

# Agricultural waste extensive mechanical pretreatment at lab-scale before anaerobic digestion: An integral approach

H. Coarita Fernandez<sup>1</sup>, P. Buffiere<sup>1</sup> and R. Bayard<sup>1</sup>

<sup>1</sup>Univ. Lyon, INSA Lyon, DEEP Laboratory, EA7429, F-69621 Villeurbanne cedex, France

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Presenting author email: helen.coarita@insa-lyon.fr

## Introduction

In France, about 65% of anaerobic digestion sites use agricultural waste, green waste or municipal wastewater as main substrate. Indeed, France produce a considerable amount of feedstock of manure and crop residues to be converted into energy (1). More of 70% biogas produced in Europe comes from anaerobic digesters using agricultural wastes, manure and energy crops as input (2). Despite the feedstock availability and knowledge on anaerobic digestion process, there is still some limitations and restraints in profit to treat a large range of biomass. Pretreatments are often used in order to improve biomass bioaccessibility and bioconversion to methane. Mechanical pretreatments seem to be the pretreatments most used at full-scale (3,4). They are mainly conceived for avoiding feeding problems on the reactor (5). In addition Tsapekos *et al.* (6) reported an increase on methane production rate and yield. Indeed, mechanical pretreatments could be advantageous but a highly energy requirement could be needed (7). The main objective of this research work was to investigate three successively mechanical pretreatments effects at lab-scale on three types of feedstock: cattle manure, maize silage and a mixture of cattle manure and maize silage. A physical and biochemical characterization methods were carried out in order to elucidate a better understanding of mechanical pretreatments application on different substrates.

## Material and methods

*Feedstock:* Agricultural wastes as cattle manure, maize silage, and a mix of cattle manure and maize silage were collected from a biogas unit located in the Auvergne-Rhône-Alpes Region of France. These agricultural biomass are used as main inputs on the site in order to convert organic matter to methane, and producing heat and electricity. The samples were stored at 4°C in a cold chamber and immediately analyzed.

*Mechanical pretreatments:* Three successive mechanical pretreatments were performed at lab-scale on different biomass samples. First at all, samples were shredded with a BLIK BB350 rotary shear crusher. Then, samples were mixed during five minutes. As part of this second pretreatment, water was added in a ratio proportion of 10 of water/dry matter). Finally, samples were blended during five minutes as well.

*Biochemical and physicochemical characterization:* An integral approach of biochemical, physical and physicochemical characterization was performed before and after each pretreatment for all feedstocks selected for this study. A fractionation of organic matter was carried out in order to check up organic compounds solubilisation and their evolution in the particulate and liquid fraction as described by Teixeira Franco *et al.* (8). The procedure consists in a sample fractionation over a water extraction after a leaching test. Leaching was made with 10:1 water/dry matter proportion during 2h under a constant bottle rotation. Afterwards, samples were centrifuged (20 min at 5000 G) and 1.2µm filtered. Parameters like chemical oxygen demand (COD), Total Kjeldahl Nitrogen (TKN), volatile fatty acids (VFA), water-soluble carbohydrates (WSC) were analyzed on the particulate and the liquid phase. Biomethane Potential (BMP) test assays were carried out for the liquid phase, and untreated sample. In addition, physical characteristics were evaluated, including the particle size distribution, the Water Retention Capacity (WRC), and the rheological properties.

## Results and discussions

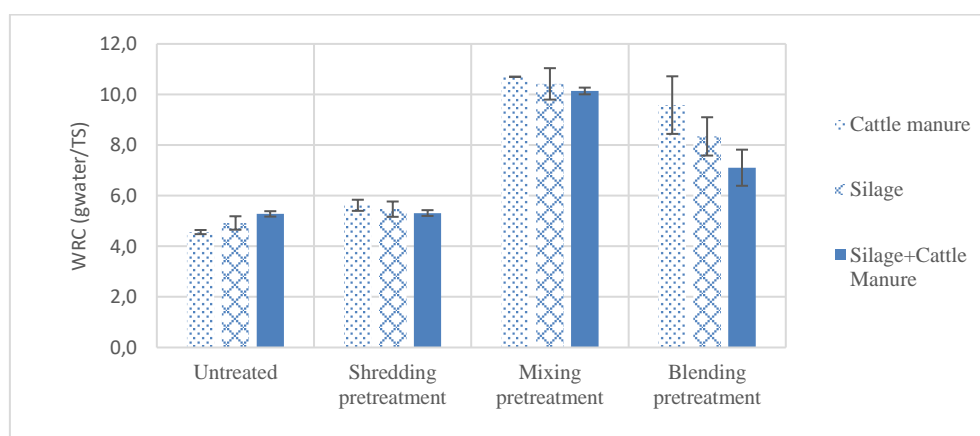
The main characteristics of untreated and every pretreated biomass feedstock samples are described on Table 1. Pretreatments did not a led a significant increase (about 10%) on methane yield (BMP) for cattle manure, silage and cattle manure + silage substrate. An increase more interesting on methane yield was found, for cattle manure substrate on blending pretreatment (32% compared to untreated samples), for silage substrate on shredding pretreatment (26% compared to untreated samples) and for silage+cattle manure on blending pretreatment (90% compared to untreated samples).

**Table 1.** Main characteristics of untreated and treated feedstock.

Cattle Manure					
Parameters	Unit	Untreated-CM <sup>1</sup>	CM <sup>1</sup> -SP <sup>2</sup>	CM <sup>1</sup> -MP <sup>3</sup>	CM <sup>1</sup> -BP <sup>4</sup>
TS	% (total weight)	29.9±0.1	29.1±0.2	9.05*±0.2	9.1*±0.04
VS	% (TS)	78.6±0.6	80.9±0.8	78.03±0.4	76.7±2.2
pH	-	8.33	8.44	8.39	8.34
BMP <sub>RS</sub> <sup>(7)</sup>	mL/gVS <sub>RS</sub>	220±8	211±6	217±3	227±7
k <sub>RS</sub> <sup>9</sup>	j <sup>-1</sup>	0.056±0.002	0.061±0.003	0.069±0.005	0.074±0.004
BMP <sub>WSF</sub> <sup>(8)</sup>	mL/gVS <sub>RS</sub>	33.05±0.4	37.5±0.5	35.5±1.2	44.2±0.6
WSP contribution	% BMP <sub>RS</sub>	15.01±0.7	17.5±0.7	16.4±0.8	19.6±0.8
Maize Silage					
Parameters	Unit	Untreated-S <sup>5</sup>	S <sup>5</sup> -SP <sup>2</sup>	S <sup>5</sup> -MP <sup>3</sup>	S <sup>5</sup> -BP <sup>4</sup>
TS	% (total weight)	16.03±0.1	16.6±0.1	9.8*±0.2	9.2*±0.04
VS	% (TS)	83.9±0.4	83.9±0.3	84.4±0.3	84.2±0.1
pH	-	5.57	5.53	5.57	5.54
BMP <sub>RS</sub> <sup>(7)</sup>	mL/gVS <sub>RS</sub>	338±18	339±6	312±19	364±8
k <sub>RS</sub> <sup>(9)</sup>	j <sup>-1</sup>	0.197±0.008	0.25±0.005	0.22±0.01	0.22±0.01
BMP <sub>WSF</sub> <sup>(8)</sup>	mL/gVS <sub>RS</sub>	94.1±6.6	110.5±1.4	115.9±0.4	111.5±2
WSP contribution	% BMP <sub>RS</sub>	27.8±3.1	32.6±0.6	35.9±2.9	30.6±1
Silage + Cattle manure					
Parameters	Unit	Untreated-SCM <sup>6</sup>	SCM <sup>6</sup> -SP <sup>2</sup>	SCM <sup>6</sup> -MP <sup>3</sup>	SCM <sup>6</sup> -BP <sup>4</sup>
VS	% (TS)	78.5±0.6	76.8±2.5	77.3±0.5	78.2±0.4
pH	-	8.3	8.02	7.97	7.72
BMP <sub>RS</sub> <sup>(7)</sup>	mL/gVS <sub>RS</sub>	180±7	198±2	195±4	188±7
k <sub>RS</sub> <sup>9</sup>	j <sup>-1</sup>	0.070±0.011	0.129±0.001	0.133±0.006	0.133±0.008
BMP <sub>WSF</sub> <sup>(8)</sup>	mL/gVS <sub>RS</sub>	7.6±0.3	39.2±0.4	34.7±1.2	40.8±3.05
WSP contribution	% BMP <sub>RS</sub>	4.2±0.3	18.04±0.4	17.8±0.9	21.8±2.2

<sup>1</sup> CM: Cattle manure <sup>2</sup>SP: Shredding pretreatment <sup>3</sup>MP: Mixing pretreatment <sup>4</sup>BP: Blending pretreatment <sup>5</sup>S: Silage SCM: <sup>6</sup>Silage+Cattle Manure <sup>7</sup> WSP: Water-soluble phase <sup>8</sup>RS: Raw Sample <sup>9</sup>k: kinetics constant  
\*TS contents after water addition for pretreatment

Water Soluble Fraction contribution to BMP was slightly increased with pretreatments mainly on cattle manure and silage + cattle manure samples. For silage samples, the water soluble fraction contribution was not proportionally correlated with pretreatments, a higher contribution to BMP was found on mixing pretreatment.



**Figure 1.** Water Retention Capacity of evaluated biomass feedstock.

Physical characteristics were also analysed on three substrates. Water Retention Capacity (WRC) was improved with pretreatments mainly on cattle manure substrate while silage and silage + cattle manure, WRC was increased with shredding and mixing pretreatment and it was decreased with the blending pretreatment. Considering this result an exhaustive pretreatment on silage substrate could not necessarily improve the physical properties.

### **Conclusions**

According to our results, mechanical pretreatment was able to optimize the biochemical characteristics. Water soluble COD phase and methane production were enhanced, and the bioconversion to methane. Physical characteristics were also strongly modified, in particular the water retention capacity, but also the particles size distribution. Complementary results will be presented to illustrate the influence of mechanical pretreatments on the rheological characteristics of the feedstocks.

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