39% to  
263 65%). These values are similar to those observed in the work by Hansen et al. (2007) that found  
264 the TS values between 23% w/w and 28% w/w into the biomass fraction (soft and wet waste,  
265 mainly organic waste) with the screw press from households waste. Micolucci et al. (2015)

266 found a slightly lower value, about 18% of TS, in wet fraction from a pilot scale press to pre-  
 267 treat collected biowaste. However, pressure values were not reported in these studies.

268



269

270 **Fig. 2.** Evolution of mass fraction of TS and COD at different L/S ratio: 5 and 10 gWater.gTS<sup>-1</sup>  
 271 <sup>1</sup> of HBW pressed with air-compressed press (PT#1) and HBW pressed worm screw press  
 272 (PT#2)

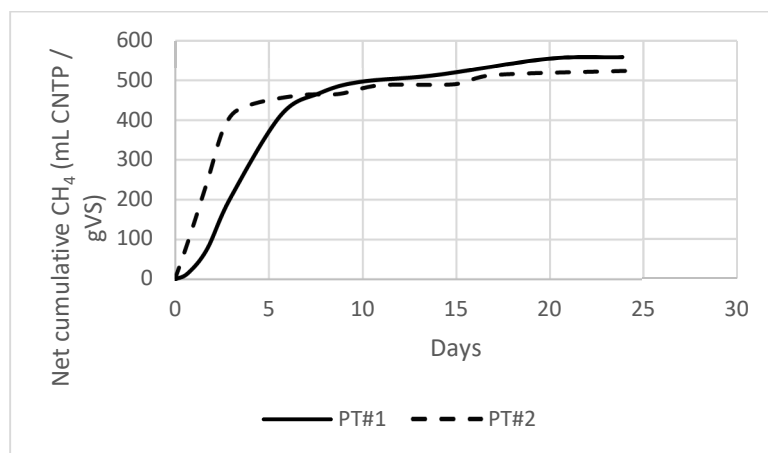
273

274 Similar behaviour was observed in the proportion of COD in the ASF. In both pre-  
 275 treatments, there was an increase in the proportion of COD in the ASF due to the increase in  
 276 the L/S ratio, from 37% to 64% for the PT#1 system and from 21% to 29% for the PT#2 system.  
 277 These facts could be explained by the higher extraction of particulate matter from ASF due to  
 278 the increase in the amount of free water. TS and VS results showed that water addition before  
 279 mixing decreases TS concentration of ASF but increases the total fraction of extracted TS.

280 According to the data discussed, as well as can be seen in Fig. 2, worm screw press process  
 281 PT#2 was less efficient than air compressed press PT#1 on TS and COD extraction, since PT#1  
 282 system was able to extract a higher percentage of these parameters in the ASF, by increasing  
 283 the L/S ratio with the addition of water. It could be explained by higher pressure extraction in  
 284 this pre-treatment process, about 6 bars, that involve higher strength to extract valuable matter  
 285 in ASF.

286 Fig. 3 presents cumulative production of methane during BMP test for ASF from air-  
 287 compressed press PT#1 and worm screw press PT#2. Methane production from the ASF from  
 288 HBW processing with PT#1 system ( $523.8 \text{ NLCH}_4.\text{kgVS}^{-1}$ ) and PT#2 system  
 289 ( $524.8 \text{ NLCH}_4.\text{kgVS}^{-1}$ ) obtained was similar. However, the kinetic constants were higher in  
 290 ASF from worm screw press PT#2 ( $0.57.\text{d}^{-1}$ ) when compared to ASF from air-compressed press  
 291 PT#1 ( $0.29.\text{d}^{-1}$ ). These results suggest less readily bioavailable compounds in the last fraction.  
 292 The values obtained are similar to those obtained by Alessi et al. (2020) of  $536 \text{ NLCH}_4.\text{kgVS}^{-1}$   
 293 <sup>1</sup> and slightly above those found in other studies: 360 to  $460 \text{ NLCH}_4.\text{kgVS}^{-1}$  (Hansen et al.,  
 294 2007; Jank, et al., 2015; Fisgativa et al., 2016).

295



296

297 **Fig. 3.** Evolution of net cumulative biomethane production through BMP test of ASF after PT#1  
 298 (air compressed press) and PT#2 (worm screw press) at L/S 5 ratio. Meaning of triplicated  
 299 assays

300

301 In order to feed the anaerobic digestion process to the next step, the choice of HBW pre-  
 302 treated was done considering the TS and VS contents present in ASF, the proportion of TS and  
 303 COD extract in ASF, the BMP production, and other variables, like the capability to produce  
 304 large quantity ASF and consume of water and air-compressed. Despite the higher values of TS  
 305 and COD extraction in the ASF in air compressed press PT#1 at the L/S ratio 10, in order to

306 feed the anaerobic digestion process, the worm screw press pre-treatment PT#2 at L/S of 5  
 307  $\text{gWater.gTS}^{-1}$  was chosen. The L/S 5 ratio was selected because of the interesting TS  
 308 concentration (8%) of ASF produced, with high BMP while minimizing water addition during  
 309 pre-treatment. Worm screw press PT#2 was preferred than air-compressed PT#1 because of its  
 310 capability to produce more quantity of ASF, higher kinetic constants, and no need of  
 311 compressed air. Table 2 presents characterization of ASF from HBW destined for anaerobic  
 312 digestion after PT#2 system.

313

314 **Table 2.** Bio-chemical characterization of aqueous slurry fraction (ASF) from household  
 315 biowaste (HBW), supermarket biowaste (SBW) and feeding mix (ASF Mix)

Parameter	Unit	HBW ASF	SBW ASF	ASF Mix 65% HBW 35% SBW
pH	-	4.7	4.3	4.4
N-NH <sub>3</sub>	mgN.L <sup>-1</sup>	384.0 ± 38	594.0 ± 55	430.0 ± 41
N-TKN	mgN.kgTS <sup>-1</sup>	33.1 ± 4	22.9 ± 11	26.0 ± 2.0
COD	gCOD.kgTS <sup>-1</sup>	949.0 ± 48	1064.0 ± 22	1025.0 ± 41
VFA	equivalent gCOD.L <sup>-1</sup>	45.0 ± 5	43.4 ± 4	38.7 ± 5.0
WSC	equivalent gCOD.L <sup>-1</sup>	0.6 ± 0.01	2.2 ± 0.3	1.4 ± 0.2
BMP	LCH <sub>4</sub> .kgVS <sup>-1</sup>	525.0 ± 13	300.0*	340.0 ± 8.5

316 \* BMP calculated from BMP of HBW and ASF, considering VS of each fraction in the Mix.

317

### 318 **3.2 Treatment of ASF mix by anaerobic digestion in a reactor**

#### 319 **3.2.1 ASF mix composition: ASF of HBW and SBW**

320 ASF mix was produced using HBW and SBW aqueous slurry from mechanical pre-  
 321 treatment (see section 2.2.1). The results from the chemical analyses of ASF from HBW, SBW,  
 322 and ASF mix are presented in Table 2.

323 HBW and SBW showed similar concentrations of NTK and COD, however, the presence  
 324 of N-NH<sub>4</sub> and WSC was much higher in SBW. This suggests that SBW stream was charged in  
 325 sugar food and proteins highly available. The ASF mix is highly concentrated in COD with a



326 COD/N ratio around 39 gCOD.gN<sup>-1</sup>. A high proportion of VFA has been observed, representing  
327 39 gCOD.L<sup>-1</sup>, principally lactic acid, which indicated that acidogenesis was currently  
328 beginning. The ASF mix is highly biodegradable and ready to feed the anaerobic digestion  
329 process. A high concentration of VFA was measured in HBW of ASF traducing the beginning  
330 of acidogenesis of ASF.

331 Methane production from the ASF from HBW was high, about 525 LCH<sub>4</sub>.kgVS<sup>-1</sup>. BMP  
332 of SBW was not measured, but BMP of ASF mix, 340 LCH<sub>4</sub>.kgVS<sup>-1</sup>, resulting from SBW and  
333 HBW mixing involve a recalculated BMP of about 300 LCH<sub>4</sub>.kgVS<sup>-1</sup> considering VS of each  
334 fraction.

335

### 336 *3.2.2. Performance of mesophilic anaerobic digestion on ASF mix*

337 No drop of pH was observed during this run. After 10 days, VFA started increasing from  
338 day 10 and continued in this trend until day 18, when a decreasing trend started. The overall  
339 anaerobic process and methane production were not inhibited by the low concentrations of  
340 VFA, indicating that the VFAs produced by acidogens were simultaneously consumed by  
341 methanogens for biogas production (Mu et al., 2018). The system did not show any upset to its  
342 stability, thus indicating good robustness of the process also in transient conditions, a relevant  
343 aspect for the full-scale implementation of the process. As for pH, this remained constant,  
344 particularly in steady state conditions, with an average value around 7.7 because of the high  
345 buffer capacity of the system.

346 The average content of TS in the reactor remained almost constant with an average value  
347 of 38 gTS.kg<sup>-1</sup> (st. dev. ± 0.5) and an average VS content of 26 gVS.kg<sup>-1</sup> (st. dev. ± 0.7). The  
348 ratio between VS and TS showed an average value of 69%. It was also observed the capability  
349 of the system to convert the organic matter into biogas, with a conversion yield around 77%.

350 Ammonium in the liquid phase was around 1064 mg N-NH<sub>4</sub>.L<sup>-1</sup>, below the critical level  
351 for inhibition (Yenigün and Demirel, 2013). This value is sufficient to provide enough

352 bioavailable nitrogen for microbe growth without causing any inhibition (Nguyen et al., 2016).  
353 The value is related to the high degradation of the pressed biowaste liquid fraction that leads to  
354 a rapid release of ammonium in the liquid phase due to protein deterioration (Micolucci et al.,  
355 2014, Micolucci et al., 2016). The free ammonia nitrogen can be a significant inhibitor of CH<sub>4</sub>  
356 yield, as reported by Kong et al. (2016) that found that increasing from 30 to 140 mg.L<sup>-1</sup> resulted  
357 in a 25% methane yield reduction.

358 The soluble COD remained almost constant and relatively low with an average value of  
359 3.5 gCOD.L<sup>-1</sup>. Stable performance was observed all along the run with no decrease in  
360 performance. The process was able to consume a high rate of COD load with a yield of 81%.  
361 Kong et al (2016) investigated CH<sub>4</sub> yield from high-pressure extruded food waste, as well as  
362 the VS and soluble COD removal efficiencies. The VS removal efficiencies were similar,  
363 achieving 73%. The highest COD<sub>soluble</sub> removal efficiency obtained was 69%.

364 The average biogas composition was 57% CH<sub>4</sub> and the remaining part CO<sub>2</sub>. The average  
365 specific methane production was found around 345 LCH<sub>4</sub>.kgVS<sup>-1</sup>. Comparatively, BMP of ASF  
366 mix was about 340 LCH<sub>4</sub>.kgVS<sup>-1</sup>. This result highlights the capability of the system to convert  
367 organic matter into biogas with a high conversion yield. The value of methane production was  
368 similar to other researches that used the biowaste liquid fraction, Zang et al. (2013) obtained  
369 659 LCH<sub>4</sub>.kgVS<sup>-1</sup> and Micolucci et al. (2016) achieved an average biogas production of 79  
370 LCH<sub>4</sub>.kgVS<sup>-1</sup> with 66% methane content.

371 The mesophilic digestion process of ASF mix at OLR of 3.5 gVS.L<sup>-1</sup>.d<sup>-1</sup> in steady state  
372 condition showed great resilience. In comparison with previous results from Nayono et al.  
373 (2010a) and Micolucci et al. (2016) it can be suggested that at this applied OLR the reactor  
374 showed a high efficiency on VS removal during transient conditions.

375

#### 376 **4 Conclusions**

377           The production of urban biowaste cannot be reduced extensively since they result from  
378 basic human activities such as alimentary needs. The implementation of relevant strategies to  
379 avoid environmental impacts that these waste flows may generate, and in parallel recover  
380 energy and/or materials from them, is therefore particularly necessary. Many studies have  
381 already been published on biowaste composition and their possible treatments. The difficulties  
382 encountered to guarantee over the long term a stable quality of the biowaste materials collected  
383 in big cities suggest potential limitations in the objective of processing them into an organic  
384 soil conditioner. Energy recovery would be less demanding in that respect. In this perspective,  
385 anaerobic digestion is considered as a promising technique. Their pre-treatments before  
386 transformation into biomethane is however often regarded as limiting these developments. The  
387 present study investigated different physical pre-treatments of HBW and SBW prior to their  
388 anaerobic digestion for methane production.

389           Results showed that water addition and mixing before pressing allow to extract  
390 biodegradable organic compounds in high proportions from the biowaste samples. Both air-  
391 compressed press PT#1 and worm screw press PT#2 were found applicable to extract digestible  
392 organic fractions from urban biowaste. The most adapted solution depends on the mass flows  
393 of collected biowaste, the space available for the implementing the treatments, and TS  
394 concentration needed for subsequent anaerobic digestion. In case of severe space and/or time  
395 limitations, worm screw press PT#2 should be selected for its simplicity of use.

396           Continuous digestion of ASF mix was experimentally validated using a lab-scale CSTR  
397 bioreactor operated at  $3.5 \text{ gVS.L}^{-1}.\text{d}^{-1}$  OLR. The reactor showed high removal efficiency on VS  
398 (77%) and COD (81%), with a relatively high and stable methane content in the biogas  
399 produced (around 57% v/v).

400

401 **Formatting of funding sources**

402 French National Environmental Agency (ADEME) has funded the URBANBIOM project  
403 through the GRAINE-ADEME program 2016 (grant number n° 1806C0003).

404 This work was performed within the framework of the EUR H2O'Lyon (ANR-17-EURE-0018)  
405 of Université de Lyon (UdL), within the program "Investissements d'Avenir" operated by the  
406 French National Research Agency (ANR).

407

#### 408 **Acknowledgements**

409 The authors would like to thank the technical team of the DEEP laboratory who participated in  
410 the development of the analytical techniques used in this study: Ms. Nathalie Dumont and Mr.  
411 David Lebouil. We also thank our colleagues at the PROVADEMSE consultancy for their  
412 contribution to the selection and sampling of the biowaste studied in this research program. The  
413 authors would like to thank also the CAPES (*Coordenação de Aperfeiçoamento de Pessoal de*  
414 *Nível Superior*) for providing a doctoral scholarship.

415

#### 416 **References**

417 Alessi, A., Lopes, A. do C.P.L., Müller, W., Gerke, F., Robra, S., Bockreis, A., 2020.  
418 Mechanical separation of impurities in biowaste: Comparison of four different pretreatment  
419 systems. *Waste Manag.* 106, 12-20, doi: <https://doi.org/10.1016/j.wasman.2020.03.006>.

420

421 APHA, A. WEF., 2012. Standard methods for the examination of water and wastewater, 22nd  
422 Edition. Washington DC: American Public Health Association.

423

424 Baird R., Bridgewater L., 2017. Standard methods for the examination of water and wastewater,  
425 23rd Edition.. American Public Health Assoc, Washington D.C.

426

427 Bayard, R., Liu, X., Benbelkacem, H., Buffiere, P., Gourdon, R., 2016. Can Biomethane  
428 Potential (BMP) Be Predicted from Other Variables Such As Biochemical Composition in  
429 Lignocellulosic Biomass and Related Organic Residues? *Bioenerg. Res.* 9, 610-623, doi:  
430 <https://doi.org/10.1007/s12155-015-9701-3>.

431

432 Bernstad, A., Malmquist, L., Truedsson, C., la Cour Jansen, J., 2013. Need for improvements  
433 in physical pretreatment of source-separated household food waste. *Waste Manag.* 33(3), 746-  
434 54, doi: <https://doi.org/10.1016/j.wasman.2012.06.012>.

435

436 Cesaro, A., Belgiorno, V., 2014. Pretreatment methods to improve anaerobic biodegradability  
437 of organic municipal solid waste fractions. *Chem. Eng. J.* 240, 24-37, doi:  
438 <https://doi.org/10.1016/j.cej.2013.11.055>.

439

440 European Union, 2018. Directive 2018/851 of the European Parliament and the Council of 30  
441 May 2018, amending Directive 2008/98/EC on waste. *Official J of the European Union*  
442 14/06/2018.

443

444 European Environmental Agency, 2020. Bio-waste in Europe — turning challenges into  
445 opportunities. European Union. Luxembourg: Publications Office of the European Union.  
446 doi:10.2800/630938.

447

448 Fisgativa, H., Tremier, A., Dabert, P., 2016. Characterizing the variability of food waste quality:  
449 A need for efficient valorization through anaerobic digestion. *Waste Manag.* 50, 264-274, doi :  
450 <https://doi.org/10.1016/j.wasman.2016.01.041>.

451

452 Fisgativa, H., Tremier, A., Le Roux, S., Bureau, C., Dabert, P., 2017. Understanding the  
453 anaerobic biodegradability of food waste: Relationship between the typological, biochemical  
454 and microbial characteristics. *J. Environ. Manage.* 188, 95-107, doi :  
455 10.1016/j.jenvman.2016.11.058.

456

457 Hansen T.L., Jes la Cour J., Davidsson A., Christensen T.H., 2007. Effects of pretreatment  
458 technologies on quantity and quality of source-sorted municipal organic waste for biogas  
459 recovery. *Waste Manag.* 27 (3), 398-405, doi: <https://doi.org/10.1016/j.wasman.2006.02.014>.

460

461 Hartmann, H., Møller, H.B., Ahring, B.K., 2004. Efficiency of the anaerobic treatment of the  
462 organic fraction of municipal solid waste: collection and pretreatment. *Waste Manag Res.* 2 (1),  
463 35–41, doi: 10.1177/0734242X04042748.

464

465 Holliger, C., Alves, M., Andrade, D., Angelidaki, I., Astals, S., Baier, U., ... & Wierinck, I.,  
466 2016. Towards a standardization of biomethane potential tests. *Water Sci. Technol.* 74 (11),  
467 2515–2522, doi: <https://doi.org/10.2166/wst.2016.336>.

468

469 Jank, A., Müller, W., Schneider, I., Gerke, F., Bockreis, A., 2015. Waste Separation Press  
470 (WSP): A mechanical pretreatment option for organic waste from source separation. *Waste*  
471 *Manag.* 39, 71-77, doi: <https://doi.org/10.1016/j.wasman.2015.02.024>.

472

473 Jank, A., Muller, W., Waldhuber, S., Gerke, F., Ebner, C., Bockreis, A., 2017. Hydrocyclones  
474 for the separation of impurities in pretreated biowaste. *Waste Manag.* 64, 12-19, doi:  
475 10.1016/j.wasman.2017.03.001.

476

477 Komilis D., Barrena R., Lora Grando R., Vogiatzi V., Sanchez A., Font X., 2017. A state of the  
478 art literature review on anaerobic digestion of food waste: influential operating parameters on  
479 methane yield. *Rev Environ Sci Biotechnol.* 16, 347–360, doi : [https://doi.org/10.1007/s11157-](https://doi.org/10.1007/s11157-017-9428-z)  
480 [017-9428-z](https://doi.org/10.1007/s11157-017-9428-z).

481

482 Kong, X., Xu, S., Liu, J., Li, H., Zhao, K., & He, L., 2016. Enhancing anaerobic digestion of  
483 high-pressure extruded food waste by inoculum optimization. *Journal of environmental*  
484 *management*, 166, 31-37, doi: [10.1016/j.jenvman.2015.10.002](https://doi.org/10.1016/j.jenvman.2015.10.002).

485

486 LTECV - Loi No 2015-992 du 17 Août 2015 Relative à La Transition Énergétique Pour La  
487 Croissance Verte. LTECV 2015, No. DEVX1413992L, 76.

488

489 Lopes, A., Robra, S., Müller, W., Meirer, M., Thumser, F., Alessi, A., Bockreis, A., 2019.  
490 Comparison of two mechanical pretreatment systems for impurities reduction of source-  
491 separated biowaste. *Waste Manag.* 100, 66-74, doi:  
492 <https://doi.org/10.1016/j.wasman.2019.09.003>.

493

494 Malinowsky, C., Nadaleti, W., Debiassi, L. R., Moreira, A. J. G., Bayard, R., Castilhos Junior,  
495 A., B. de, 2021. Start-up phase optimization of two-phase anaerobic digestion of food waste:  
496 Effects of organic loading rate and hydraulic retention time. *J. Environ. Manage.* 296, 113064,  
497 doi: <https://doi.org/10.1016/j.jenvman.2021.113064>.

498

499 Micolucci, F., Gottardo, M., Bolzonella, D., & Pavan, P., 2014. Automatic process control for  
500 stable bio-hythane production in two-phase thermophilic anaerobic digestion of food  
501 waste. *International Journal of Hydrogen Energy*, 39(31), 17563-17572, doi:  
502 [10.1016/j.ijhydene.2014.08.136](https://doi.org/10.1016/j.ijhydene.2014.08.136).

503

504 Micolucci, F., Gottardo, M., Malamis, D., Bolzonella, D., Pavan, P., Cecchi, F., 2015. Analysis  
505 of meso/thermo anaerobic digestion process applied to pressed biowaste. *Waste Biomass*  
506 *Valorization*. 6, 723–731, doi: 10.1007/s12649-015-9407-y.

507

508 Micolucci, F., Gottardo, M., Cavinato, C., Pavan, P., & Bolzonella, D., 2016. Mesophilic and  
509 thermophilic anaerobic digestion of the liquid fraction of pressed biowaste for high energy  
510 yields recovery. *Waste Manag.* 48, 227-235, doi:  
511 <https://doi.org/10.1016/j.wasman.2015.09.031>.

512

513 Moretti P., De Araujo J.M., De Castilhos Jr, A.B., Buffière, P., Gourdon, R., Bayard R., 2020.  
514 Characterization of municipal biowaste categories for their capacity to be converted into a  
515 feedstock aqueous slurry to produce methane by anaerobic digestion. *Sci. Total Environ.* 716,  
516 137084, doi: <https://doi.org/10.1016/j.scitotenv.2020.137084>.

517

518 Moretti, P., de Oliveira, M. M., Bayard, R., Buffiere, P., de Araujo, J. M., de Castilhos Jr., A.  
519 B., Gourdon, R., 2021. Mechanical Pretreatment of Municipal Biowaste to Produce an Aqueous  
520 Slurry Dedicated to Anaerobic Digestion. *Environ. Sci. Pollut. Res.* 28, 20586-20597, doi :  
521 <https://doi.org/10.1007/s11356-020-11836-3>.

522

523 Mu, L., Zhang, L., Zhu, K., Ma, J., & Li, A., 2018. Semi-continuous anaerobic digestion of  
524 extruded OFMSW: Process performance and energetics evaluation. *Bioresource*  
525 *technology*, 247, 103-115, doi: 10.1016/j.biortech.2017.09.085.

526



527 Nayono, S.E, Winter, J., Gallert, C., 2010a. Anaerobic digestion of pressed off leachate from  
528 the organic fraction of municipal solid waste. *Waste Manag.* 30, 1828-1833, doi:  
529 <https://doi.org/10.1016/j.wasman.2009.09.019>.

530

531 Nayono, S. E., Gallert, C., Winter, J., 2010b. Co-Digestion of Press Water and Food Waste in  
532 a Biowaste Digester for Improvement of Biogas Production. *Bioresour. Technol.* 101, 6998-  
533 7004, doi: <https://doi.org/10.1016/j.biortech.2010.03.123>.

534

535 Naroznova, I., Møller, J., Larsen, B., Scheutz, C., 2016. Evaluation of a new pulping technology  
536 for pretreating source-separated organic household waste prior to anaerobic digestion. *Waste*  
537 *Manag.* 50, doi: 65-74, [10.1016/j.wasman.2016.01.042](https://doi.org/10.1016/j.wasman.2016.01.042).

538

539 Nguyen, D. D., Chang, S. W., Jeong, S. Y., Jeung, J., Kim, S., Guo, W., & Ngo, H. H., 2016.  
540 Dry thermophilic semi-continuous anaerobic digestion of food waste: Performance evaluation,  
541 modified Gompertz model analysis, and energy balance. *Energy Conversion and*  
542 *Management*, 128, 203-210, doi: <https://doi.org/10.1016/j.enconman.2016.09.066>.

543

544 Novarino, D., Zanetti, M. C., 2012. Anaerobic digestion of extruded OFMSW. *Bioresour.*  
545 *Technol.* 104, 44-50, doi: <https://doi.org/10.1016/j.biortech.2011.10.001>.

546

547 Saint-Joly, C., Desbois, S., Lotti, J.P., 2000. Determinant impact of waste collection and  
548 composition on anaerobic digestion performance: industrial results. *Water Sci. Technol.* 43,  
549 291-297, doi: [10.2166/wst.2000.0083](https://doi.org/10.2166/wst.2000.0083).

550

551 Shahriari, H., Warith, M., Hamoda, M., Kennedy, K.J., 2012. Anaerobic digestion of organic  
552 fraction of municipal solid waste combining two pretreatment modalities, high temperature

553 microwave and hydrogen peroxide. Waste Manag. 32, 41-52, doi :  
554 <https://doi.org/10.1016/j.wasman.2011.08.012>.

555

556 Zhang, C., Su, H., & Tan, T., 2013. Batch and semi-continuous anaerobic digestion of food  
557 waste in a dual solid–liquid system. Bioresource Technology, 145, 10-16, doi:  
558 <https://doi.org/10.1016/j.biortech.2013.03.030>.

559

560 Yenigün, O., Demirel, B., 2013. Ammonia inhibition in anaerobic digestion: a review. Process  
561 Biochem, 48, 5-6, doi: <https://doi.org/10.1016/j.procbio.2013.04.012>.

562

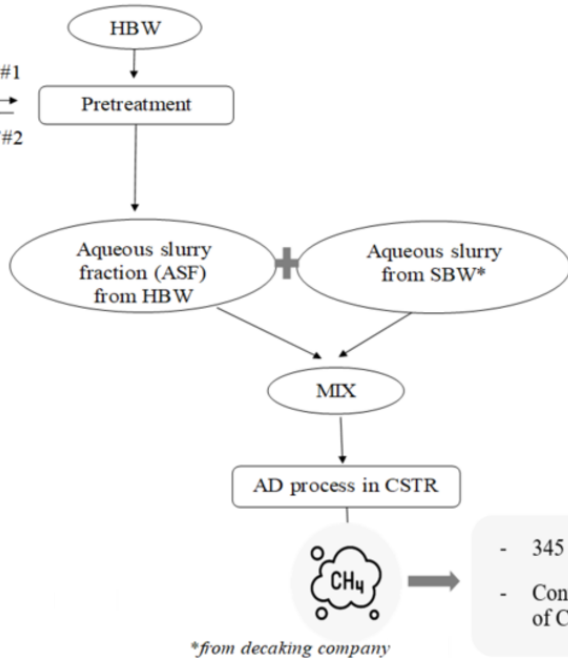
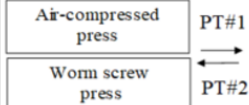
### 563 **List of the figure captions**

564 Fig. 1. Flowchart of global scheme of pre-treatment and anaerobic digestion process

565 Fig. 2. Evolution of mass fraction of TS and COD at different L/S ratio: 5 and 10 gWater.gTS<sup>-1</sup>  
566 of HBW pressed with air-compressed press (PT#1) and HBW pressed worm screw press  
567 (PT#2)

568 Fig. 3. Evolution of net cumulative biomethane production through BMP test of ASF after PT#1  
569 (air compressed press) and PT#2 (worm screw press) at L/S 5 ratio. Meaning of triplicated  
570 assays

- BMPs (up 525 LCH<sub>4</sub>.kgVS<sup>-1</sup>)
- COD (949 gCOD.kg-1TS)



- 345 LCH<sub>4</sub>.kgVS<sup>-1</sup>
- Convert a high fraction of COD load (81%)

\*from decaking company

