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

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

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- 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
- investigated on urban household biowaste. Two liquid to solid ratios were tested in each pre-
- treatment. Anaerobic digestion of pre-treated biowaste was studied by measuring their
- biomethane potentials and by controlled experiments in a continuously stirred-tank reactor with
- a feed load of 3.5 gVS.L<sup>-1</sup>.d<sup>-1</sup>. It was observed that increasing liquid to solid ratio in the pre-
- treatments allowed to increase the proportion of biodegradable organic matter extracted from
- the biowaste, up to 949 gCOD.kgTS<sup>-1</sup> from household biowaste. The biomethane potentials of
- pre-treated waste were very high (up 525 LCH<sub>4</sub>.kgVS<sup>-1</sup>) and COD (949 gCOD.kg<sup>-1</sup>TS) from
  - household biowaste. Anaerobic digestion in continuously stirred-tank reactor showed a very
- strong conversion of COD load (81%) and a high methane production up to 345 LCH<sub>4</sub>.kgVS<sup>-1</sup>.

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

- 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

# 1 Introduction

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Over the last decade, European and French legislations on waste management have been strongly pushing the prevention of waste production and the recovery of materials and /or energy from the waste stream, with a strong incentive to reduce drastically landfill disposal. The European Directive 2018/851/EC encourages European Union member states to implement systems for the specific collection of source-separated biowaste to facilitate the recovery of organic matter (European Union, 2018). In France, the energy transition for green growth act requires that municipalities take proper measures to achieve before 2025 specific collection and resource recovery targets from municipal biowaste streams (LTECV, 2015). Despite the efforts to prevent waste generation, Municipal Solid Waste (MSW) production is still growing in most cities, following the growth of the population in urban areas (Fisgativa et al., 2016, Jank et al., 2017). MSW includes several categories of waste collected on the urban territories, including biowaste. Municipal biowaste consists of gardens and parks waste, food and kitchen waste from households, offices, restaurants, wholesalers, canteens, catering and retail units, and similar waste from food processing industry (European Union, 2018). Household waste represents the main category of biowaste, corresponding to more than 34% of the mass of MSW generated in Europe in 2017 (European Environmental Agency, 2020).

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

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

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

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

#### 2 Material and Methods

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

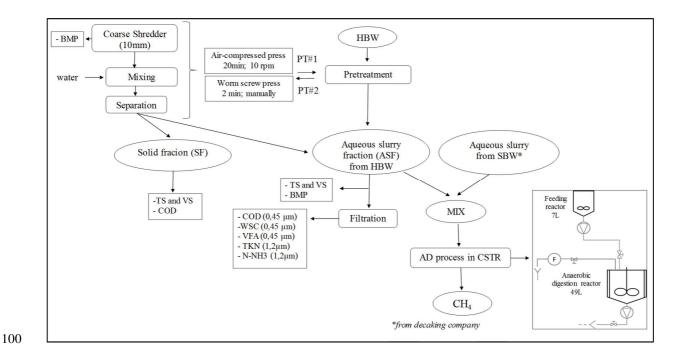


Fig. 1. Flowchart of global scheme of pre-treatment and anaerobic digestion process

#### 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.

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

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

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

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

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

# 2.1.4 Physico-chemical analyses of the ASF

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

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

Volatile fatty acids (VFA) were measured by gas chromatography (Shimadzu Corp.) equipped with a HP-FFAP fused silica capillary column (30 m×0.25 mm Agilent Technologies), using H<sub>2</sub> as carrier gas and a flame ionization detector. Lactic acid, formic acid, and water soluble carbohydrates (WSC) were analysed by liquid chromatography (LC Module 1 plus, Waters), equipped with a Supelcogel<sup>TM</sup> C-610 column (300 mm×7.8 mm, Sigma-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 at 0.45 μm before analyses. All the analyses were performed in triplicates.

# 2.1.5 Biochemical methane potentials (BMP) analyses of ASF

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

Biogas production was measured by monitoring the pressure in the flasks using a Digitron precision manometer. The gas composition was analysed 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. The tests were stopped when the biogas production in one day was less than 1% of the total volume of biogas produced (Holliger et al., 2016). The rate of methane production was determined from the net methane production and was used a first order kinetic model, as presented in Eq.1.

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

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

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

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

- 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.

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

# 2.2.2. Anaerobic digestion process and operational condition

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

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

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

#### 2.2.3. Reactor monitoring

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

# 3 Results and Discussion

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

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

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

ASF	PT#1: HBW	pressed with	air-	PT#2: H	BW	pressed	with	worm
	compressed Press			screw press				
Ratio	L/S 5	L/S 10		L/S 5		L/S	10	
TS	$7 \pm 0.4\%$	$5 \pm 0.4\%$		$8\pm0.0\%$		$4 \pm 0$	0.0%	
VS	$79 \pm 1.0\%$	$84 \pm 1.0\%$		$86 \pm 0.0\%$	ó	87 ±	1.0%	

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

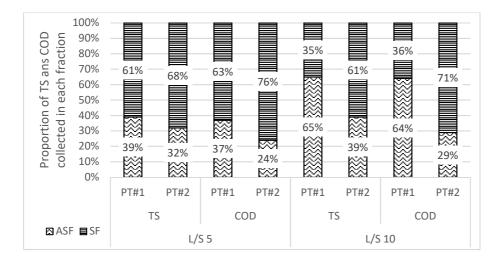
A wide range of TS value of ASF was found in literature, ranging from 10 to 42% (Hansen et al., 2007, Bernstad et al., 2013, Jank et al. 2015, Micolucci et al., 2015, Lopes et al., 2019). Similarly, Lopes et al. (2019) analysed, among other processes, the pressing of the mixture of food and garden waste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

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

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

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



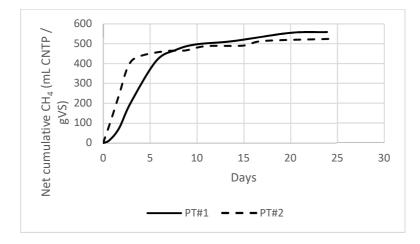


**Fig. 2.** Evolution of mass fraction of TS and COD at different L/S ratio: 5 and 10 gWater.gTS<sup>-1</sup> of HBW pressed with air-compressed press (PT#1) and HBW pressed worm screw press (PT#2)

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

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

Fig. 3 presents cumulative production of methane during BMP test for ASF from air-compressed press PT#1 and worm screw press PT#2. Methane production from the ASF from HBW processing with PT#1 system (523.8 NLCH4.kgVS<sup>-1</sup>) and PT#2 system (524.8 NLCH4.kgVS<sup>-1</sup>) obtained was similar. However, the kinetic constants were higher in ASF from worm screw press PT#2 (0.57.d<sup>-1</sup>) when compared to ASF from air-compressed press PT#1 (0.29.d<sup>-1</sup>). These results suggest less readily bioavailable compounds in the last fraction. The values obtained are similar to those obtained by Alessi et al. (2020) of 536 NLCH4.kgVS<sup>-1</sup> and slightly above those found in other studies: 360 to 460 NLCH4.kgVS<sup>-1</sup> (Hansen et al., 2007; Jank, et al., 2015; Fisgativa et al., 2016).



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

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

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

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

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

<sup>\*</sup> BMP calculated from BMP of HBW and ASF, considering VS of each fraction in the Mix.

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

# 3.2.1 ASF mix composition: ASF of HBW and SBW

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

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

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

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

# 3.2.2. Performance of mesophilic anaerobic digestion on ASF mix

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

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

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

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

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

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

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

# 4 Conclusions

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

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

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

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416

#### References

- 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

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

- Bayard, R., Liu, X., Benbelkacem, H., Buffiere, P., Gourdon, R., 2016. Can Biomethane
- 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.

- Bernstad, A., Malmquist, L., Truedsson, C., la Cour Jansen, J., 2013. Need for improvements
- 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

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

443

- European Environmental Agency, 2020. Bio-waste in Europe turning challenges into
- 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:
- 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.

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

- 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
- recovery. Waste Manag. 27 (3), 398-405, doi: https://doi.org/10.1016/j.wasman.2006.02.014.

460

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

464

- Holliger, C., Alves, M., Andrade, D., Angelidaki, I., Astals, S., Baier, U., ... & Wierinck, I.,
- 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

- 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

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

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

- 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.

485

- 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

- 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

- Malinowsky, C., Nadaleti, W., Debiasi, 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:
- 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.

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

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

- Micolucci, F., Gottardo, M., Cavinato, C., Pavan, P., & Bolzonella, D., 2016. Mesophilic and
- 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

- 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
- 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

- Moretti, P., de Oliveira, M. M., Bayard, R., Buffiere, P., de Araujo, J. M., de Castilhos Jr., A.
- 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

- 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
- technology, 247, 103-115, doi: 10.1016/j.biortech.2017.09.085.

- 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.

- Nayono, S. E., Gallert, C., Winter, J., 2010b. Co-Digestion of Press Water and Food Waste in
- 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

- Naroznova, I., Møller, J., Larsen, B., Scheutz, C., 2016. Evaluation of a new pulping technology
- 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.

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
- Management, 128, 203-210, doi: https://doi.org/10.1016/j.enconman.2016.09.066.

543

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

546

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

- 551 Shahriari, H., Warith, M., Hamoda, M., Kennedy, K.J., 2012. Anaerobic digestion of organic
- 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.

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

559

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

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- Fig. 3. Evolution of net cumulative biomethane production through BMP test of ASF after PT#1
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