

Urban bio-waste valorization – resource evaluation and characterization for energy recovery by anaerobic digestion

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Abstract

Source separation will help divert fermentescible organic wastes, in particular from landfilling and incineration. In agreement with the European Directives, the French Energy Transition for Green Growth Act (TECV, 2015) has set a series of objectives with respect to waste prevention and bio-waste valorization. In many urban areas however, the efficiency of source separated collection often remains relatively poor, in particular for urban biowaste. The environmental quality of bio-waste is strongly affected by the presence of several undesirable fractions, including in some cases hazardous domestic waste, making it difficult to recycle organic matter for agricultural purposes. Energy recovery on the other hand is less demanding in terms of biowaste quality, and appears more adapted to urban bio-wastes. However, with the exception of the MBT projects for the treatment of residual MSW, there is still little industrial feedback on the process of urban bio-waste conversion to methane. A new strategy to treat urban bio-waste could be the production of a single energy vector, methane. This fuel can indeed be produced using proven technologies: anaerobic digestion and methane production for liquid and easily fermentescible fraction, and thermochemical process combining gasification and methanation of syngas to treat solid fraction diverted to the main stream. The objective of this work was to characterize bio-waste streams produced in an urban territory, with regards to their potential use as feedstock for anaerobic digestion.

1- Introduction

European and French legislation and regulations have been strongly pushing for 3 decades waste production prevention, material and / or energy recovery and minimization of landfilling (Directive 2008/98 / EC). Yet, bio-wastes production is still growing in most cities, following the growth of population (Fisgativa *et al*, 2016; Braguglia *et al.*, 2018). Urban bio-waste include of organic waste produced in urban areas, such as garden and park waste, food and kitchen waste from households, restaurants, caterers and retail premises, and comparable waste from food processing plants, as defined by the Waste Framework Directive (European Union, 2008). In France, the Energy Transition for Green Growth Act (TECV, 2015) has set a series of objectives with respect to waste prevention and bio-waste valorization. In agreement with the European Directives, separated source collection of bio-waste and the implementation of a public service for resources recovery from them will be made mandatory in 2025.

Separation at source helps divert fermentescible organic wastes, in particular from landfill and incineration, thus reducing the overall emissions of greenhouse gases and the negative environmental and health effects related to these waste disposal methods.

Source separation helps divert fermentescible organic waste, in particular from landfilling and incineration (IEA Bioenergy, 2013). In many urban areas however, the efficiency of source separated collection often remains relatively poor. The environmental quality of bio-waste is strongly affected by the presence of several undesirable fractions, including in some cases hazardous domestic waste, making it difficult to recycle organic matter for agricultural purposes. Energy recovery on the other hand is less demanding in terms of biowaste quality, and appears more adapted to urban bio-waste.

Since the control of the environmental quality of bio-waste is more problematic in urban areas than in the agricultural area, particularly because of the heterogeneity of the sources and their natures, material recovery (return to the soil) is more constrained, and restricted to certain particular conditions with short circuit and / or reduced size. The energy sectors are then likely to open up wider and promising recovery prospects.

Among the available and proven energy strategies, anaerobic digestion has been deployed in urban areas on bio-waste mainly for the treatment of residual municipal solid waste RMSW associated with mechanical-biological pre-treatment (MTB) facilities. Anaerobic digestion from readily biodegradable organic fractions produced in urban areas collected separately is still underdeveloped.

For organic fractions that are less easily biodegradable (or whose digestates are unsuitable for return to soil), thermochemical processes appear as complementary approaches to anaerobic digestion. Gasification in particular is a promising approach allowing a higher energy conversion efficiency than combustion even in small units. In addition, gasification allows the production of a synthesis gas that can have various applications including transformation into biomethane *via* the methanation process.

In France, the separate collection of bio-waste only concerned 3% of households in 2011. The organic fraction of municipal solid waste (OFMSW) landfilling is estimated at around 8 Mt per year in France (around 30% of the mass of household waste). The selective collection of OFMSW is developing in several European cities, as reported by the scientific literature (Hansen et al., Bernstad et al., 2010, 2014). Sidaine and Gass (2013) report on the state of the art of separate collection and local bio-waste management. According to the ADEME study mentioned above, it is clear that the national regulatory measures Residual MSW support the development of operations to limit the use of incineration or burial of fermentable organic matter.

However, with the exception of the MBT projects for the treatment of RMSW, there is still little industrial feedback on the process of urban bio-waste conversion to methane. A recent study highlights the influence of collection conditions on bio-waste characteristics (Heaven et al., 2013). The bio-methane potentials (BMP) range from $150 \text{ NL}_{\text{CH}_4} \cdot \text{g}^{-1}_{\text{VM}}$ for RMSW and $600 \text{ NL}_{\text{CH}_4} \cdot \text{g}^{-1}_{\text{VM}}$ for food waste. Some bio-waste, highly biodegradable, because of rich in simple sugars, proteins and lipids are likely to hydrolyze quickly and lose some of their biomethanogenic potential, while others are characterized by a high content of lignocellulosic organic matter, more difficult to biodegrade in anaerobic conditions, for which, in this project we will study the thermochemical pathway.

The present study was based on these considerations. The objective was to characterize biowaste streams produced in an urban territory, with regards to their potential use as energy sources. The approach was to investigate energy recovery through the production of methane as a single energy vector with multiple possible usages including grid injection. Injection of methane into the urban gas grid is an interesting option since dense grids are already in place for natural gas distribution. In most cities, the French law for energy transitions has set on objective of 10% of

methane from renewable sources by 2030, and the French Environmental Protection Agency (ADEME) is expecting more than 55% of renewable gas in French consumption in 2050. Mature technologies are available for methane production from biowaste, but different technologies are to be used depending on waste characteristics'. Anaerobic digestion (AD) is adapted for methane production from liquid and readily biodegradable biowaste fractions, whereas thermochemical gasification followed by methane production from syngas was selected to treat more recalcitrant solid waste fraction.

A methodology was established to select and test the ability of the urban biowaste streams to produce methane by biological or thermochemical processes. The major streams of urban bio-waste were firstly identified on the basis of their quantitative production, and further evaluated with regards to several criteria were considered. A specific fractionation procedure was developed to investigate the distribution of organic constituents between soluble and particulate phases, their respective biodegradability and potential methane production by anaerobic digestion or gasification. Finally, experimental procedure at lab-scale was been developed to determining the technical condition, at lab-scale, of the separation of the liquid fraction dedicated to the bioconversion of methane under anaerobic condition, to the solid residual fraction dedicated to the gasification and methanation of the syngas generated from the gasification.

2- Material and methods

2.1- Bio-waste resources - identification and selection

The biowaste identified exceed the capacity of local treatment facilities. The UrbanBioM project, in its conclusions, will provide decision support for solutions and, ultimately, projects for new recovery facilities to be favored. These elements will also feed into the reflections initiated by Lyon Metropole on the implementation of bio-waste selective sorting by 2025, and the treatment methods to be considered in the case of the capture of part of the bio-waste generated in this urban area.

The urban bio-waste targeted under this project are green waste from private households, and waste generated in municipal parks and in landscape management; domestic bio-waste from food preparation collected at source; bio-waste for catering collected at source (public or private, including schools); bio-waste from the food supply chain, including local agro-food industries and supermarkets.

According to the methodology developed to select the main urban bio-resources, four major bio-waste have been selected for the next steps of our experimental work: green bio-waste (GBW), food waste from households (HBW), food waste from public restauration (RBW), and bio-waste from unsold and breaking the cold chain of supermarket (SMBW).

This selection was based on several criteria:

- Potential mobilizing quantity: take into account the quantitative issues of valorization of the deposit. In fact, the larger the deposit, the greater the value of valuation.
- Availability (dispersion, accessibility, adhesion): take into account the issues related to the effective implementation of the sector and more particularly the collection.
- Territorial orientation and Political Priority: take into account the political and societal issues specific to the territory: political decision, local dynamics, ongoing projects.
- Local context of valuation: take into account the existing sectors established locally (maturity of the processing and valorization, development, implantation projects), and thus preferentially target the flows currently little or not valued.

Four bio-waste were selected for the characterization and waste pretreatment to generate a solid fraction for thermochemical treatment and a liquid fraction for AD:

- Urban Green Waste (GBW) from domestic, municipal and private activities. This urban resource has two strong interests. This is the second flow in terms of tonnage and the GBW have interesting properties allowing to balance a mixture of bio-waste;
- Food waste from restaurants (RBW). The deposit from the school catering and subcontract is relatively easy to access and the quantities are interesting. RBW has been collected from 2 public restaurant facilities located on the campus of our institution;
- Food waste from households (HBW). The important part represented by this bio-waste makes it essential for the project dealing with urban bio-waste. HBW has been collected a set 200 hundred people, faculty members of our institution who separated at source their own food waste;
- Food wastes from Supermarkets (SMBW). This resource also has a fairly large amount of urban biowaste.

The strong presence of unwanted (packaging) in the deposit however requires deconditioning equipment.

For each bio-resource selected, a sampling methodology has been developed, within the framework procedure for the development and implementation of a plan for waste sampling (NF EN 14899, 2006). To guaranty the homogeneity of samples dedicated to the characterization procedure, the biowaste collected were preliminary shredded, immediately after reception in our laboratory. The operation was consisted of a two times shredding with a BLIK BB350 rotary shear crusher before use (8 mm output particle size according to the manufacturer). Samples were stored at 4°C before analysis.

2.2- Bio-waste characterization

The characterization described in this work was based on global procedure described in Figure 1. The fractionation procedure was recently developed in our laboratory to evaluate the potential of organic wastes for AD (Teixeira Franco *et al.*, 2019). This procedure enabled to evaluate the contributions of water-soluble and particulate phases to the investigated properties. For the purpose of this study, it has been done by complementary analyses to obtain other characteristics of the selected bio-waste, including measurement of water retention capacity; particle size distribution under wet conditions; Oxydative Organic Material (OOM), and inert fraction identification (plastics, gravel, glass).

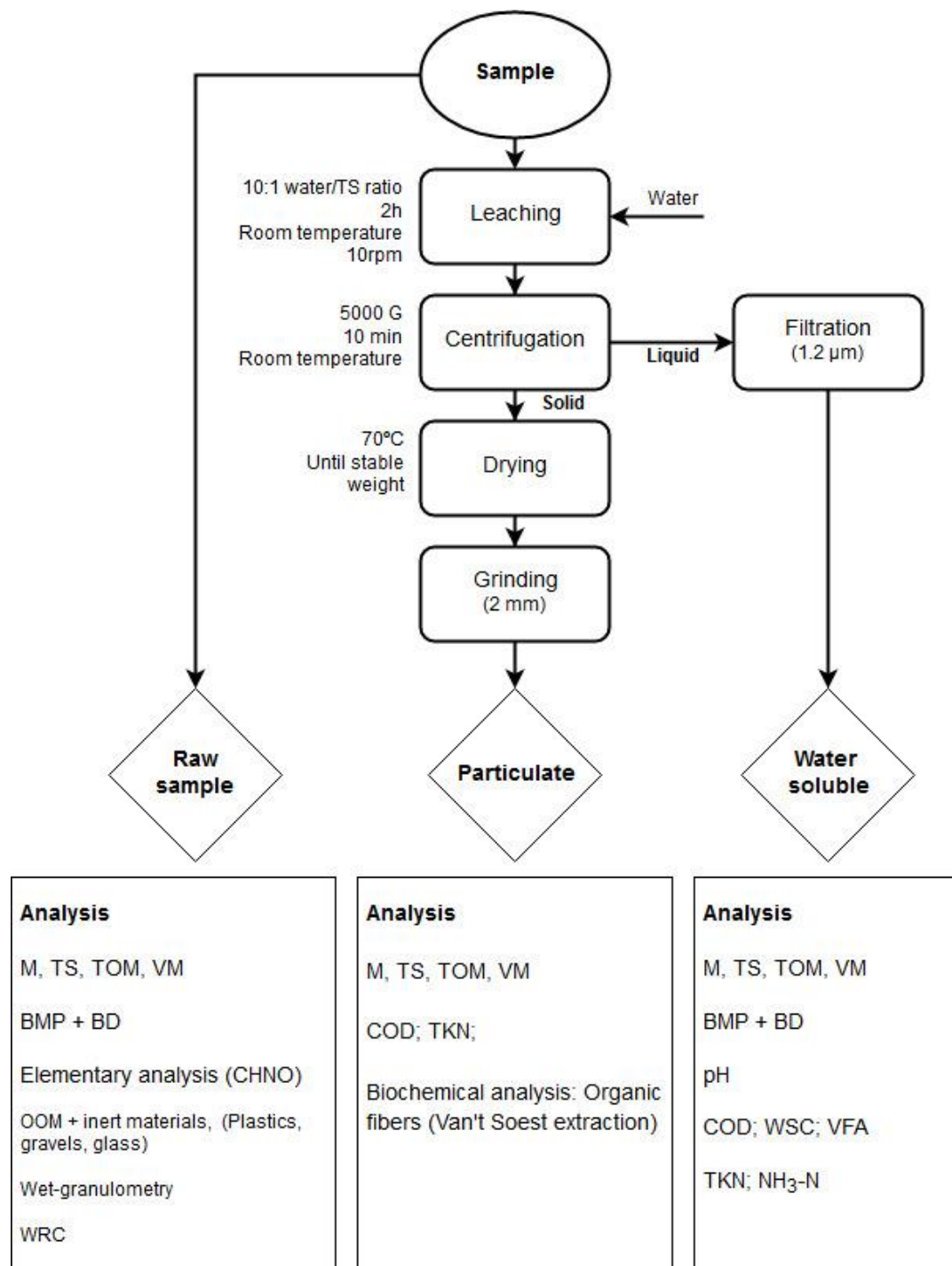


Figure 1: Flowchart of the experimental methodology to characterize biowaste.

Leaching procedure

The developed fractionation procedure was based on water extraction of *raw samples*, according to the NF EN 12457-4 (AFNOR, 2002). The leaching test was performed in triplicate with a standard 10:1 water/TS ratio during 2 h under constant flip-flop rotation (10 rpm) at room temperature. Phase separation was done by centrifugation (5000 G; 10 min), followed by 0.7 mm particle size filtration. Finally, the particulate phase was dried at 70 °C until constant weight and grounded with a Retsch SM 200 cutting mill and a bottom sieve with an aperture size of 2 mm. Raw and water-soluble samples were stored at 4 °C until analysis and particulate ones were stored at -20 °C.

Raw sample analyses

All measurements were performed in triplicate. The total solid (TS) content and Moisture content (M) was measured by weighing the sample (100 g_{WM}), drying the sample at 105°C for 24 h and weighing it again (ISO 11465, 1993). The Volatile Matter (VM) content was measured by weighing a dried sample (20 g_{TS}), calcining the sample and weighing it again. Calcinations were performed at 550°C for 4 h, in a reduction atmosphere (anoxic conditions). Fixed Carbon (FC): relative part of carbon contained in a material that can be degraded only in oxic conditions and high temperature; It is obtained by calculation by deducting from the total material, the percentage of humidity, volatile matters and ash content ($FC = 100\% - M - VS - \text{Ash}$). Total Organic Matters (TOM) consists of the sum of volatile matter and fixed carbon ($TOM = VS + FC = 100\% - M - \text{Ash}$).

Oxydable Organic Matter (OOM) content was determined on raw samples using the gravimetric procedure AFNOR, XP U44-164 (2014) used to identify and quantify inert material quantification in urban compost. This procedure has been used to determine the impurities, which could be present in the four-selected bio-waste. The method consisted of a total chemical oxidation of the organic matter of a dried sample (200 g_{TS}) with sodium hypochlorite. This step was followed by solid matter fractionation by sieving and weighing of inert material (IM), and plastic material, (PM). Finally, the OOM is estimated by the difference: $OOM = TS - PM - IM$. The OOM content can be considered to be representative of the potentially biodegradable organic matter content. For all samples (de Araujo Morais *et al.*, 2008).

The particle size distribution and the Water Retention Capacity (WRC) are of interest to determine the suitability of solid waste for specific pretreatment and sorting method (Tchobanoglous *et al.*, 1993). For determining the particle size distribution, samples (2 kg_{WM}) were sieved under wet conditions with gentle shaking for 1 h through a stack of sieves with seven different size fractions: ≥ 31.5 mm; 31.5-20 mm; 20-10 mm; 10-4 mm; 4-1 mm; 1-0.5 mm; 0.5-0.25 mm; < 0.25 mm. The WRC measurement was consisted to determine the capacity of bio-waste to absorb water in order to evaluate the condition to separate the liquid fraction to the solid fraction, with the perspective to generate two fractions respectively dedicated to AD, and thermo-chemical treatment. The following procedure, column test, has been adapted from the French standard procedure NF U44-175 (AFNOR, 1992).

Finally, BMP on raw samples was also determined following the procedure described on the following specific paragraph entitled biomethane potential measurement.

Liquid phase and particulate phase analyses

Liquid phase collected after the leaching procedure was characterized: TS, TOM, VS and BMP, pH, Water Soluble Carbohydrates (WSC), Volatile Fatty Acids (VFA), Chemical Oxygen Demand (COD), Total Kjeldahl Nitrogen (TKN), and ammonia nitrogen (NH₃-N) were determined in triplicates. Solid particles collected after the leaching procedure was analyzed for its TS, TOM, VS, COD, and TKN.

Cell wall constituents of the organic matter in the particulate phase was achieved by successive steps of hydrolysis and extraction using different solvents. The protocol was developed by Van Soest and Wine (1967), the principle of which has been incorporated in the standard NF FD U44-162 (AFNOR, 2016). It was conducted on aliquots of powdered (grinding < 2 mm) dry samples corresponding to 2 g of volatile matter. At each step, the residual solids were dried and weighed, and their TOM contents analyzed. Four fractions were obtained, namely (1) neutral detergent soluble fraction (SOL) extracted at the first step by a neutral detergent aqueous solution; (2) Hemicellulose-like (HEM) extracted with a dilute acidic aqueous detergent solution; (3) Cellulose-like (CELL)

extracted with a concentrated 72 % sulfuric acid solution; and (4) lignin-like residual organic matter (RES) which was not extracted in the procedure.

Biomethane potential measurement

The BMP tests followed the guidelines provided by Holliger *et al.* (2016), and were conducted in a temperate room at 35 °C using glass vessels of 2 L for raw sample and 0.1 L for water-soluble phase. Vessels were filled with the equivalent 5 g TOM of raw sample or liquid phase obtained from the leaching test, inoculum so as to keep a substrate/inoculum VS ratio of 0.5 and a certain volume of a mineral solution to achieve 60% of the total volume of the vessel. The inoculum used (TS 2.0-3.3%wt; VS 1.4-2.2%wt) was a digested sludge originating from the wastewater treatment plant of La Feyssine, Lyon, France. The mineral solution, which contains essential elements to microbial growth, and also gives the solution a buffer able to control any pH adjustments, was prepared according to the recommendations of ISO 11734 standard (ISO, 1995). Once filled, reactors were purged with a N₂/CO₂ mixture (80/20%v) for about 5 minutes, sealed and equilibrated at 35°C. Blanks with only inoculum and mineral solution were performed for each batch series in order to correct the BMP from residual methane production of the inoculum. All tests were performed in triplicates. Biogas production was determined by pressure measurement using a Digitron precision manometer. Biogas was released when the pressure exceeded 1200 hPa. Gas composition was analysed using an Agilent 3000 micro gas chromatography with 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, with Argon and Helium as carrier gases, respectively. BMP was considered achieved when daily biogas production represented less than 1% of the total volume of biogas produced.

TKN, COD and BMP distribution between solid fraction and liquid fraction

In order to assess the distribution of the various components of interest, some properties were determined through mass balances. For instance, the BMP of the particulate phase was estimated as the difference between the BMP of the raw material and the BMP of the water-soluble phase. On the other hand, COD and TKN of the raw sample were calculated as the sum of the water-soluble and particulate contributions. For instance, non-measured properties BMP and COD were determined with the following mass balances:

$$BMP_P[L_{STP}/kgTOM_{RS}] = BMP_{RS}[L_{STP}/kgTOM_{RS}] - BMP_{WS}[L_{STP}/kgTOM_{RS}] \quad (1)$$

$$COD_{RS}[kg/kgTOM_{RS}] = COD_{WS}[kg/kgTOM_{RS}] + COD_P[kg/kgTOM_{RS}] \quad (2)$$

$$COD_{RS}[kg/kgTOM_{RS}] = COD_{WS}[kg/kgTOM_{RS}] + COD_P[kg/kgTOM_{RS}] \quad (3)$$

Where:

P: particulate fraction;

RS: raw substrate;

WS: water-soluble fraction.

The biodegradability of each fraction was calculated from BMP and COD values considering the theoretical BMP of 0.35 L_{STP}/kg_{COD}, as described below:

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

In order to assess the kinetics of methane production, the experimental data of BMP assays was fitted to the following equation:

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

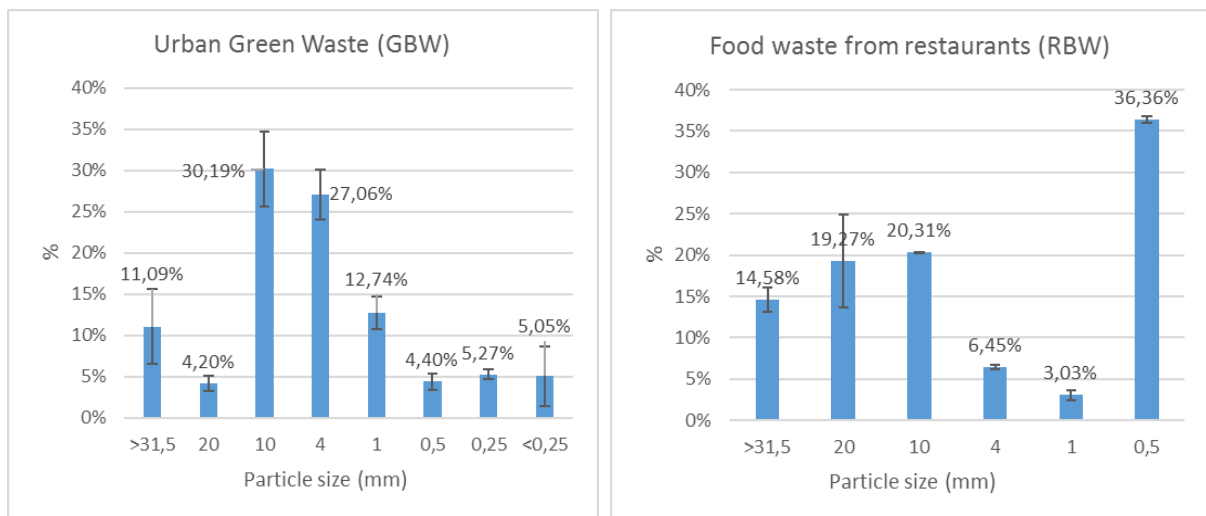
Where, V_{CH_4} corresponds to the volume of methane produced; t is the time of the assay; V_{max} is the maximum volume of methane produced, which was equivalent to the experimental BMP value and; k is the rate coefficient of CH₄ production. Since gas production started almost immediately after reactors were sealed, lag time was not considered in the calculations.

3- Results and discussion

3.1- Global characterization of the bio-waste

Results of particle size distribution analyses of the overall samples are illustrated in Figure 2. Other analytical results are given in Table 1 and illustrated in Figure 3. All analyses were done in triplicates.

The particles size distribution was found to be quite wide in all samples. 60%. Nearly 60% w/w of bio-waste material was present as particles larger than 4 mm, and nearly 40% w/w were below 1mm. Some differences were observed however between the samples. Particles smaller than 0.5 mm were found only in the green waste, probably due to the presence of clay or other fine mineral particles originating from soils. The heterogeneity of the biowaste from supermarket explained the strong dispersion of the data especially at particles sizes above ca. 30 mm, probably related to the presence of packaging type macro-waste.



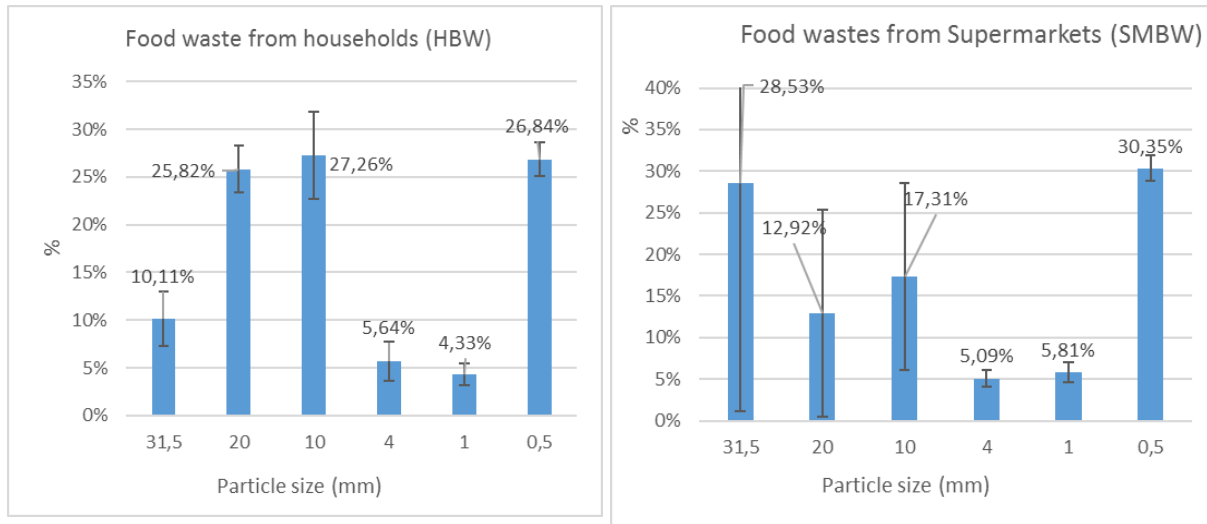


Figure 2: Particle size distribution on the four-selected biowaste. Data expressed based on TS.

Compared to the other biowaste samples, green waste revealed a moisture content ($33\%_{WM}$) twice as less as analyzed in the other samples, and a lower TOM content ($72\%_{MS}$ vs. more than $90\%_{MS}$ in the other samples). GWB also showed the highest contents in inert materials, confirming the probable presence of soil particles already suggested by the particles size analyses. The contents in inert material was around of $3\%_{TS}$ in biowaste from households and biowaste from supermarket biowaste, and close to $5\%_{TS}$ in biowaste from restauration.

The contents in oxidizable organic matter were in good concordance with TOM contents as shown in Table 1, except for supermarket biowaste. This observation was attributed to the presence of close to $40\%_{TS}$ synthetic plastic-type organic materials from packaging. Food waste collected from restaurants, households and supermarkets were more acidic (pH around 4.5) than green waste (pH 7.4), confirming the data reported by Zhang *et al.* (2014), and Fisgativa *et al.* (2016). This observation suggested that the acidogenic phase of anaerobic digestion was already dominant within the sample.

Table 1: Global characteristics of the bio-waste selected. Moisture (M), Total dry Solid (TM), Total Organic Matter (TOM), Volatile Matter (VM), inert material (IM), plastic material, (PM), and Oxydable Organic Matter (OOM).

#	Bio-waste	pH	M (% _{WM})	TS (% _{WM})	WRC (% _{TS})	TOM (% _{TS})	VM (% _{TS})	IM (% _{TS})	PM (% _{TS})	OOM (% _{TS})
G1	Urban Green Waste (GBW)	7.6	33.85	66.15	171	71.71	54.62	30.69	0.05	71.66
G2	Food waste from restaurants (RBW)	4.5	74.42	25.58	311	95.01	80.57	4.95	1.07	94.05
G3	Food waste from households (HBW)	4.7	80.94	19.06	425	90.76	74.88	3.09	0.00	90.76
G4	Food wastes from Supermarkets (SMBW)	4.4	69.19	30.81	2,51	94.24	77.92	2.83	38.95	59.18

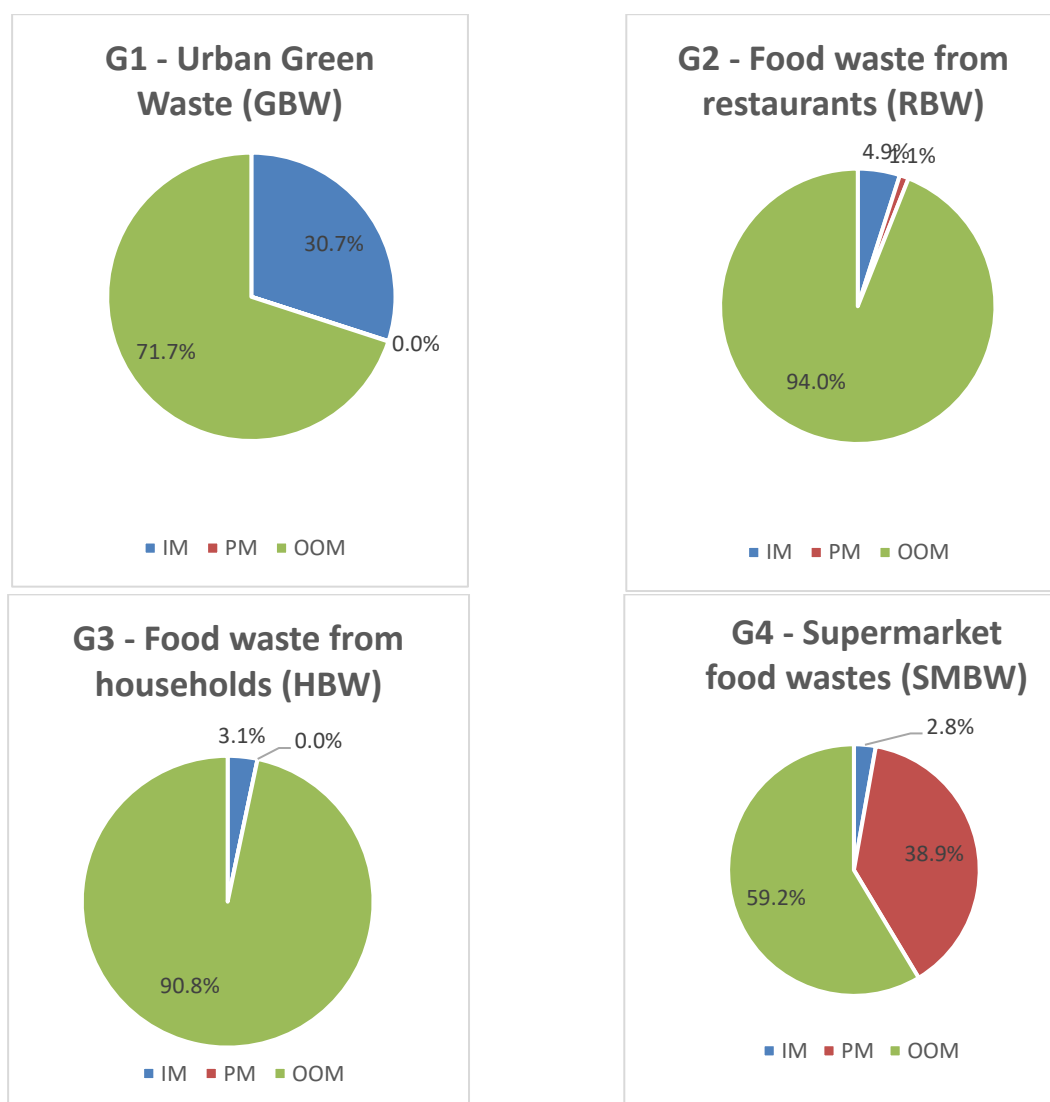


Figure 3: Distribution of impurity levels on the four selected biowaste - inorganic inert materials (IM) or plastics (IM, and non-synthetic oxydisable organic matter (OOM) on the 4 biowaste. Data based on TS.

3.2- Elementary analyses and biochemical characterization of the organic matter

The biochemical compositions of the MOT of the different samples, determined according to Van Soest's sequential extraction protocol, are presented in Table 2. The results confirmed the particular characteristics of green biowaste, whose content in ligno-cellulosic type residual constituents was about 25%_{TOM}, the cellulose corresponding to 44%_{TOM}. In contrast, household biowaste was found to contain nearly 83% of "soluble" organic matter and almost no ligno-cellulosic compounds. Household Food Waste HFW and Supermarket Food Waste SMFW revealed relatively similar biochemical profile with a predominant fraction of "soluble" organic compounds, between 20 and 30%_{TOM} of Hemi-cell and cellulose fractions, and below 10% of ligno-cellulosic type residual constituents.

C, H, and N contents were found to be relatively similar in all the selected biowaste samples. However, a higher ash content was measured in the green biowaste as compared to the other samples, confirming the presence of mineral components probably from soil. C / O and C / N ratios were however different: C / O higher in green biowaste, C / N around 13 in food waste from restaurant RBW, and biowaste from households HBW (richer in

proteins / RBW), 23 in HBW and 31 in green biowaste, due to the predominant presence of plant material rich in hydrocarbon polymers.

Table 2: elemental and Biochemical analysis of the organic matter present in the four bio-waste from the collection campaign.

Bio-waste	C (%TS)	H (%TS)	O (%TS)	N (%TS)	C/O	C/N	Ash (%TS)	Soluble (%TOM)	Hemicelluloses (%TOM)	Cellulose (%TOM)	Residues (%TOM)
Urban Green Waste (GBW)	40.3	4.7	16.6	1.3	36.9	2.4	36.9	18.6	10.3	44.0	27.2
Food waste from restaurants (RBW)	52.6	7.3	29.4	4.0	5.2	1.8	5.2	82.7	4.0	12.6	0.7
Food waste from households (HBW)	47.2	6.2	33.6	2.1	10.2	1.4	10.2	57.5	11.6	21.0	9.9
Food wastes from Supermarkets (SMBW)	50.9	7.0	29.9	4.1	6.3	1.7	6.3	74.6	7.4	11.1	6.9

3.3- Leaching behavior and characterization of the liquid fraction

Table 3 shows the results obtained from the analysis of the aqueous solutions generated after 2 hours of leaching of the different samples. It can be seen that food waste, either from restaurants (RBW), households (HBW), or Supermarkets (SMBW), were characterized as compared to the green waste GBW by pH below 5 in the leachates. This acidity was attributed the presence of free sugars (WSCs, see Table 3) in these waste, which were probably rapidly metabolized into volatile fatty acids (VFAs). This assumption was confirmed by the very high COD measured in the leachates from these samples (nearly 35 g.L⁻¹, i.e. ten times more than in green waste sample GBW) and the analysis of VFAs in the leachates which were found to represent around 10-15% of the leachates COD.

Table 3: Characteristics of the liquid phase after leaching ($L/S = 10$, 2h, T_{amb} , 10 rpm) of the 4 deposits from the first sampling campaign. Data expressed on MS basis (mg / kgTS).

Analyses	Urban Green Waste	Food waste from restaurants	Food waste from households	Food waste from Supermarkets
	GBW	RBW	HBW	SMBW
pH	7,6	4,5	4,7	4,4
Nitrogen				
N-NH ₃ (mg.L ⁻¹)	0	101	79	98
N-TNK (mg.L ⁻¹)	51	1157	240	1165
Soluble organic matter				
COD _L (mg.L ⁻¹) – total COD in solution	2540	34260	36460	34500
VFA (equivalent mg _{COD} .L ⁻¹)	22	3758	7442	6497
% _{COD}	0,9	11,0	20,4	18,8
WSC (equivalent mg _{COD} .L ⁻¹)	229	98	2493	1240
% _{COD}	5,5	0,3	6,8	3,6
Anaerobic biodegradation				
PBM lix (NmL _{CH4} .L ⁻¹)	1160	9220	9370	10800
PBM lix (NmL _{CH4} .g _{COD} .L ⁻¹)	268	260	243	312
BD (% _{CODL})	48,9	76,9	73,5	89,4
k (d ⁻¹)	0,27	0,29	0,40	0,36

Water Soluble Carbohydrates (WSC), Volatile Fatty Acids (VFA), Chemical Oxygen Demand (COD), Total Kjeldahl Nitrogen (TKN), and ammonia nitrogen (NH₃-N)

Ammonia was analysed at low concentrations in the leachates from all samples, representing a very low proportion of the total nitrogen contents estimated by Kjeldahl method (see Table 3). The samples giving the highest soluble N-NTK concentrations were RBW and SMBW with around 1.2 g of nitrogen dissolved per litre of solution. Sample GBW showed lower water-soluble N-TKN concentrations (ca. 0.05 g.L⁻¹). These results suggested that GBW contained less proteins than the other samples.

The methane potentials BMP of the leachates were of the same order of magnitude in food waste samples from restaurants (RBW), households (HBW), or Supermarkets (SMBW). Values close to 10 NL of methane per L of leachate, corresponding to the bioconversion *BD* (%) of 85- 90 of the COD_L, were measured. It was therefore concluded that the dissolved organic matter from these samples was readily biodegradable into methane. The kinetics of biogas production was similar for all the samples..

3.4- Distribution of nitrogen, COD and methane yield (PBM) between the solid (particulate) and the liquid fraction

The results are expressed with respect to the TS content of the soluble and solid fractions obtained by leaching of the different samples, or the TS content of the overall respective samples. The main results are illustrated by Figure 4.

As already observed from the previous analyses, the green biowaste GBW differed here from the other three samples by a very high DCO_L / DCO_S ratio of 26 whereas the other samples ranged between 2.9 to 3.3. GBW also showed lower overall BMP, and a very low BMP of the solid fraction, between 40 and 71 NL_{CH₄} .g⁻¹_{TS}, ie 5 to 10 times lower than observed with the other biowaste. However, the distribution of BMP was relatively similar in the 4 samples.

The overall BMP of food waste from restauration (RBW), households (HBW) and supermarket (SMBW) ranged between 250 and 470 NL_{CH₄}.g⁻¹_{TS}. HBW showed the lowest BMP value, probably in relation to the characteristics of the organic matter contained in this biowaste, as confirmed by the C / N ratio of 22 (see Table 3), which is significantly higher than the ratio determined on RBW (G2) and SMBW (G4), two biowaste apparently richer in nitrogen compounds (proteins of plant or animal origin probably present in significant amounts in these two organic resources). These hypotheses were also confirmed by the analysis of N-NTK in the liquid phase, which was markedly higher for HBW and SMBW (see Table 3). Finally, despite the presence of plastic packaging in SMBW sample (Figure 4Figure 3) the BMP of this sample expressed per unit mass of MS, was close to that of RBW sample. Moreover, the methane bioconversion rate of the total COD is higher to this biowaste: *BD* (%) 77 for RBW and 88 for SMBW, see Table 4. The presence of food waste, rich in compounds such as proteins could explain this high BMP, despite the presence of plastics packaging (accounted for in the measure of total COD).

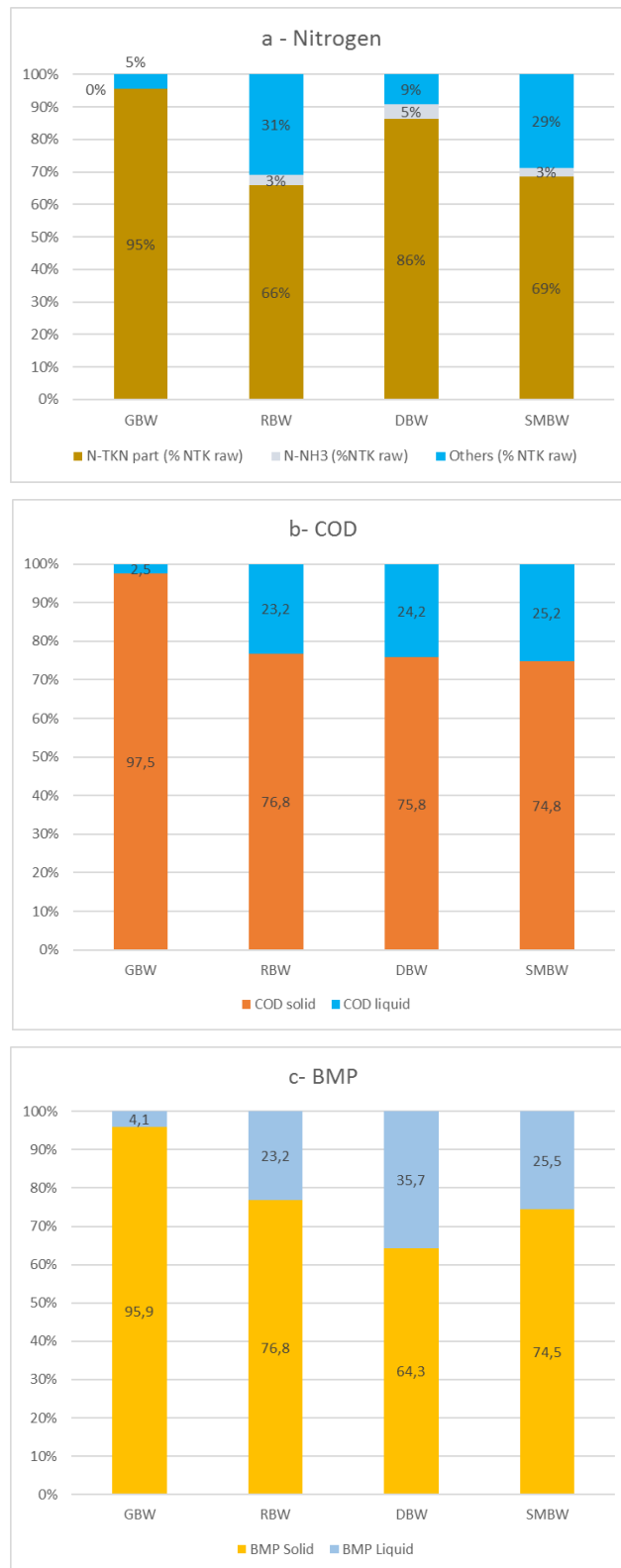


Figure 4: Distribution of Nitrogen (a), COD (b), and PBM (c) on liquid and solid fractions after the leaching test on the four-selected biowaste. Data expressed on TS basis ($\text{mg} \cdot \text{kg}^{-1}_{\text{TS}}$).

Table 4: COD and BMP bioconversion rate on the selected biowaste.

Samples:	Green biowaste	Restauratation. food biowaste	Home food biowaste	Supermarket food biowaste
	GBW	RBW	DBW	SMBW
Raw sample				
COD ($\text{g.kg}^{-1}_{\text{TS}}$)	1035	1477	1505	1372
BMP ($\text{NL}_{\text{CH}_4}.\text{kg}^{-1}_{\text{TS}}$)	31	397	263	450
BD (%)	8.4	76.8	49.9	88.2
Liquid phase (standard leaching on raw sample L/S = 10. 3h. T_{amb})				
COD ($\text{g.kg}^{-1}_{\text{TS}}$)	25	342	364	345
BMP ($\text{NL}_{\text{CH}_4}.\text{kg}^{-1}_{\text{TS}}$ after leaching)	2	92	93	108
BD (%)	17.4	77	74	89
Solid phase after leaching, drying and milling (standard leaching on raw sample L/S = 10. 3h. T_{amb})				
COD ($\text{g.kg}^{-1}_{\text{TS}}$)	1010	1135	1142	1027
BMP ($\text{NL}_{\text{CH}_4}.\text{kg}^{-1}_{\text{TS}}$ after leaching)	29	305	169	316
BD (%)	8.4	77	50	88
COD and BMP ratio between solid to liquid phase				
COD ratio P/S	25.8	3.3	3.1	3.0
BMP P/S	3.2	3.3	1.8	2.9

The 1st order constant k ranged from 0.27 d^{-1} to 0.4 d^{-1} for raw samples, respectively for green biowaste GBW and households biowaste HBW (see Figure 5). As already mentioned in our previous work (Teixera et al., 2019), the comparison of the BMP kinetics is subject to caution, since the anaerobic inoculum used for the BMP tests may not have a similar activity. In the present study, the same inoculum was used to compare the BMP production kinetics of the biowaste. Biowaste with higher contribution of water-soluble phase to the BMP should therefore have a faster anaerobic conversion as it is the case for biowaste from households (Figure 4). Despite the different biochemical characteristics of the biowaste studied here, no significant differences in methane bioconversion rates were observed. Except for green biowaste, 95% of the BMP were expressed in less than 20 days of incubation. This results suggested that foodwaste from restauration, supermarket and households have fairly the same potential of bioconversion in AD.

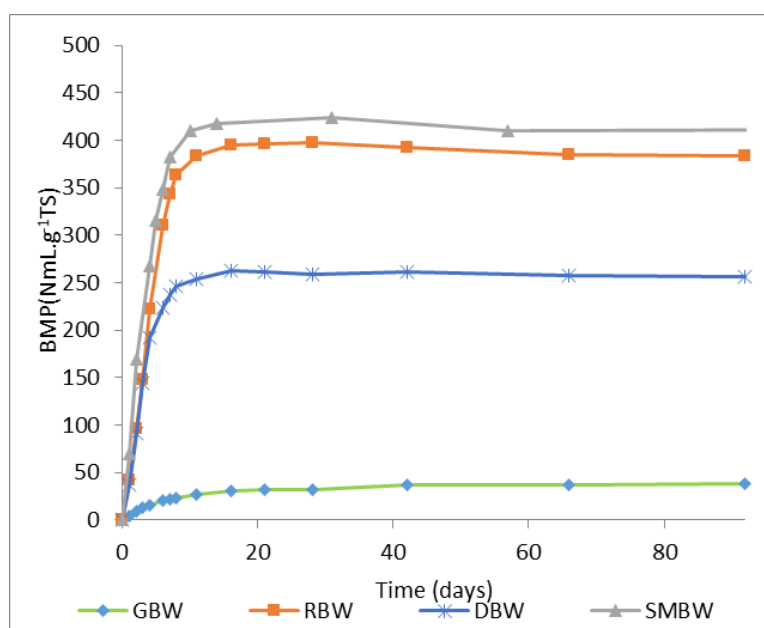


Figure 5: Methane yield in PBM tests on biowaste. Results expressed by NmL of CH₄ per g of TS.

4- Discussion and conclusion

The purpose of this task was to have accurate information on the characteristics of the four biowaste pre-selected for this research project. The data presented highlight several points:

The Green Waste does not have the favorable characteristics for methane bioconversion: soluble fraction that can not be easily mobilized in contact with water. PBM of the particle fraction is much too low to justify its selection for anaerobic digestion. Its high content of inert and mineral materials (nearly one third of the total content) leads to consider it as being unsuitable for recovery by AD.

Despite a lower bioconversion rate, biowaste obtained from a "source" selective collection from households (HBW) remains interesting, since part of the PBM is easily extractable in contact with water - leaching (31%, with a simple contact L / S ratio 10, 2h with gentle stirring), which suggests its selection for AD and a good potentiality of pretreatment for liquid to solid separation.

With its highest BMP potential, biowaste collected from collective catering (RBW) is well suited to recovery methane by anaerobic digestion. The extraction rate of the BMP in contact with water is of the order of 21% (and 23% for COD), requiring the need to pretreat this biowaste in order to extract the PBM predominantly present in the solid fraction;

- The biowaste collected from supermarket (SMBW) differs from the other three deposits with the presence of nearly 40% of plastics from packaging. Despite this, its total BMP is between 450 NLCH₄.kg⁻¹TS, close to those determined for biowaste collected from restaurant, with a high conversion rate to methane, despite the presence of synthetic organic materials. Obviously, this biowaste requires pretreatment operations to separate unwanted fractions, mainly packaging plastics, and to extract the fermentable organic matter in a liquid fraction dedicated to the anaerobic digestion.

The preparation conditions of the four biowaste will be the subject of future research, the objective of which is to determine their suitability for trituration pretreatment (mechanical preparation) and liquid / solid separation in order to produce a solid grade dedicated to thermochemical treatment and a liquid grade, pulp consisting mainly of biodegradable organic material dedicated to anaerobic digestion, wet process.

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