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Characterization of municipal biowaste categories for their capacity to be converted into a feedstock aqueous slurry to produce methane by anaerobic digestion

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Abstract

Biowaste material is a good candidate for the production of energy in urban territories. The presence of undesirable or constituents mixed with the biowaste collected by municipalities makes it difficult to recycle organic matter of sufficient quality for agricultural uses. Methane production is particularly attractive for energy recovery notably because this energy vector can be distributed using the grid already in place for natural gas in many cities. Depending on the origin and biochemical composition of biowaste, methane can be produced using thermochemical (gasification then syngas methanation) or biological processes (anaerobic digestion). The objective of this work was to characterize the ability of biowaste to be used as a feedstock for anaerobic digestion. Based on considerations such as the quantities produced and the availability, four categories of biowaste produced in the city of Lyon were identified as potential key resources: Garden biowaste (GBW), Restauration biowaste (RBW), Household biowaste (HBW) and Supermarkets biowaste (SMBW). Representative samples were taken from the sites of production and analyzed for parameters including biomethane potential (BMP). Each sample was then fractionated by leaching and the distribution of the BMP between the particulate fraction and the readily soluble fraction was assessed. GBW organic matter exhibited high hemicellulose content (over 81% of VS) and a low BMP which was very poorly distributed into its soluble fraction (2 NL.kg_{TS}⁻¹). RBW, HBW and SMBW showed a much higher BMP with a strong distribution in the soluble fraction (100 NL.kg_{TS}⁻¹). Plastic materials were found to account for up to 40% of the mass of SMBW sample. Altogether, GBW was identified as non-favorable for anaerobic digestion and recommended rather for thermochemical conversion. HBW, RBW and SMBW revealed adapted to anaerobic. Pulping was shown to be applicable in order to convert the 3 biowaste materials into a pumpable slurry with high biomethane potential.

Keywords: Biowaste, food waste, municipal waste, methane, anaerobic digestion, characterization, orientation, pulping, leaching, conditioning, pretreatment.

1- Introduction

Despite the growing efforts made at different levels to reduce municipal solid waste (MSW) production, minimize landfilling and increase resource or energy recovery from waste, (EU Directive 2008/98) the production of MSW is either still growing in most European cities and elsewhere around the world (Fisgativa *et al.*, 2016; Braguglia *et al.*, 2018).

Municipal solid waste (MSW) includes several categories of waste which are produced on urban territories and collected by the municipalities. They originate from households, offices, workshops, markets, etc. Biowaste is a specific category of organic waste defined in the Waste Framework European Directive (European Union, 2008). Biowaste includes garden and park waste, food products and kitchen waste from households, restaurants and supermarkets, and similar waste from food processing plants. Depending on their nature and origins, biowaste may contain (i) various proportions of readily biodegradable organic constituents such as simple sugars, starch, proteins and lipids, (ii) more recalcitrant biopolymers such as lingo-cellulosic polymers, and (iii) undesirable fraction such plastic, package and inorganic materials.

Due to their biological origins and specific characteristics, biowaste materials are alternative resources for the production of compost, nutrients, fuels or heat. Selective collection of biowaste is therefore developing in several European cities (Hansen *et al.*, 2007; Bernstad *et al.*, 2014; Sidaine and Gass, 2013) to optimize this approach and at the same time divert biodegradable organic waste from landfilling and incineration (IEA Bioenergy, 2013). In France, the Energy Transition for Green Growth Act (TECV, 2015) has implemented a series of rules in agreement with the European Directives. Source-separated collection of biowaste and the development of a public service to conduct the proper recovery operations will be mandatory in 2025. Today however, the efficiency of source-separated collection still remains poor in many medium and large size cities. The environmental quality of collected biowaste flows is thereby affected by the presence of several undesirable fractions, including hazardous domestic waste. Material recovery strategies such as the production of soil conditioner for agricultural purposes therefore appears difficult to implement if a safe manner. Energy recovery processes are less demanding in terms of biowaste quality, and therefore seem more adapted to urban biowaste.

Several strategies are potentially available to recover energy from biowaste. Among them, methane production appears as the most attractive. Methane gas can be produced from all types of biowaste using appropriate technologies of conversion. It can be easily separated and collected from waste due to its gaseous form and low solubility in water, and it offers several possible energy outputs. Injection of methane into the urban gas grid is an interesting option since dense grids are already in place in most cities for natural gas distribution. France has set an objective of the injection of 10% of methane from renewable sources by 2030, and the French Environmental Protection Agency (ADEME) is calling for more than 55% of renewable gas in French consumption by 2050 (ADEME, 2018).

Mature technologies are available for methane production from biowaste. Anaerobic digestion (AD) is adapted to humid and readily biodegradable biowaste fractions. In urban areas, AD has been developed mainly for the treatment of residual municipal solid waste (RMSW) produced from mechanical-biological pre-treatment (MBT) facilities (JUNIER, 2005). Thermochemical processes are more adapted to more recalcitrant (less biodegradable)

89 biowaste fractions (Vakalis et al., 2017). Gasification is a promising technology with high energy conversion
90 efficiency even in relatively small units (Watson *et al.*, 2018). This technology allows to convert almost
91 quantitatively biowaste organic constituents into syngas which can have various applications including
92 transformation into biomethane *via* the methanation process (Grimalt-Alemany *et al.*, 2018).

93
94 The present study reports detailed characterizations of biowaste streams produced in the metropolitan area of Lyon,
95 with regards to their potential use as energy sources through their conversion into methane. A methodology was
96 established to assess the ability of biological or thermochemical processes to convert urban biowaste into methane.
97 The major streams of municipal biowaste were firstly identified based on their quantitative production, and further
98 evaluated with respect to several criteria. A specific fractionation procedure was used to investigate the distribution
99 of organic matter and the associated methane potential between soluble and particulate phases. These
100 investigations were meant to provide information needed to optimize the overall system of energy recovery from
101 biowaste of different natures. The objective was to define adapted pretreatment operations to prepare the different
102 types of biowaste into two streams, namely i) a biodegradable aqueous pulp dedicated to anaerobic digestion, and
103 ii) a particulate recalcitrant fraction dedicated to gasification followed by syngas methanation. The present article
104 focuses on anaerobic digestion issues.

105 106 **2- Material and methods**

107 *2.1- Selection and sampling of relevant biowaste streams*

108 The nature of the biowaste streams investigated in the present study was identified and selected in collaboration
109 with the technical services of Lyon Metropolitan Area, France. Several issues were considered:

- 110 - Quantitative production of biowaste streams on the considered territory;
- 111 - Availability (dispersion, accessibility) of the waste stream, in particular with respect to the efficiency and
112 costs of its collection;
- 113 - Territorial specificities, public policies, political strategies, ongoing projects and other organizational
114 considerations.

115 Based on these considerations, four categories of biowaste were selected amongst the different biowaste streams
116 identified on the territory:

- 117 • Garden biowaste (GBW) : Collected from domestic, municipal and private sources, amount of 16000 –
118 17000 t/year available on Lyon Metropolitan area,
- 119 • Biowaste from collective restaurants (RBW): Collective catering from private and public sources including
120 schools, amount of 2500 – 6000 t/year available on Lyon Metropolitan area,
- 121 • Household kitchen biowaste (HBW): Collected from the production of food waste of a set 200 faculty
122 members and staff of our Institute, amount of 30000 – 45000 t/year available Lyon Metropolitan area,
- 123 • Food biowaste from supermarkets (SMBW): Collected from an industrial site of depackaging of mixed
124 supermarket food waste, amount of 3000 – 6500 t/year available on Lyon Metropolitan area.

125
126 Representative samples of about 200 kg of each of the four selected categories of biowaste were collected in
127 September 2018 following adapted sampling protocols according to the standard guidelines NF EN 14899, 2006.

130 2.2- *Biowaste characterization*

131

132 2.2.1- Particle size distribution

133 The particle size distribution was determined directly from the integral samples collected. To ensure a good
134 uniformity of the material for all the following analyses or assays, each primary sample was shredded twice using
135 a BLIK BB350 rotary shear crusher down to a particle size below 10 mm, then stored at 4°C.

136

137 The analyses were done in triplicates, by sieving each sample under wet conditions through a stack of seven sieves
138 of decreasing openings: 31.5 mm; 20 mm; 10 mm; 4 mm; 1 mm; 0.5 mm; 0.25 mm.

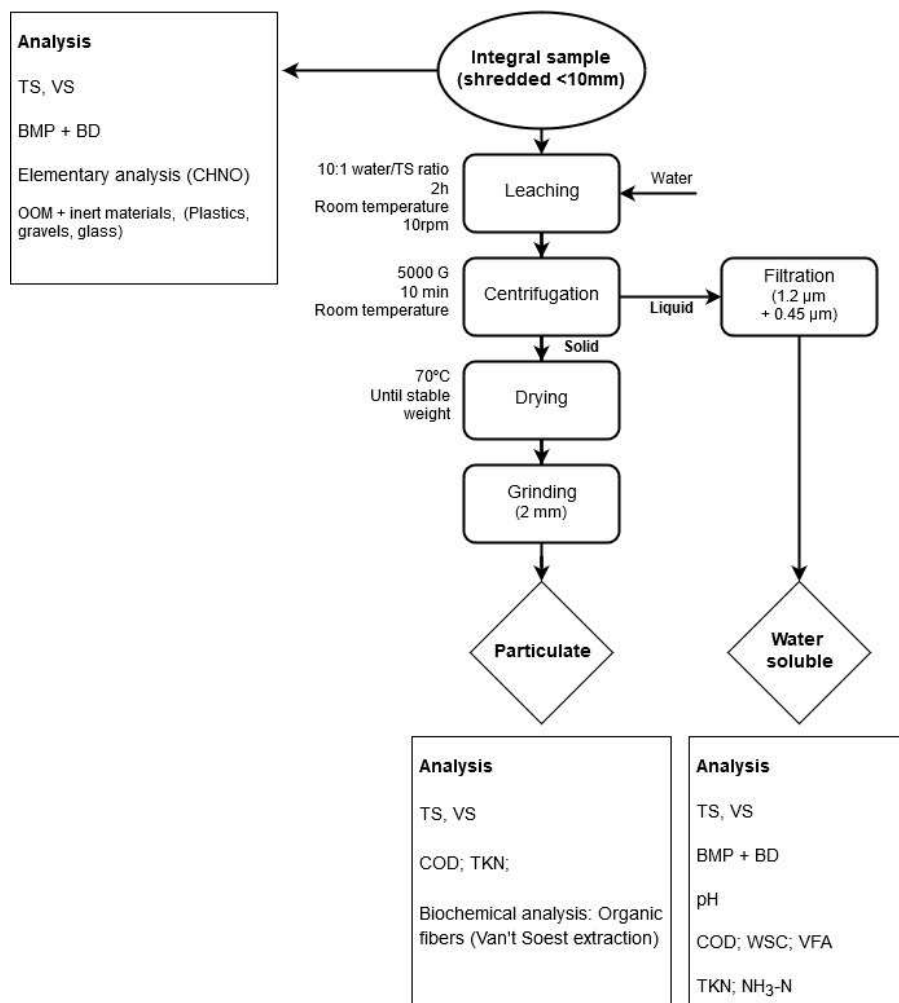
139

140 2.2.2- Fractionation of biowaste constituents by leaching

141 Each of the shredded samples was analyzed and characterized following the approach illustrated in Figure 1. The
142 protocol was designed to (i) separate water-soluble biowaste constituents from the solid particles by leaching and
143 (ii) assess the respective contributions of each fraction thereby obtained to a series of properties.

144 A leaching procedure was used to separate the soluble and particulate fractions according to standard protocol
145 AFNOR NF EN 12457-4 (AFNOR, 2002). The operation was performed in triplicates. For each crushed sample
146 of biowaste, a mass of 10 g_{wM} of integral sample (shredded < 10 mm) was placed into a flask where de-ionized
147 water was added to obtain a Liquid to Solid ratio (L/S) of 10 mL water per g of dry matter. The flasks were closed
148 and placed in a tumbler where they were mixed for 2 h by flip-flop rotation at 10 rpm and room temperature. The
149 suspensions were then collected and centrifuged at 5000 g for 10 min, followed by 2 successive filtrations at 1.20
150 and 0.45 µm using respectively glass and cellulose acetate filters. The particulate fractions were dried at 70 °C
151 until constant weight and grounded down to below 2 mm with a Retsch SM 200 cutting mill. Finally, the soluble
152 fractions and the initial integral samples were stored at 4 °C while the dried particulate fractions obtained after
153 leaching were freezed and stored at -20°C. All analyses were performed in triplicates.

154



155

156 *Figure 1: Flowchart of the experimental methodology to characterize biowaste - adapted from Teixeira Franco et al. (2019).*
 157 *Analyses were performed in triplicate, except for OOM and elementary analysis.*

158

159 2.2.3. Physical and chemical analyses of integral solid samples

160 The total solid (TS) content and moisture content (M) of the integral solid samples were measured by weighing
 161 (100 g_{WM}), drying at 105°C for 24 h and weighing again the samples (ISO 11465, 1993). The Volatile Solid (VS)
 162 content was measured by weighing a dried sample (20 g_{TS}), burning it at 550°C for 4h and weighing it again.
 163 Oxidizable Organic Matter (OOM) content was determined according to the standard gravimetric procedure
 164 AFNOR XP U44-164 (AFNOR, 2014). This procedure allows to oxidize the reactive organic matter using an
 165 aqueous solution of sodium hypochloride. After reaction, the suspension was filtered, the solids collected and
 166 dried. They were considered as impurities such as plastics, glass, sand or gravel, etc. Inert material (IM) and plastic
 167 material (PM) were weighed. Finally, the OOM content was calculated as the difference: OOM = TS - PM - IM.
 168 The OOM content was considered as a rough estimation of the biodegradable organic matter content (de Araujo
 169 Morais *et al.*, 2008).

170

171 2.2.4. Physical and chemical analyses of particulate fractions

172 Dried particulate fractions obtained after leaching were analyzed, including TS and VS contents measured in the
 173 same condition as integral solid samples. COD, Total Kjeldahl Nitrogen (TKN), and ammonia nitrogen (NH₃-N),
 174 were analyzed in triplicates. Cell wall constituents (biochemical composition) were analyzed by successive

175 hydrolysis steps and extraction with different solvents, according to the protocol developed by van Soest and Wine
176 (1967), following the standard NF FD U44-162 (AFNOR, 2016). Analyses were done on aliquots of powdered
177 dry samples (obtained by grinding down to < 2 mm) corresponding to 2 g of volatile matter. At each step, the
178 residual solids were dried and weighed, and their VS contents analyzed before the solids were used in the next
179 extraction step. Four fractions were obtained, namely (1) neutral detergent soluble fraction (SOL) extracted at the
180 first step by a neutral detergent aqueous solution; (2) Hemicellulose-like (HEM) extracted with a dilute acidic
181 aqueous detergent solution; (3) Cellulose-like (CELL) extracted with a concentrated 72 % sulfuric acid solution;
182 and (4) lignin-like residual organic matter (RES) which was not extracted in the procedure.

183

184 2.2.5. Physico-chemical analyses of aqueous solutions

185 Aqueous solutions were analyzed in triplicates for TS, VS, pH, Water Soluble Carbohydrates (WSC), Volatile
186 Fatty Acids (VFA), Chemical Oxygen Demand (COD) in water-soluble fraction, Total Kjeldahl Nitrogen (TKN),
187 and ammonia nitrogen (NH₃-N).

188

189 2.2.6. Biochemical methane potential - Biodegradability

190 Biomethane potential (BMP) were measured on the integral waste and the aqueous fraction obtained by leaching
191 (Fig. 1) of each selected biowaste, following the guidelines reported by Holliger *et al.* (2016). The assays were
192 conducted at 35°C in 2 L glass vessels for integral samples and 0.25 L for the water-soluble phase. Digested sludge
193 from the wastewater treatment plant of La Feysine, Lyon, France was used as an inoculum (TS 2.0-3.3% wt; VS
194 1.4-2.2% wt) at an inoculum to substrate VS ratio of 2 g/g. A mineral solution providing essential nutrients to
195 microbial growth and a buffer solution were added to the reactors according to the recommendations of ISO 11734
196 standard (ISO, 1995). Once filled, reactors were purged with a N₂/CO₂ (80/20% v/v) gas flow for about 2 minutes,
197 sealed and equilibrated at 35°C. Blanks containing only the inoculum and the mineral solutions were systematically
198 monitored along with each series of assays in order to correct the recorded BMP from residual methane production
199 of the inoculum.

200 All tests were performed in triplicates. Biogas production was determined by pressure measurement using a
201 Digitron precision manometer. Biogas was released when the pressure exceeded 1200 hPa. Gas composition was
202 analysed using an Agilent 3000 micro gas chromatography with thermal conductivity detector (GC-TCD).
203 Molsieve 5A (14 m length; pore size: 5 Å) and Poraplot A (10 m length; 0.320 mm ID) columns were used as
204 stationary phases for GC-TCD, with Argon and Helium as carrier gases, respectively. The tests were stopped when
205 the daily biogas production was less than 1% of the total volume of biogas produced.

206 The anaerobic biodegradability of each fraction was calculated from BMP and COD values as described below:

207

$$BD (\%) = \frac{BMP [L_{STP}/kg_{TS}]}{COD [kg/kg_{TS}] \times 0.35} \quad (1)$$

208

209 where 0.35 is the theoretical BMP of 1 kg of COD in L_{STP}/kg_{COD}

210

211 Following guideline from Holliger *et al.* (2016), duration of the BMP test was not fixed in advance. However, in
212 order to standardize the results, data were stopped at 60 days ensuring production <1% of the accumulated
213 volume of methane for all tests. The kinetics of methane production during 60 days of the BMP tests was

214 determined from the net methane production (i.e., after subtracting the blank methane production) according to a
215 first order model (equation 2).

216

$$V_{CH_4}(t) = V_{max}(1 - e^{-kt}) \quad (2)$$

217

218 where V_{CH_4} is the cumulated volume of methane produced at time t ; t the time, V_{max} the maximum volume of
219 methane produced, and k the first order kinetic constant.

220

221 2.2.7. Evaluation of the distribution of TKN, COD and BMP parameters into water-soluble and particulate
222 fractions

223 Results from analyses of aqueous and particulate fractions after leaching operation of integral samples (Fig. 1)
224 were used to assess the contribution of each phase (i.e, soluble and particulate). Selected characteristics of the
225 biowaste samples were calculated using equations (3), (4) and (5) below for BMP, COD and TKN parameters,
226 respectively.

227

228 The contribution of the particulate phase to the overall BMP of the integral sample was calculated as the difference
229 between the BMP of the integral sample and the BMP of the water-soluble phase (equation 3).

230 In a similar manner, COD and TKN of the integral sample were calculated as the sum of the water-soluble and
231 particulate contributions (equations 4 and 5, respectively).

$$BMP_P[L_{STP}/kgTS_{IS}] = BMP_{RS}[L_{STP}/kgTS_{IS}] - BMP_{WS}[L_{STP}/kgTS_{IS}] \quad (3)$$

$$COD_{IS}[kg/kgTS_{IS}] = COD_{WS}[kg/kgTS_{IS}] + COD_P[kg/kgTS_{IS}] \quad (4)$$

$$TKN_{IS}[kg/kgTS_{IS}] = TKN_{WS}[kg/kgTS_{IS}] + TKN_P[kg/kgTS_{IS}] \quad (5)$$

232 where subscript P refers to the Particulate fraction, subscript IS to the Integral sample and WS to the water-soluble
233 fraction.

234

235 2.3- Sampling, analyses and data treatment strategies

236 Fractionation by leaching was performed twice on each integral waste. Depending of TS content, first fractionation
237 was performed with 100 to 300 g_{int. biowaste} and second fractionation with 450 to 1500 g_{int. biowaste}. Samples were taken
238 from integral biowaste using quartering method to ensure homogeneity.

239

240 Physical and chemical analyses (TS/VS, COD, BMP, N-TKN, Ligno-cellulose matter) were achieved in triplicate,
241 except for elemental analyses and OOM measurements carried on a large quantity of samples (200 g)
242 representative of the biowaste selected. For each result, the mean value and standard deviation rate are presented.

243

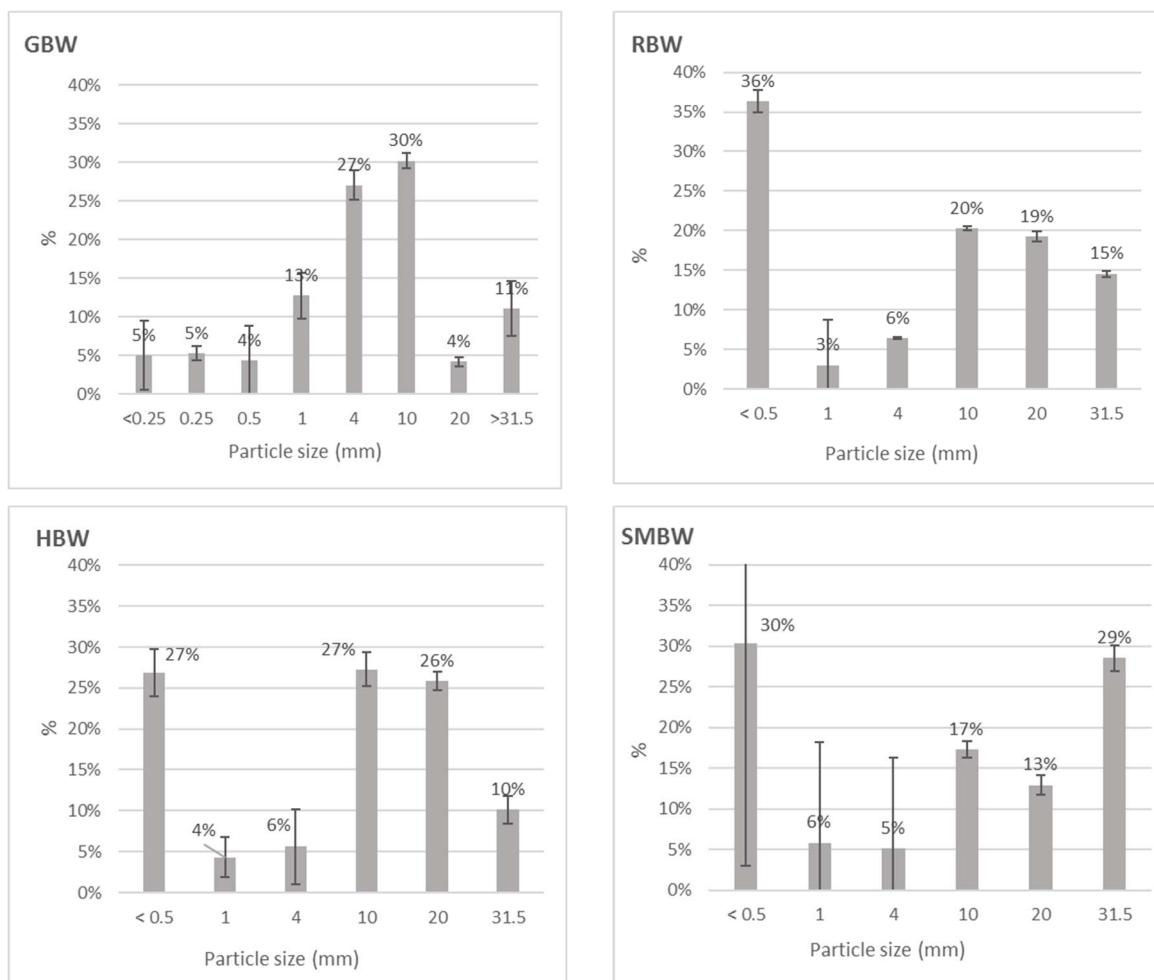
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245 3- Results and discussion

246 3.1- Physical and chemical analyses of integral biowaste

247 Figure 2 showed that all samples exhibited a relatively wide particle size distribution. Restaurants (RBW),
248 Household (HBW) and Supermarkets (SMBW) biowaste revealed a similar profile of distribution, with a dual

249 predominance of particles below 0.5 mm (27 to 36% of TS) and above 10mm (54 to 63% of the total). In contrast,
 250 Garden waste GBW revealed a small proportion of particles below 0.5 mm (10% of TS). This was probably due
 251 to the relative predominance of bigger particles of lignocellulosic materials. The strong dispersion of the size
 252 distribution in SMBW may be explained by the presence of packaging material in the integral biowaste sample.
 253
 254

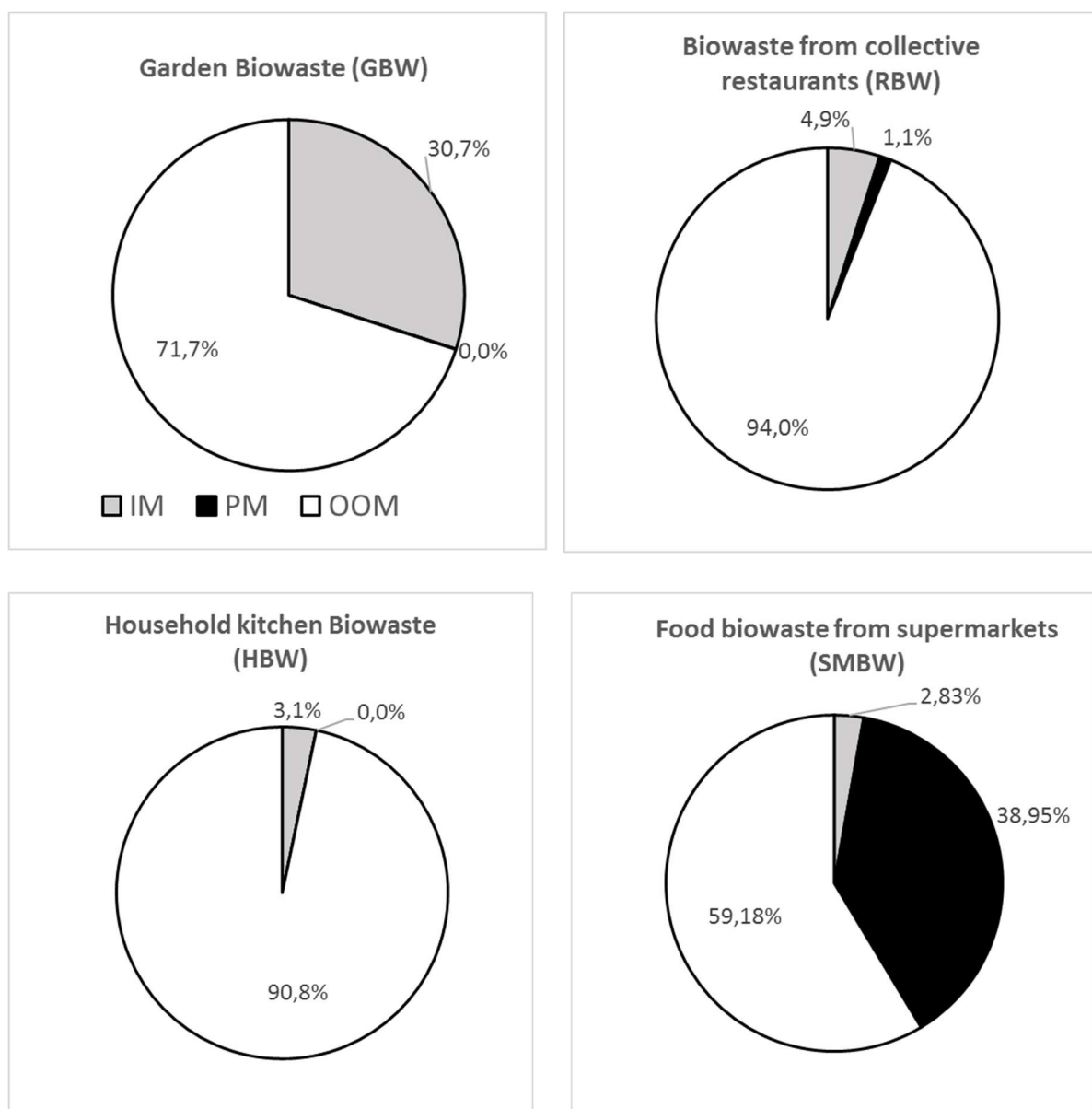


255 *Figure 2: Particle size distribution on the four-selected biowaste. Data expressed on a TS basis. GBW: Garden biowaste;*
 256 *RBW: Restaurants biowaste; HBW: Household biowaste; SMBW Supermarkets biowaste. Result are average values of*
 257 *triplicated assays. Vertical bars show standard deviations.*

258
 259 The proportions of OOM, IM and PM in the TS of each sample are illustrated in Figure 3. TS and VS concentration
 260 are presented in Table 1. Garden waste (GBW) exhibited a moisture content of 33%_{WM}, almost 2-fold lower than
 261 that of the other samples. Its VS content (72%_{TS}) was also lower than measured in the other samples (more than
 262 90%_{MS}). Figure 3 showed that GBW contained by far the highest contents in inert materials with 30.7 %_{TS}
 263 whereas the proportions of inert materials were around 3%_{TS} in households biowaste HBW and supermarket
 264 biowaste SMBW and close to 5%_{TS} in restaurants biowaste. These observations suggested the presence of sand
 265 or gravel particles in GBW, which probably explained the specific profile of particle size distribution (Fig. 2)
 266 discussed above.

267 Results of OOM analyses were in good agreement with the measured VS contents except for SMBW. This was
 268 attributed to the presence in SMBW of 39%_{TS} synthetic plastic-type organic materials from packaging. Food waste

269 collected from restaurants, households and supermarkets were more acidic (pH around 4.5) than garden waste (pH
 270 7.4), probably due to the acidogenic microbial evolutions of their readily biodegradable constituent during the
 271 collection operations as already reported by Zhang *et al.* (2014), and Fisgativa *et al.* (2016).
 272



273 *Figure 3: Impurity contents in the four selected biowaste (expressed on a TS basis): inorganic inert materials (IM), plastic*
 274 *materials (PM), and non-synthetic oxidizable organic matter (OOM).*

275
 276 The biochemical compositions (cell wall constituents) and the elemental contents of the different samples are
 277 presented in Table 1. The results confirmed the specific composition of garden biowaste GWB, whose lignin-like
 278 (27.2%_{VS}) and cellulose (44%_{VS}) contents revealed clearly the lignocellulosic nature of the waste materials. In
 279 contrast, household biowaste HBW contained nearly 83%_{VS} of "soluble" organic matter and almost no ligno-
 280 cellulosic compounds. HBW and Supermarket biowaste SMFW revealed relatively similar biochemical profile
 281 with a predominant fraction of "soluble" organic compounds, between 20 and 30%_{VS} of hemicellulosic and
 282 cellulosic fractions, and below 10% of residual constituents (lignin-like).

283 C, H, and N contents were found to be relatively similar in all the selected biowaste samples, due to the common
 284 nature of their organic constituents. C / O and C / N ratios were however different in the samples. GBW exhibited
 285 the highest C / O and C / N ratios. The high lignin content in GBW probably explained the relatively low O content
 286 and high C/O ratio as compared to the other samples, while the low N content and high C/N was attributed to the
 287 low protein content.

288
 289
 290

291 *Table 1: Total Solid, Volatile Solid, Elemental and biochemical analysis of the organic matter present in the four biowaste*
 292 *from the collection campaign. GBW: Garden biowaste; RBW: Restaurants biowaste; HBW: Household biowaste; SMBW*
 293 *Supermarkets biowaste. Result are average values of triplicated analyses.*

Biowaste	TS (% _{wM})	VS (% _{TS})	C (% _{TS})	H (% _{TS})	O (% _{TS})	N (% _{TS})	Soluble (% _{vs})	Hemicelluloses (% _{vs})	Cellulose (% _{vs})	Residues (% _{vs})
GBW	66.2 ± 0.5	71.7 ± 3.5	40.3	4.7	16.6	1.3	18.6 ± 4.8	10.3 ± 6.4	44.0 ± 1.7	27.2 ± 0.1
RBW	25.6 ± 2.4	95.0 ± 0.1	52.6	7.3	29.4	4.0	82.7 ± 0.1	4.0 ± 0.3	12.6 ± 0.3	0.7 ± 0.04
HBW	19.1 ± 0.94	90.8 ± 0.1	47.2	6.2	33.6	2.1	57.5 ± 0.4	11.6 ± 1.4	21.0 ± 1.0	9.9 ± 0.01
SMBW*	30.8 ± 2.2	94.2 ± 0.1	50.9	7.0	29.9	4.1	74.6 ± 0.6	7.4 ± 0.9	11.1 ± 0.8	6.9 ± 0.5

294 * Analyses were done on the SMBW sample after extraction of the packages fraction,

295

296 3.2- BMP and biodegradability of the integral biowaste

297 Table 2 shows BMP values and kinetic constant (k) measured during the course of the incubations of the integral
 298 biowaste samples at 35°C. For each biowaste sample, cumulated methane production curve over the time was used
 299 to determine BMP at the end of the incubation period (see supplementary data). The k constant was deduced
 300 considering equation (2).

301 As already observed for the other parameters analyzed before, the garden biowaste GBW exhibited very different
 302 results as compared to the other three samples. Its BMP (36 NL_{CH₄}.kg⁻¹_{TS}) was found to be ten-fold smaller than for
 303 the other biowaste samples. This observation confirmed the poor biodegradability of GBW sample studied here.
 304 At the season when the sample was collected (fall), garden waste contains predominantly recalcitrant lingo-
 305 cellulosic constituents as discussed above (Table 1). RBW and SMBW exhibited very similar BMP, 397 and 417
 306 NL_{CH₄}.kg⁻¹_{TS} respectively. The BMP measured for HBW was about 30% smaller (260 NL_{CH₄}.g⁻¹_{TS}). This difference
 307 was attributed to the lower soluble fraction measured in HBW (57.5% of the VS content, see Table 3), as compared
 308 to RBW and SMBW. This observation was attributed to the presence of waste materials other than biowaste in the
 309 HBW due to the fact that source selection by the households was probably not perfectly specific. SMBW also
 310 contained undesirable materials (packages) but these were removed from the sample before the BMP assays were
 311 done.

312

313 *Table 2 : Methane potentials and 1st order kinetic constants of its production from the four selected biowaste,*
 314 *calculated from the curves shown in Fig. 4. GBW: Garden biowaste; RBW: Restaurants biowaste; HBW: Household*
 315 *biowaste; SMBW Supermarkets biowaste. Result are average values of triplicated assays.*

Analyses	GBW	RBW	HBW	SMBW*
BMP (NL _{CH₄} .kg ⁻¹ .Ts)	36.2 ± 7.3	385.5 ± 8.9	258.2 ± 13.9	417.4 ± 11.1
<i>k</i> (d ⁻¹)	0.137 ± 0.020	0.209 ± 0.004	0.275 ± 0.005	0.269 ± 0.001

316 * SMBW sample was treated before the test to remove manually all materials other than bio waste (packages).

317

318 The kinetic constant *k* ranged from 0.14 d⁻¹ for GBW to 0.27 d⁻¹ for SMBW (Table 4), confirming the poor
319 biodegradability of GBW. RBW, HBW and SMBW showed high kinetics of biomethane production, with almost
320 90% of methane being produced in less than 10 days. Organic matter from these biowaste samples were therefore
321 easily available for anaerobic digestion.

322

323 3.3- Fractionation of biowaste constituents by leaching in water

324 Table 3 shows the composition of the aqueous solutions obtained after 2 hours of leaching of the biowaste samples.
325 Leachates were successively filtered at 1,20 µm and 0.45 µm before analyses. Biowaste samples from restaurants
326 (RBW), households (HBW) or supermarkets (SMBW) all produced leachates with a low pH (< 5) and a high COD,
327 unlike garden biowaste GBW whose leachates were neutral with a low COD. RBW, HBW and SMBW contained
328 food waste materials that were readily biodegradable as shown by the BMP results discussed above. The acidity
329 of the leachates was therefore explained by a probable acidogenic evolution of the biowaste materials during the
330 collection process. The presence of free sugars (WSCs, see Table 3) in these waste, which can be rapidly
331 metabolized into volatile fatty acids (VFAs), and the high VFAs concentrations measured in the leachates
332 confirmed this assumption.

333 *Table 3: Composition of the leachates obtained after 2h of leaching of the 4 integral waste for 2h with water at a L/S of 10,*
334 *room temperature, mixing at 10 rpm. GBW: Garden biowaste; RBW: Restaurants biowaste; HBW: Household biowaste;*
335 *SMBW Supermarkets biowaste. Result are average values of triplicated assays.*

Parameter	GBW	RBW	HBW	SMBW
pH	7.6 ± 0.2	4.5 ± 0.1	4.7 ± 0.1	4.4 ± 0.1
Nitrogen				
N-NH ₃ (mg.L ⁻¹)	< DL	101 ± 2	79 ± 1	98 ± 1
N-TKN (mg.L ⁻¹)	51 ± 0.7	1 157 ± 13.8	240 ± 6.5	1 165 ± 15.4
Water-soluble organic matter				
COD _L (g.L ⁻¹) – total COD in solution	2.5 ± 0.1	34.3 ± 1.7	36.5 ± 1.8	34.5 ± 1.7
VFA (equivalent g _{COD} .L ⁻¹)	0.02	3.76	7.44	6.50
(% of COD _L)	0.9	11.0	20.4	18.8
WSC (equivalent g _{COD} .L ⁻¹)	0.2	0.1	2.5	1.2
(% of COD _L)	5.5	0.3	6.8	3.6
Anaerobic biodegradation				
BMP (NL _{CH₄} .kg _{COD} ⁻¹)	268 ± 5	260 ± 3	243 ± 11	312 ± 15
BMP (NL _{CH₄} .L ⁻¹)	1.20 ± 0.01	9.20 ± 0.11	9.40 ± 0.42	10.80 ± 0.52
<i>k</i> (d ⁻¹)	0.270 ± 0.012	0.285 ± 0.018	0.399 ± 0.158	0.360 ± 0.017
BD (%)	17 ± 0.3	77 ± 0.9	74 ± 3.3	89 ± 3.1

336 Water Soluble Carbohydrates (WSC), Volatile Fatty Acids (VFA), Chemical Oxygen Demand (COD), Total Kjeldahl Nitrogen (TKN), and
337 ammonia nitrogen (N-NH₃), < DL: below the detection limit.

338

339 Regarding the 4 biowaste, ammonia nitrogen represented a very low fraction of the total nitrogen content (TKN)
340 in the leachates. The leachates from RBW and SMBW showed the highest N-NTK concentrations with around

341 1200 mg of dissolved nitrogen per litre of solution. The leachate from GBW exhibited much lower N-TKN
342 concentrations ($51 \text{ mg}\cdot\text{L}^{-1}$), indicating that as expected GBW contained much less proteins than the other samples.
343 The methane potentials BMP of the leachates from restaurants (RBW), households (HBW) and supermarkets
344 (SMBW) were in the same order of magnitude, around 10 NL of methane per L of leachate, confirmed the similar
345 composition of these 3 categories of food biowaste. The leachate from GBW showed a BMP 10-fold smaller,
346 confirmed the results discussed above. Biodegradability during BMP test (BD) was very high for SMBW with of
347 89% biodegradation. SMBW produce high digestible leachate. BD were lower for RBW and HBW with 77% and
348 74 % respectively, meaning that leachate component are less digestible for these biowaste. According to his BMP
349 value, GBW showed only 17% of BD after BMP test.

350

351

352 *3.4- Distribution of the key characteristics of the biowaste between soluble and particulate compartments*

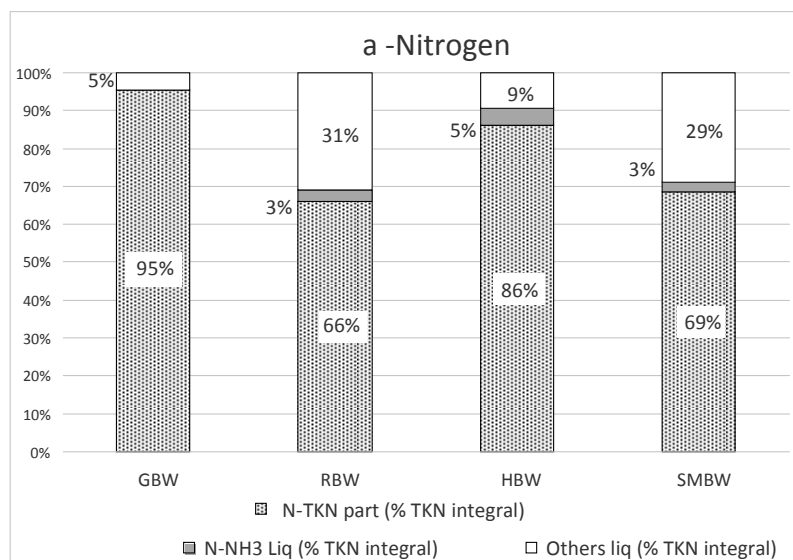
353 The distribution of nitrogen, COD, methane potential (BMP) and anaerobic biodegradability between the solid
354 (particulate) and the water-soluble (leachate) fractions is illustrated in Figure 4 a, b and c. The numerical values
355 of the parameters are gathered in Table 4. COD and BMP are expressed with respect to the TS content of the
356 particulate and water-soluble fractions obtained by leaching of the different samples, or the TS content of the
357 respective integral samples.

358 As already observed from the previous analyses, GBW differed here from the other three samples by a very high
359 particulate over soluble COD ratio of $39 \text{ gCOD}_{\text{part.}} / \text{gCOD}_{\text{soluble}}$ whereas the ratios in the other samples were more
360 than ten-fold smaller (3.0 to $3.3 \text{ gCOD}_{\text{part.}} / \text{gCOD}_{\text{soluble}}$). A small proportion (5%) of total COD was solubilized
361 by leaching of GBW as compared to 23% for RBW, HBW and SMBW.

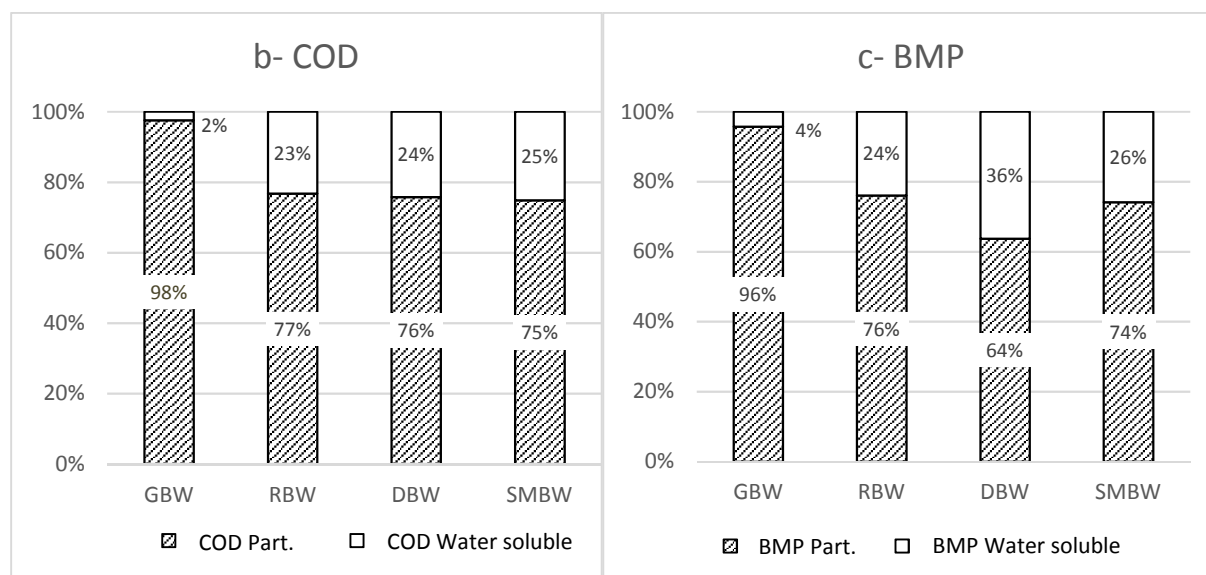
362 The distribution of the BMP between particulate and water-soluble (leachate) phase was also very different in
363 GBW with a BMP S/L ratio of 17 whereas the ratio ranged between 2 and 3 for the other samples. GBW also
364 showed by far the lowest BMP values both for the integral sample ($36 \text{ NL}_{\text{CH}_4}\cdot\text{kg}^{-1}\text{TS}$) and the solid fraction (34
365 $\text{NL}_{\text{CH}_4}\cdot\text{kg}^{-1}\text{TS}$) which were 5 to 10 times lower than observed with the other biowaste.

366 Nitrogen distribution show more than 30% of total nitrogen in soluble phase ($< 0.45 \mu\text{m}$ filtration) for RBW and
367 SMBW. GBW and HBW have a poor nitrogen soluble phase with respectively 5% and 14 % of total distribution.

368



370



371

372 Figure 4: Distribution of Nitrogen (a), COD (b), and BMP (c) between water soluble and particular fractions obtained by
 373 leaching Garden biowaste (GBW), Restaurant biowaste (RBW), Household biowaste (HBW) and Supermarket biowaste
 374 (SMBW). Result are average values of triplicated assays.

375 Table 4: COD concentration, BMP value and anaerobic biodegradability of the four selected biowaste. GBW: Garden
 376 biowaste; RBW: Restaurants biowaste; HBW: Household biowaste; SMBW Supermarkets biowaste. Result are average
 377 values of triplicated assays.

		GBW	RBW	HBW	SMBW
Integral sample					
COD	$\text{g.kg}^{-1}\text{TS}$	1035 ± 9	1477 ± 44	1506 ± 27	1372 ± 32
BMP	$\text{NL}_{\text{CH}_4}.\text{kg}^{-1}\text{TS}$	36 ± 7	385 ± 9	258 ± 14	417 ± 11
BD	%	10 ± 0.1	75 ± 2.2	49 ± 0.9	87 ± 2.0
N-TKN	$\text{gN.kg}^{-1}\text{VS}$	15.6 ± 0.3	35.8 ± 0.6	19.2 ± 0.3	39.4 ± 1.0
Water-soluble phase					
COD	$\text{g.kg}^{-1}\text{TS}$	25 ± 1	343 ± 2	365 ± 2	345 ± 2
BMP	$\text{NL}_{\text{CH}_4}.\text{kg}^{-1}\text{TS}$	1 ± 0.1	92 ± 1	94 ± 4	108 ± 5
BD	%	17 ± 0.3	77 ± 0.9	74 ± 3.3	89 ± 3.1
N-TKN	$\text{gN.kg}^{-1}\text{VS}$	0.7 ± 0.01	12.2 ± 0.14	2.6 ± 0.07	12.4 ± 0.16
Particulate phase *					

COD	g.kg ⁻¹ _{TS}	1010 ± 9	1135 ± 34	1142 ± 21	1027 ± 24
BMP	NL _{CH₄} .kg ⁻¹ _{TS}	34 ± 7	293 ± 8	166 ± 1	309 ± 8
BD	%	8 ± 1.6	77 ± 2.0	50 ± 0.3	88 ± 2.3
N-TKN	gN.kg ⁻¹ _{VS}	14.9 ± 0.2	23.7 ± 0.4	16.6 ± 0.3	27.0 ± 0.7

378 *calculated with equation (3)

36.2 ± 7.3 385.5 ± 8.9 258.2 ± 13.9 417.4 ± 11.1

379

380

381 **4- Conclusion**

382 This study was conducted as a contribution to develop and implement a general strategy to recover energy from
383 biowaste streams in urban territories. More specifically, the objective was to develop an integrated management
384 pattern of the available biowaste resources in order to produce methane efficiently with adapted conversion
385 technologies. The idea was to estimate the feasibility of pretreatment operations that would convert the variety of
386 solid biowaste materials into (i) one aqueous slurry feedstock concentrating the most readily biodegradable
387 constituents dedicated to anaerobic digestion, and (ii) one particulate, dryer and more recalcitrant fraction
388 dedicated to gasification followed by methanation.

389 Four biowaste streams were identified as the most attractive resources, namely Garden biowaste (GBW),
390 Restauration biowaste (RBW), Household biowaste (HBW) and Supermarkets biowaste (SMBW). The selected
391 biowaste were sampled from the urban territory of Lyon, France, analyzed for a series parameters of interest, and
392 fractionated by leaching into the desired 2 types of feedstocks (particulate and soluble fractions).

393 Results showed that garden waste GBW was inappropriate for anaerobic digestion as a whole, and also not suitable
394 either to produce an aqueous slurry that could be treated by AD. The water-soluble fraction was low and therefore
395 the bioavailability of readily biodegradable organic substrates was poor. The hydrolysis of particulate organic
396 matter (POM) would therefore probably be limiting kinetically the anaerobic digestion process. In addition, the
397 BMP of the particulate fraction was also very low, indicating that the POM contained recalcitrant polymers as
398 confirmed by the biochemical analyses which showed a very high proportion of lignocellulosic compounds. Due
399 to these characteristics, it was concluded that GBW should be dedicated to gasification rather than anaerobic
400 digestion.

401 In contrast, RBW, HBW and SMBW were identified as good candidates for pretreatment operations to convert
402 them into an aqueous slurry, with good characteristics for AD, and a particulate fraction with very different
403 characteristics that would be adapted to gasification. HBW and RBW exhibited high availability of organic matter.
404 BMP and COD concentration observed was high in leachate fraction indicate high easily water-soluble component.
405 Supermarkets biowaste (SMBW) differed from the other three biowaste samples by the presence of nearly 40% of
406 plastics from packaging. These undesirable materials should be removed from the biowaste material prior to
407 biological treatment. However, this operation would need the implementation of adapted separation techniques
408 due to the wide distribution of particle sizes and the variety of nature and composition of the undesirable particles.
409 Despite this unfavorable characteristic, the BMP of the biowaste material (separated manually in the present study
410 from the plastic materials) was around 450 NL_{CH₄}.kg⁻¹_{TS}, which was close to that obtained for RBW, with high
411 methane production kinetics. Similar to HBW, SMBW would require pretreatment operations to separate
412 undesirable fractions, mainly packaging plastics, and extract the biodegradable organic matter in a form of an
413 aqueous pulp dedicated to the anaerobic digestion.

414 Based on the encouraging results, the precise conditions of the pretreatment operations needed to generate 2
415 feedstock is under investigation in our group.

416

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