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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 fractioned 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)

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

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

2- Material and methods

2.1- Selection and sampling of relevant biowaste streams

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

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

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

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

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

2.2- Biowaste characterization

2.2.1- Particle size distribution

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

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

2.2.2- Fractionation of biowaste constituents by leaching

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

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

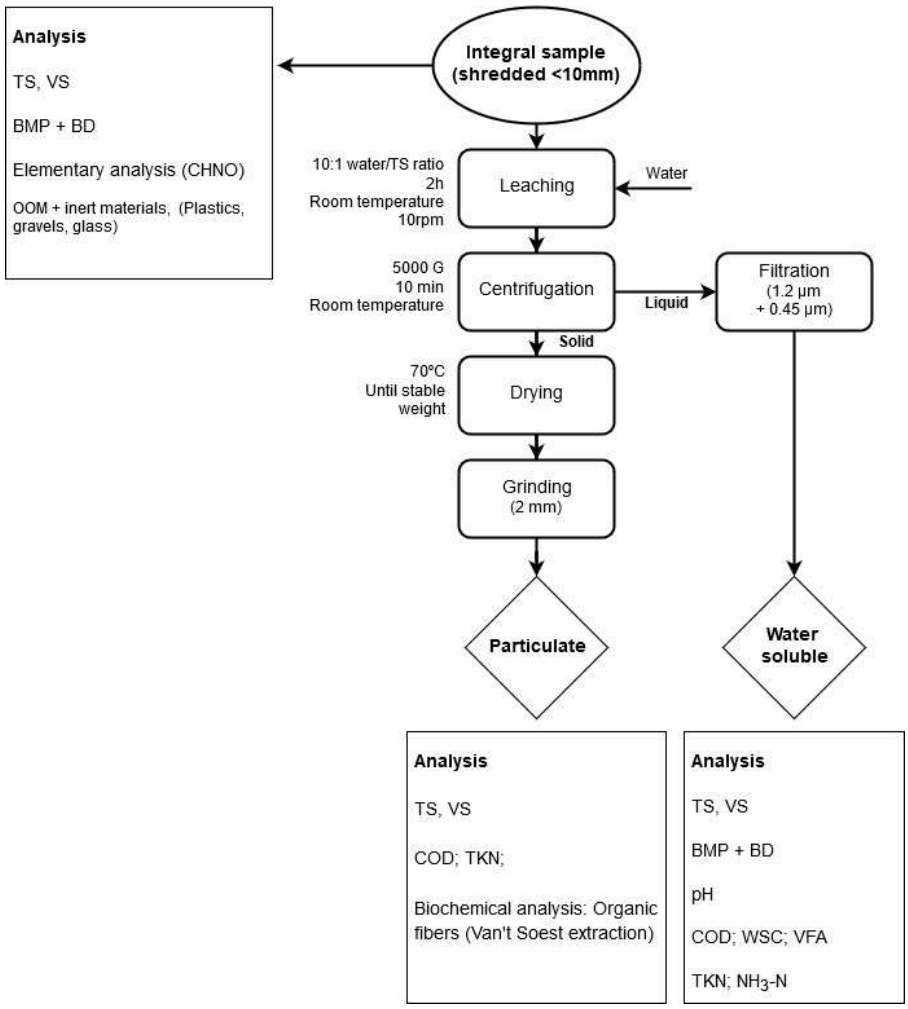

Figure 1: Flowchart of the experimental methodology to characterize biowaste - adapted from Teixeira Franco et al. *(2019). Analyses were perform in triplicate, except for OOM and elementary analysis.*

2.2.3. Physical and chemical analyses of integral solid samples

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

2.2.4. Physical and chemical analyses of particulate fractions

Dried particulate fractions obtained after leaching were analyzed, including TS and VS contents measured in the same condition as integral solid samples. COD, Total Kjeldahl Nitrogen (TKN), and ammonia nitrogen (NH_3-N), were analyzed in triplicates. Cell wall constituents (biochemical composition) were analyzed by successive

hydrolysis steps and extraction with different solvents, according to the protocol developed by van Soest and Wine (1967), following the standard NF FD U44-162 (AFNOR, 2016). Analyses were done on aliquots of powdered dry samples (obtained by grinding down to < 2 mm) corresponding to 2 g of volatile matter. At each step, the residual solids were dried and weighed, and their VS contents analyzed before the solids were used in the next extraction step. 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.

2.2.5. Physico-chemical analyses of aqueous solutions

Aqueous solutions were analyzed in triplicates for TS, VS, pH, Water Soluble Carbohydrates (WSC), Volatile Fatty Acids (VFA), Chemical Oxygen Demand (COD) in water-soluble fraction, Total Kjeldahl Nitrogen (TKN), and ammonia nitrogen (NH_3-N)**.**

2.2.6. Biochemical methane potential - Biodegradability

Biomethane potential (BMP) were measured on the integral waste and the aqueous fraction obtained by leaching (Fig. 1) of each selected biowaste, following the guidelines reported by Holliger *et al.* (2016). The assays were conducted at 35°C in 2 L glass vessels for integral samples and 0.25 L for the water-soluble phase. Digested sludge from the wastewater treatment plant of La Feyssine, Lyon, France was used as an inoculum (TS 2.0-3.3%wt; VS 1.4-2.2%wt) at an inoculum to substrate VS ratio of 2 g/g. A mineral solution providing essential nutrients to microbial growth and a buffer solution were added to the reactors according to the recommendations of ISO 11734 standard (ISO, 1995). Once filled, reactors were purged with a N_2/CO_2 (80/20% v/v) gas flow for about 2 minutes, sealed and equilibrated at 35°C. Blanks containing only the inoculum and the mineral solutions were systematically monitored along with each series of assays in order to correct the recorded 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. The tests were stopped when the daily biogas production was less than 1% of the total volume of biogas produced.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

2.3- Sampling, analyses and data treatment strategies

Fractionation by leaching was perform twice on each integral waste. Depending of TS content, first fractionation was perform with 100 to 300 g $_{\text{int. biowaste}}$ and second fractionation with 450 to 1500 g $_{\text{int. biowaste}}$. Sample were taken from integral biowaste using quartering method to ensure homogeneity.

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

3- Results and discussion

3.1- Physical and chemical analyses of integral biowaste

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

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

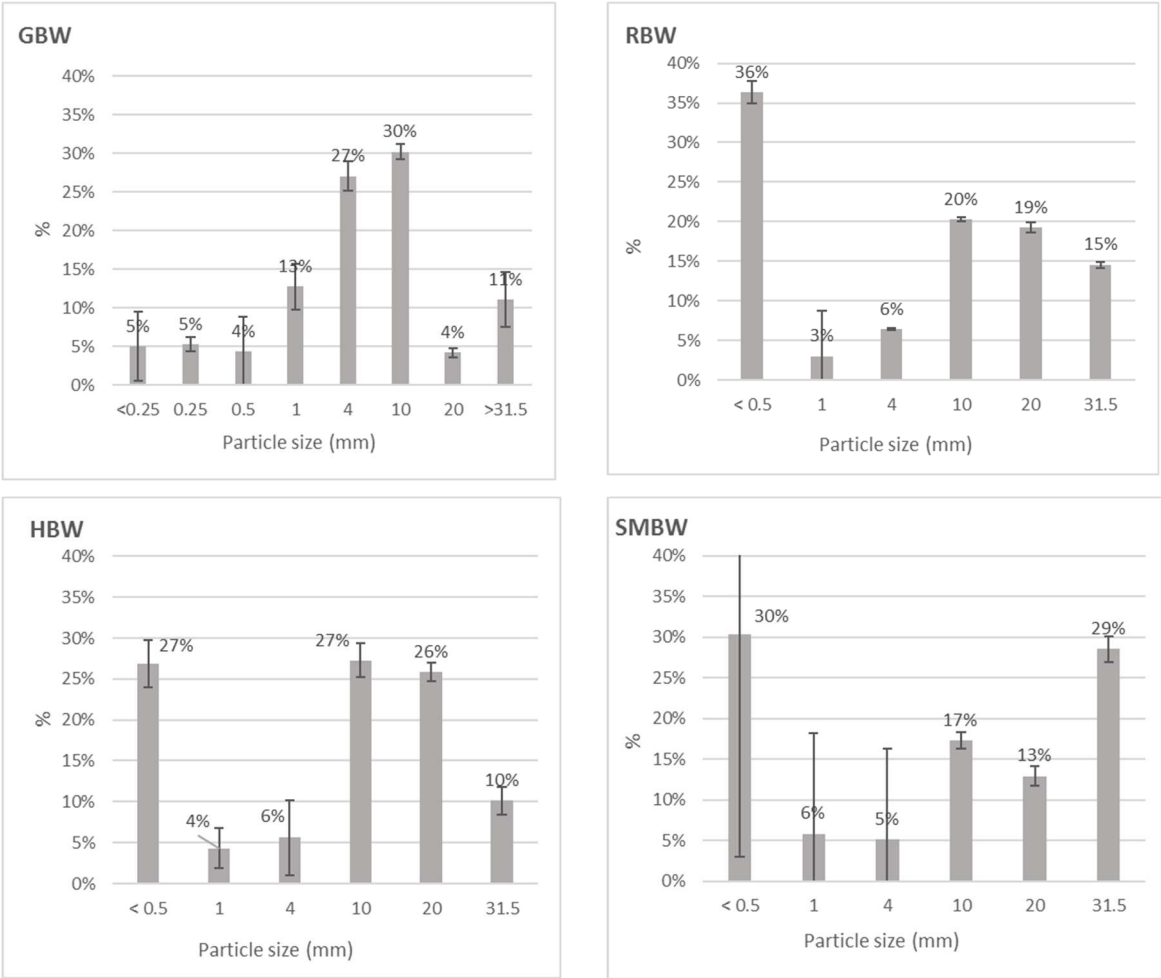

Figure 2: Particle size distribution on the four-selected biowaste. Data expressed on a TS basis. GBW: Garden biowaste; RBW: Restaurants biowaste; HBW: Household biowaste; SMBW Supermarkets biowaste. Result are average values of triplicated assays. Vertical bars show standard deviations.

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

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

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

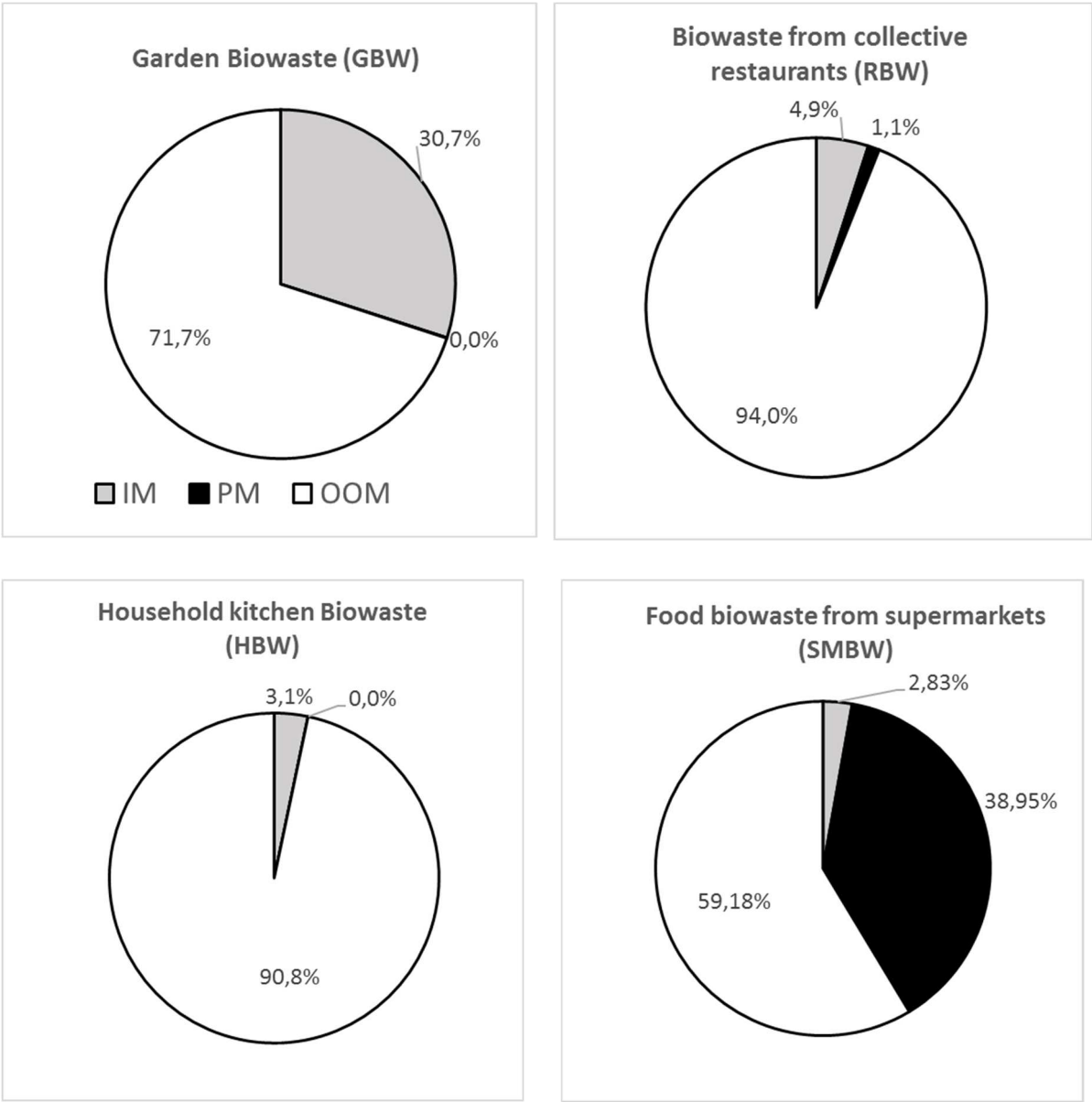

Figure 3: Impurity contents in the four selected biowaste (expressed on a TS basis): inorganic inert materials (IM), plastic materials (PM), and non-synthetic oxidizable organic matter (OOM).

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

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

Table 1: Total Solid, Volatile Solid, Elemental and biochemical analysis of the organic matter present in the four biowaste from the collection campaign. GBW: Garden biowaste; RBW: Restaurants biowaste; HBW: Household biowaste; SMBW 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*

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

3.2- BMP and biodegradability of the integral biowaste

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

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

Table 2 : Methane potentials and 1st order kinetic constants of its production from the four selected biowaste, calculated from the curves shown in Fig. 4. GBW: Garden biowaste; RBW: Restaurants biowaste; HBW: Household 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
k (d^{-1})	*0.137 ± 0.020*	*0.209 ± 0.004*	*0.275 ± 0.005*	*0.269 ± 0.001*

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

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

3.3- Fractionation of biowaste constituents by leaching in water

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

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, room temperature, mixing at 10 rpm. GBW: Garden biowaste; RBW: Restaurants biowaste; HBW: Household biowaste; 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				
$N\text{-}NH_3$ ($mg.L^{-1}$)	< 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
k (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***

Water Soluble Carbohydrates (WSC), Volatile Fatty Acids (VFA), Chemical Oxygen Demand (COD), Total Kjeldahl Nitrogen (TKN), and ammonia nitrogen ($N\text{-}NH_3$). < DL: below the detection limit.

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

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

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

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

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

The distribution of the BMP between particulate and water-soluble (leachate) phase was also very different in GBW with a BMP S/L ratio of 17 whereas the ratio ranged between 2 and 3 for the other samples. GBW also showed by far the lowest BMP values both for the integral sample (36 $NL_{CH4}.kg^{-1}_{TS}$) and the solid fraction (34 $NL_{CH4}.kg^{-1}_{TS}$) which were 5 to 10 times lower than observed with the other biowaste.

Nitrogen distribution show more than 30% of total nitrogen in soluble phase (< 0.45 µm filtration) for RBW and SMBW. GBW and HBW have a poor nitrogen soluble phase with respectively 5% and 14 % of total distribution.

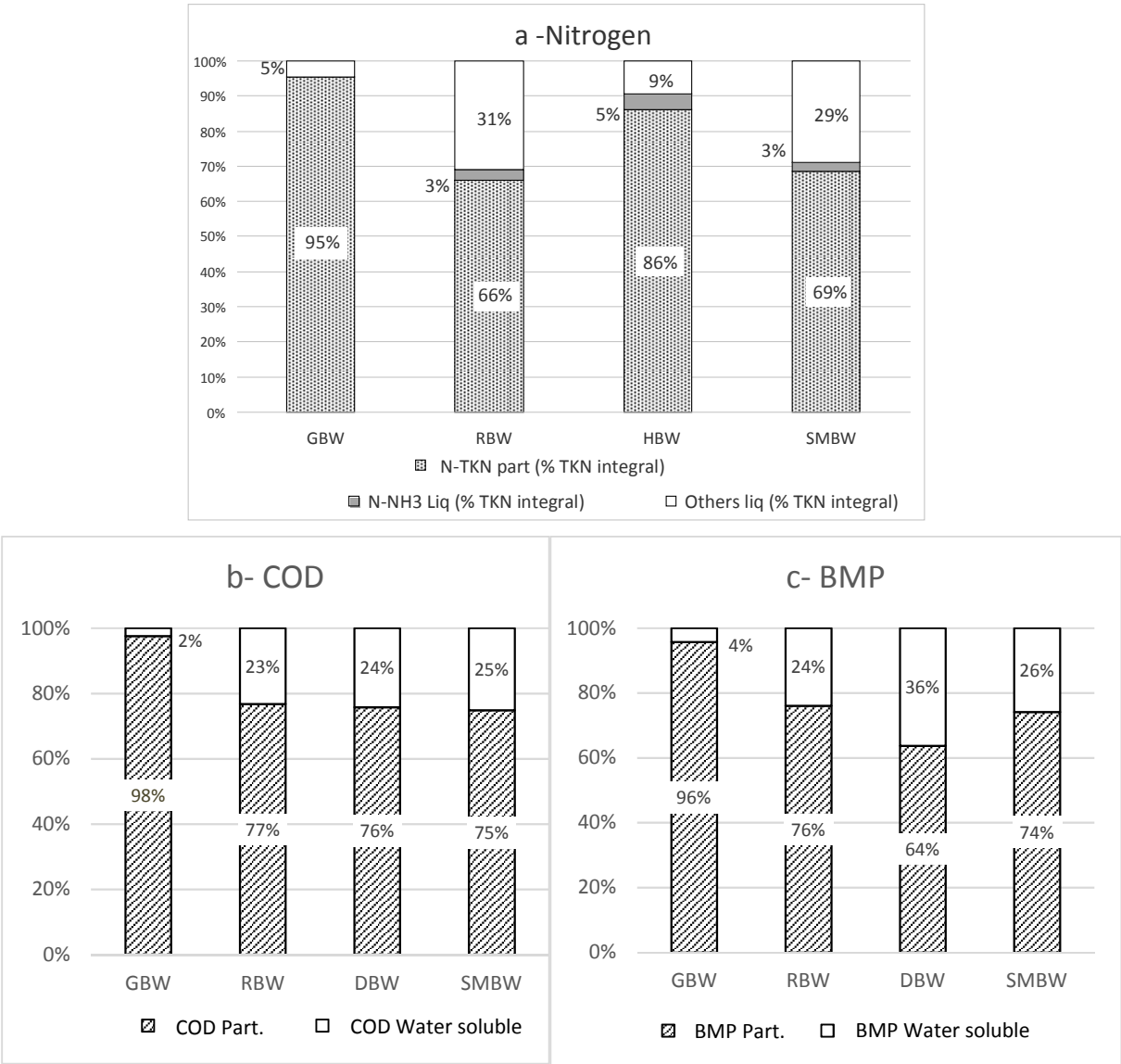

Figure 4: Distribution of Nitrogen (a), COD (b), and BMP (c) between water soluble and particular fractions obtained by leaching Garden biowaste (GBW), Restaurant biowaste (RBW), Household biowaste (HBW) and Supermarket biowaste (SMBW). Result are average values of triplicated assays.

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

		GBW	**RBW**	**HBW**	**SMBW**
Integral sample					
COD	$g.kg^{-1}_{TS}$	1035 ± 9	1477 ± 44	1506 ± 27	1372 ± 32
BMP	$NL_{CH4}.kg^{-1}_{TS}$	36 ± 7	385 ± 9	258 ± 14	417 ± 11
BD	***%***	***10 ± 0.1***	***75 ± 2.2***	***49 ± 0.9***	***87± 2.0***
N-TKN	$gN.kg^{-1}_{VS}$	15.6 ± 0.3	35.8 ± 0.6	19.2 ± 0.3	39.4 ± 1.0
Water-soluble phase					
COD	$g.kg^{-1}_{TS}$	25 ± 1	343 ± 2	365 ± 2	345 ± 2
BMP	$NL_{CH4}.kg^{-1}_{TS}$	1 ± 0.1	92 ± 1	94 ± 4	108 ± 5
BD	***%***	***17 ± 0.3***	***77 ± 0.9***	***74 ± 3.3***	***89 ± 3.1***
N-TKN	$gN.kg^{-1}_{VS}$	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

*calculated with equation (3)

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

4- Conclusion

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

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

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

In contrast, RBW, HBW and SMBW were identified as good candidates for pretreatment operations to convert them into an aqueous slurry, with good characteristics for AD, and a particulate fraction with very different characteristics that would be adapted to gasification. HBW and RBW exhibited high availability of organic matter. BMP and COD concentration observed was high in leachate fraction indicate high easily water-soluble component. Supermarkets biowaste (SMBW) differed from the other three biowaste samples by the presence of nearly 40% of plastics from packaging. These undesirable materials should be removed from the biowaste material prior to biological treatment. However, this operation would need the implementation of adapted separation techniques due to the wide distribution of particle sizes and the variety of nature and composition of the undesirable particles. Despite this unfavorable characteristic, the BMP of the biowaste material (separated manually in the present study from the plastic materials) was around 450 $NLCH_4.kg^{-1}_{TS}$, which was close to that obtained for RBW, with high methane production kinetics. Similar to HBW, SMBW would require pretreatment operations to separate undesirable fractions, mainly packaging plastics, and extract the biodegradable organic matter in a form of an aqueous pulp dedicated to the anaerobic digestion.

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

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