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Highlights

- Dry co-digestion of roadside grass with cattle manure was performed in batch.
- Filling as layers or as a blend impacted the hydrodynamics and CH_4 production.
- A seasonal impact of methane production was observed.

Abstract

Roadside grass cuttings and solid cattle manure are resources that are available as input for dry anaerobic co-digestion. Two series of measurements were carried out, one in June 2016 and one in October 2016. The methane potentials were determined on a laboratory scale and revealed a high degree of seasonality, 202.9 and 167.9 $Nm^3_{CH4}.t_{VS}^{-1}$, respectively. Moreover, these substrates were co-digested in reactors by the dry process on a pilot scale (60 L). Two

strategies for filling and optimization, as layers or as a mixture, were compared. The seasonality also determined the physicochemical parameters and the hydrodynamic properties involved in percolation of the liquid phase recirculated in the dry digestion process. The production of methane depended on the filling method, the seasonality, and the nature of the input, which in some cases resulted in inhibition of 34.8 to 44.4 $Nm^3_{CH4}.t_{VS}^{-1}$.

Keywords

Co-digestion - Dry batch anaerobic digestion – Roadside grass - Cattle manure - Biogas

1. Introduction

The use of renewables resources to produce energy is a new approach aimed at meeting increasing energy requirements. Anaerobic digestion is being applied in a range of processes and it has become a key part of renewable energies. Microorganisms can consume organic material by anaerobic digestion, thereby producing biogas composed of methane (50-70%) and carbon dioxide (30-50 %). The methane that is produced can be valorised either by the cogeneration of heat and electricity or by injection into gas networks, thereby reducing the use of fossil fuels. Anaerobic digestion can be carried out according to two main types of procedures, namely dry processing of waste with a total solid content greater than 15% or liquid processing of waste with a total solid content < 15% (Li et al., 2011; Karthikeyan, and Visvanathan, 2013; Ge et al., 2016; André et al., 2018). Although the dry process is, in principle, more suitable for wastes with a high total solid content, scientific and technological obstacles have, however, limited its uptake (André et al., 2018). Based on the data in the literature, there appears to be more merit in focusing on trials of anaerobic digestion when the substrates have a dry weight content greater than 15% (André et al., 2018).

Dry batch anaerobic digestion employs a bulk phase comprised of solid waste and a liquid phase that is periodically recirculated on this bulk material (Ge et al., 2016; André et al., 2018). Le Hyaric et al., (2011) have shown a linear relationship between the water content and the activity of the methanogenic populations. Recirculation allows transmission of the microorganisms, the temperature, the humidity, *etc*. Placing the material to be degraded in contact with the microorganisms maximizes of the quantity of biogas that can be produced (El-Mashad et al., 2006; Kusch et al., 2009; Benbelkacem et al., 2010; Shahriari et al., 2012; André et al., 2015; Degueurce et al., 2016b). Liquid and hydrodynamic transfers are key aspects of this process. Pilot trials (in the order of 60 to 100 L) using different methodologies have led to similar conclusions. The degradation of organic matter results in a change in the

hydrodynamic parameters over the course of anaerobic digestion (Shewani et al., 2015; André et al., 2015; 2018).

To date, there has been scant documentation of co-digestion by the dry process, unlike co-digestion by the liquid process (Mata-Alvarez et al., 2000; Parawira et al., 2004; Marcia-Corral et al., 2008; Ahn et al., 2010; André et al., 2018). To properly manage co-digestion by the dry process, the nature of the input needs to be taken into consideration. Indeed, inhibition is a common occurrence and needs to be managed to ensure optimal methane production. During inhibition, the equilibrium necessary for proper functioning of the methanogenic populations is perturbed. The ensuing lack of or limited production of methane reduces the potential of methanogenic populations to produce energy. Inhibition can result in the accumulation of volatile fatty acids, nitrogen, ammonia, nitrites, nitrates, salt, antibiotics, *etc*. (Mata-Alvarez et al., 2000). Trials with different types of manures (dairy, poultry, or swine manures) in co-digestion with switchgrass have been carried out in 1 L reactors by the dry process under thermophilic conditions. Inhibition with co-digestions using poultry and cow manure can be evidenced by the accumulation of volatile fatty acids (Ahn et al., 2010). Various strategies have been applied to optimize and to ensure the production of biogas. Management of certain inhibitions can be achieved by the use of highly fermentable substrates with less fermentable substrates or digestate to buffer the medium (Mata-Alvarez et al., 2000; Ward et al., 2008; Li et al., 2011; Khalid et al., 2011; Karthikeyan and Visvanathan, 2013; Ge et al., 2016; André et al., 2018). The SEBAC (Sequential Batch Anaerobic Composting) methodology applies liquid exchanges between stabilized reactors and other non-stabilized ones containing a high level of volatile fatty acids, thereby allowing digestion of highly fermentable substrates (Chugh et al., 1999; Chynoweth et al., 1991; Foster-Carneiro et al., 2004; 2007).

Although roadside grass is a readily available resource, it has not been used much to date, often being left in place once cut. The logistics of grass clippings have not been developed to

date to collect this resource that is amenable to valorisation by anaerobic digestion. Several European projects and studies have taken place to evaluate and optimize collection and to promote the use of roadside grass as input for anaerobic digestion (GR3, 2013-2016; DeLand, 2014-2015; Meyer et al., 2014; Piepenschneider et al., 2016).

In France, co-digestion of roadside grass and cattle manure is a way to optimize biogas production by agricultural biogas plants. However, this type of feedstock has seen little or no use to date despite the potential availability of large amounts of this resource. The addition of the roadside grass can increase biogas production, thereby ensuring greater profitability of biogas plants. As roadside grass has a TS content greater than 15% (Meyer et al., 2014), dry anaerobic digestion is, therefore, appropriate to digest this co-product.

The aim of this study was to valorise roadside grass by dry anaerobic digestion. Valorisation of roadside grass was studied by co-digestion with cattle manure. Two series of tests were carried out in June 2016 and in October 2016 to observe the impact of seasonality on anaerobic digestion. Physicochemical parameters were analysed to compare the seasonality of the input. The methane potential by the liquid process was first determined on a laboratory scale (500 mL). Three co-digestion blends by the dry process were then tested at a pilot scale level (60 L), with modulation of the proportion of roadside grass and of cattle manure in the reactors. Two means of filling were also employed, either as layers or as a mixture. Thus, six reactors of 60 L were studied. Comparison of the results for the different series of measurements allowed the impact of the seasonality on the hydrodynamics of the medium and the production of methane to be observed.

2 Materials and Methods

2.1 Analytical methods

2.1.1 Physicochemical parameters

The total solid (TS) and volatile solid (VS) contents were determined by drying the respective substrates at 105 °C for 24 h and at 550 °C for 2 h (APHA, 1988). The analyses were carried out in duplicate.

The pH of the *inoculum* was determined using a pH meter (Mettler Toledo, Switzerland). The analyses were carried out once a day throughout the anaerobic digestion process.

Determination of the buffer capacity and the total volatile fatty acid content was performed by two acidifications using sulphuric acid. The first acidification down to pH 5.0 allowed the buffer capacity (TAC) to be determined, and the second acidification down to pH 4.4 allowed the quantity of volatile fatty acids (FOS) to be determined. These analyses were carried by means of an automatic titrator (Mettler Toledo, Switzerland), and they were performed daily throughout the anaerobic digestion process.

2.1.2. Fibre quantification

The method of van Soest was used to determine the fibre content of the input and of the digestates. A solid and crushed sample that had been dried at 70 °C for 48 h was subjected to various hydrolyses to determine the fibre content. The first hydrolysis was by a solution of neutral detergent (NDF), followed by wash phases and a drying phase. The second hydrolysis (ADF) by an acidic solution was carried out on this insoluble residue, followed by washing and a drying phase. The residue obtained was treated with sulphuric acid at 72% (ADL), followed by rinsing and drying. Calcination allowed quantification and removal of the mineral fraction. Successive weightings between each step allowed the fraction of each component to be obtained. The fibre content was expressed as a percentage and related to the total solid content of the samples (van Soest et al., 1991). These analyses were performed in triplicate. The fibre content was calculated as follows:

NDF – ADF = hemicellulose content Eq. (1)

ADF – ADL = cellulose content Eq. (2)

ADL = lignin content Eq. (3)

2.2 Inocula and substrates

Liquid manure obtained from the farm of the UniLaSalle Polytechnic Institute was used as the *inoculum*. The liquid manure pit of the farm also collects rainwater, thus altering the total solid content. The characterization of the liquid manure at the first series of measurements (June) revealed a stable *inoculum.* In the second series of measurements (October), the characterization of the liquid manure revealed a rich and stable *inoculum*.

The roadside grass was harvested by agricultural machines specifically tailored to this study. In this study, the roadside grass was mowed along a road with heavy traffic. This substrate was collected from roadsides in Brittany (France). The roadside grass characterizations were carried out. The composition of the roadside grass differed according to the season. This difference in the compositions should affect the production of methane and the hydrodynamic properties of these media.

The cattle manure was obtained from the farm of the UniLaSalle Polytechnic Institute (Beauvais, France). The cattle manure characterizations were carried out. The variation in the TS content of these two cattle manures was due to the seasonality and the climatic conditions. The manure used for the second series appeared to be less rich in fibre and contained more moisture than the one used in the first series of measurements. This difference appears to be due to the manure storage and the climatic conditions.

2.3 Biochemical Methane Potential (BMP)

Use of an AMPTS I device (Automatic Methane Potential Test System, Bioprocess Control, Sweden) allowed automatic measurement of the methane production and determination of the cumulative volume and the flow of methane into parallel 500 mL bottles. In terms of VS, the implemented *Inoculum*/Substrate ratio was 3. The substrates were either dried and crushed or fresh material cut into centimetre-sized fragments with scissors. The trial with dried crushed material allowed the ultimate methane potential to be reached by enhancement of the accessibility of the material as a result of the drying and crushing stages.

The trial performed with fresh material provided the actual methane potential. Prior to its use in a test of the methane potential, the liquid manure that served as the *inoculum* was filtered with a mesh of 2 x 2 mm to decrease the VS load. The resulting methane potential hence reflects that of the digested substrate and not that of the liquid manure particles. In this AMPTS system, the biogas produced passed through a scrubber filled with NaOH (3 $mol.L^{-1}$) that captured the carbon dioxide. The methane was made to flow to the measurement cell and was recorded as a function of time. The volume of biogas was corrected according to the temperature and the pressure. Processing of the data allowed the $Nm^3_{CH4}.t_{MO}^{-1}$ and the $Nm^3_{CH4}.t_{MF}^{-1}$ to be obtained as a function of the experimental requirements. These tests were carried out in triplicate for each of these conditions. If the RSD (Relative Standard Deviation) was less than 15%, the triplicates were validated; if the RDS was greater than 15%, the data were duplicated until the RDS was less than 15% according to Holliger et al. (2016).

2.4 Anaerobic co-digestion in dry batch at a scale of 60 L

Six dry process reactors of 60 L (Fig. 1a) were used with three different compositions and two modes of filling during the two series of measurements based on fresh material for each substrate. No pre-treatment of the material was carried out.

Reactor 1 employed a composition of 50% roadside grass and 50% cattle manure (vol/vol). Reactor 2 was composed of 40% roadside grass and 60% cattle manure (vol/vol). The composition of reactor 3 was 25% roadside grass and 75% cattle manure (vol/vol).

For the first series of measurements, the substrates were deposited as two layers without the application of any pressure. By contrast, for the second series of measurements, both substrates were manually mixed to obtain the most homogeneous medium possible before introduction in the reactor (Fig. 1b). Twenty kilograms of the *inoculum* was added to each reactor. Prior to use, the liquid manure was filtered with a mesh of 2 x 2 mm to decrease the VS load. The unit was sealed to create anaerobic conditions and thermostatically-controlled at 37 °C. The liquid phase was recirculated periodically at a rate of 15 $L.h^{-1}$ with a frequency of

recirculation of 15 min every hour. The reactors were joined to an AMPTS I device (Bioprocess Control, Sweden) to record the production of biogas. The composition of the biogas was monitored daily by a gas analyser (Multitec 540, Sewerin, France). Analysis of the pH, the total quantity of acids, and the buffer capacity was carried out daily on each recirculation loop of the liquid phase of the reactors (Fig. 1a). The results of the cumulative methane productions were expressed in $Nm^3_{CH4}.t_{FM}^{-1}$ and in $Nm^3_{CH4}.t_{VS}^{-1}$. These experiments were not performed in duplicate, but the inoculum and substrates were the same for the three reactors in the same series. The usual experimental deviation of the AMPTS I gas counter was set at 2%, then affecting the biogas production of each reactor. Moreover, these substrates and the inoculum were used in the triplicate BMP determinations (see section 2.3).

3 Results and Discussion

3.1 Biochemical Methane Potentials

The methane potentials of cattle manure and roadside grass used in this study were established with fresh as well as dried and crushed materials by the liquid process during the two series of measurements.

3.1.1. The first series of measurements

The methane potentials obtained were 222 $Nm^3_{CH4}.t_{VS}^{-1}$ for the dried and crushed manure, and 194 $Nm^3_{CH4}.t_{VS}^{-1}$ for the fresh manure. The methane potentials obtained were 193 $Nm^3_{CH4}.t_{VS}^{-1}$ for the dried and crushed roadside grass, and 203 $Nm^3_{CH4}.t_{VS}^{-1}$ for the fresh roadside grass (Table 1). The methane potentials obtained for the fresh and the dried materials can be considered to be similar for each of the substrates, given the uncertainty of the measurements. The loss of volatile matter during drying, heterogeneity of the substrate, as well as the sampling are factors that determine the uncertainty associated with these methane potentials. The AMPTS processes were all validated with a coefficient of variation of less than 15% in light of the heterogeneous nature of the substrates (Holliger et al., 2016). The

mean value in the literature for cow and beef manure with a total solid content of approximately 30% is 317 $Nm^3_{CH4}.t_{VS}^{-1}$ (Degueurce et al., 2016b). The value obtained for the cattle manure is lower, but this data depends on the *inoculum* source (Degueurce et al., 2016a) and the nature of the substrate.

The methane potential depends on several criteria, particularly the type of livestock, the level of mulching, the animal feed, and the source of the *inoculum* chosen for the tests, as well as the methodology employed by the laboratory. These variables can result in a difference in the order of 20% in interlaboratory testing on identical substrates (Cresson et al., 2015). Meyers et al. (2014) reported a range of approximately 220 to 390 $m^3_{CH4}.t_{VS}^{-1}$ for roadside grass. Experimental values of roadside grass methane potential are mostly similar, and any differences are due to the properties of the TS and the VS contents.

The difference in methane potentials between cattle manure and roadside grass was 4% based on fresh material only. This modality of comparison is preferred, as in anaerobic digestion by the dry process the waste products are usually digested in their natural state without pre-treatment. For this first series of measurements, these tests showed that the two substrates had comparable methane potentials and that the roadside grass contributed as much to the anaerobic digestion as did the cattle manure when co-digestion was employed in the trials of anaerobic digestion by the dry process for this series of measurements.

3.1.2. The second series of measurements

Cattle manure: the methane potential obtained for cow manure based on fresh material (216 $Nm^3_{CH4}.t_{VS}^{-1}$) was 20% higher than that obtained with dry material (173 $Nm^3_{CH4}.t_{VS}^{-1}$) (Table 1). This phenomenon can be explained by volatilisation of the material during the drying stage. This volatilization appears to be enhanced with a substrate with a TS content of less than 20%. Indeed, the contact surface is increased in the presence of more water in the substrate, thereby enhancing volatilization during drying.

Roadside grass: the methane potential obtained with crushed dried material (216 $Nm^3_{CH4}.t_{VS}^{-1}$) was 22% higher than with fresh material (168 $Nm^3_{CH4}.t_{VS}^{-1}$) (Table 1). This substrate is very ligneous initially and very dry, and accessibility of the material was increased by crushing it. In this second series of measurements, the properties of the substrate were very different and yielded results that differed from those of the first series of measurements. The AMPTS processes were always validated with a coefficient of variation of less than 15% in light of the heterogeneous nature of the substrates (Holliger et al., 2016).

3.1.3. Comparison of the two series of measurements.

Cattle manure: The values obtained for the two series were similar for the cattle manures from fresh matter. The values obtained from the crushed dried substrate were slightly different, with those of the first series being higher (Table 1). This is due to the initial composition of these two cattle manures in terms of fibres, with the cattle manure of the first series of measurements having a higher quantity of cellulose and hemicellulose than the cattle manure of the second series (see section 2.3.3). The seasonality, therefore, impacted the methane potential. Amon et al. (2007) have shown that the methane potential of straw-based cow cattle manure (208 to 268 $Nm^3_{CH4}.t_{VS}^{-1}$) is influenced by the animal feed and its capacity to digest it and is hence influenced directly by the seasonality.

Roadside grass: The difference in the methane potential between that generated by fresh material versus crushed dried material was 22%. The methane potential obtained with fresh material in the first series was 17% higher than the second series. In the second series, the roadside grass was considerably drier, with 52% TS, and it had a considerably higher level of lignin. Belanger et al. (2012) have shown that the production of methane from silaging of switchgrass increases with a lower fibre content. The values obtained, and the ensuing conclusions, are hence consistent. The methane potentials obtained with dry crushed material were, therefore, similar for the two series (Table 1).

These trials show that the properties of the substrates, and consequently the ensuing methane potential, varied with the season. Depending on the respective properties of these substrates, a difference of 20% in the methane potentials was obtained. To design an anaerobic digestion unit, the BMP needs to be determined for the substrate in question, rather than taking a value from the literature, as the BMP depends on the nature and the seasonality of the substrate.

3.2 Anaerobic co-digestion of roadside grass and cattle manure by the dry process (60 L)

Roadside grass and cattle manure substrates were subject to anaerobic digestion by the dry process in reactors of 60 L. The modalities are stated in paragraph 2.4.

3.2.1 The first series of measurements

3.2.1.1. The production of methane and physicochemical analyses

In this series of measurements, the three co-digestions were carried out with layering of the substrates. The occurrence of inhibition by the accumulation of volatile fatty acids is common with digestion by the dry process. To manage these inhibitions, the proportion of less fermentable materials can be increased to buffer the medium (Chynoweth et al., 1991; Foster-Carneiro et al., 2004; 2007; Dotal et al., 2013a; 2013b). As roadside grass is readily fermentable, it was decided to generate three reactors with different proportions of roadside grass and cow cattle manure.

The following methane production levels were obtained: 44.4 $Nm^3_{CH4}.t_{FM}^{-1}$ for the 50% - 50% blend (reactor 1); 39.8 $Nm^3_{CH4}.t_{FM}^{-1}$ for the 40% - 60% blend (reactor 2); and 34.8 $Nm^3_{CH4}.t_{FM}^{-1}$ for the 25% - 65% blend (reactor 3) (Table 2; Fig. 2a). The differences in the cumulative productions between the three reactors were due to the different proportions of roadside grass. The difference in production between reactor 1 and reactor 3 was 21%. Reactor 2 exhibited a difference in production of 10% with reactor 1 and 12% with reactor 3. The usual production in a 60 L batch reactor under identical conditions with just cattle manure is of the order of 35 $Nm^3_{CH4}.t_{FM}^{-1}$ (André et al., 2015). This value is fully compatible

with the value obtained for reactor 3, which had a composition of 75% cattle manure and 25% roadside grass (Table 2).

For several days, the reactors exhibited signs of acidification that to varying degrees was rapidly reversible and recoverable without intervention. Reactor 1 had the most volatile fatty acids, with a plateau for approximately 10 days at 8 $g.L^{-1}$, followed by a progressive consumption of these fatty acids (Fig. 3a). The kinetics of the production of biogas by reactor 1 was impacted slightly by the absence of production for 2.5 days (Fig. 2a). The accumulation of fatty acids for reactors 2 and 3 was identical despite their different compositions, exhibiting a peak at 7.5 $g.L^{-1}$, a stabilization for about ten days around 5 $g.L^{-1}$, and then nearly complete consumption of these volatile fatty acids (Fig. 3a). The variations in pH and the buffer capacity varied according to the overall volatile fatty acid content. Reactor 1 was at the limit of irreversible acidification with a pH that struggled to increase again and become stable (Fig. 3b-c). These reversible inhibitions could be managed with an *inoculum* with a higher buffer capacity. The buffer capacity and the pH of the *inocula* after 32 days of degradation increased (Fig. 3c).

Ahn et al. (2010) performed a co-digestion by the dry process with cattle manure and switchgrass in a reactor of 1 L, resulting in nearly complete inhibition with an accumulation of volatile fatty acids between 12 and 14 $g.L^{-1}$ and a production of 0.028 $L_{CH4}.g_{VS}^{-1}$. Macia-Corral et al. (2008) employed co-digestions with cattle manure and an organic fraction of household waste that resulted in inhibitions representative of varying degrees of reduced rates of methane production.

For this first series of measurements, it should be noted that percolation of the liquid phase in the bulk material took place properly, as did the recirculation of the liquid phase during the process (Table 2). It was possible to differentiate two digestates in these reactors with layers. Table 3 presents the variations in the contents of TS and VS of the different solid phases involved in the three reactors placed in degradation mode. The TS contents of the digestates

obtained were substantially lower than the substrates at the beginning; this being the consequence of the degradation of these substrates. The change in the digestates of the roadside grass was identical, except for reactor 3 for which the roadside grass was degraded slightly less. On the other hand, the cattle manure in reactor 3 was degraded to a greater extent than in the other two reactors. With the two other conditions, the cattle manure was degraded to the same extent.

For the fibre content of the different solid phases, the initial substrate served as a reference that was analysed and the one prior to digestion without contact with the *inoculum*. To obtain an unequivocal comparison of the levels, the substrate should have been wetted in the same manner as the *inoculum*. Nonetheless, these analyses allowed us to observe the differences between the initial substrates and the rates of degradation between the reactors and to also observe a decrease in the levels of these components relative to the initial substrate (Fig. 4a-4b). In this series of measurements, more degradation of the fibres of roadside grass was observed for reactor 2, which had a composition of 40% roadside grass and 60% cattle manure. The level of fibres in this digestate of roadside grass in reactor 2 was slightly lower than the other digestates (Fig. 4a-4b). In this series of measurements, the fibre composition of the digestates shows that the anaerobic digestion allowed more extensive degradation of the cattle manure in reactor 3, which was also the reactor that had the largest proportion of manure. Conversely, the cattle manure underwent the least degradation in reactor 1 (Fig. 4b).

3.2.1.2 Liquid and mass balances of the reactors

The liquid and mass balances of the reactors placed in degradation mode at a scale of 60 L allow the events involved in anaerobic digestion by the dry process to be understood (André et al., 2015). The quantity of *inoculum* was set at 20 kg, representing an immersion of 22% of the solid bulk and involving an I/S ratio of 0.06 in these reactors and under these conditions. The liquid and mass balances of these reactors after anaerobic digestion are presented in Table 4. Reactor 3 had the greatest decline in total weight. This is directly related to the loss

of weight due mainly to degradation of the cattle manure. The decrease in the height of the bulk material in this series of measurements was 54% for reactor 3 and 60% for reactors 1 and 2 (Table 4). Degradation of the cattle manure was more pronounced (57%) in reactor 3, which was composed mainly of cattle manure (Tables 3-4).

Degradation of the roadside grass did not follow this same pattern. The increase in the weight of the roadside grass was due to the liquid phase that recirculated periodically onto it as it is placed on top of the cattle manure. In light of this, it accumulates organic matter of the cattle manure and of the *inoculum*. Its weight increased from 21 to 37% depending on the reactor. The percentage of water produced increased with the quantity of cattle manure present in the reactor and ranged from 4 to 6%. The cattle manure produced water while the roadside grass absorbed water. These features were characteristic of the respective TS contents of each of the co-substrates of this co-digestion. The loss of total weight in each of the reactors was more pronounced when the reactor was composed mainly of cattle manure, which contains more organic material and has a smaller ligneous content that needs to be degraded than roadside grass.

Recirculation of the liquid phase allows nutrients, the temperature, water, and the microorganisms to be distributed within the solid bulk material. The activity of methanogens, therefore, increases in the presence of water (Le Hyaric et al., 2012; André et al., 2016). This recirculation is, however, limited if it is coupled with degradation. Indeed, studies of liquid transfers (André et al., 2015; Shewani et al., 2015) within solid bulk materials during anaerobic digestion have revealed stagnation of the degradation and changes in the bulk material after approximately 15 days. These studies have allowed static and dynamic states of the system to be defined (André et al., 2015). These phenomena were encountered in trials carried out in co-digestion. Tracing experiments would be needed to quantify the liquid transports involved. Nonetheless, the final extent of the subsidence was nearly identical, thus showing that the medium reached a static state (Table 4).

For reactor 3 (25% roadside grass and 75% cattle manure), the contents of TS and VS of the digestates (Table 3) reveal a more degraded digestate for the cattle manure of 27%. Moreover, the roadside grass was not degraded as well as for the other two conditions, with a difference of 27%. The cattle manure was degraded in the same manner as for the other reactors (Table 3).

The quantity of VS provided by the cattle manure was greater than for the roadside grass. Yet the roadside grass was more fermentable. Consequently, with more grass, the methane productions were greater. Reactor 3 had the greatest amount of digested organic matter. Nonetheless, its methane potential was the lowest, as it was essentially only generated by the cattle manure.

The amount of organic matter amounted to 69% for reactor 1, 71% for reactor 2, and 75% for reactor 3 (Table 4). The anaerobic digestion employed in this case appears to be satisfactory, although there was a residual degradation potential of the order of 30% for this series of measurements. Optimization of the recirculation of the liquid phase, the immersion, or the arrangement of the substrates could increase this production of methane.

3.2.2. The second series

3.2.2.1. The production of methane and physicochemical analyses

In this series of measurements, the substrates were not arranged in layers (as was the case for the first series) but mixed in order to optimize contact between the cattle manure and the roadside grass. The filling of the reactors was otherwise identical to the first series of measurements. The I/S ratios induced by the mode of filling by volume and the properties of the input were 0.22 to 0.26. The methanogenic productions obtained after 32 days for the reactors were 39.9 $Nm^3_{CH4}.T_{FM}^{-1}$ for reactor 1 (50% cattle manure- 50% roadside grass), 39.5 $Nm^3_{CH4}.T_{FM}^{-1}$ for reactor 2 (60% cattle manure– 40% roadside grass), and 35.4 $Nm^3_{CH4}.T_{FM}^{-1}$ for reactor 3 (75% cattle manure– 25% roadside grass) (Table 2; Fig. 2b).

In this series of measurements, the composition of reactor 1 did not allow proper recirculation of the liquid phase to be established. Sampling of the liquid phase was hence not possible with the exception of a few data points. For reactor 1, these parameters appear to be hard to interpret in light of the paucity of values obtained (data not shown). Observation of the production flow of methane (data not shown) indicates identical flows between the three reactors, while for reactor 1 no recirculation could be established prior to 25 days of anaerobic digestion. This shows that recirculation of the liquid phase is not necessary with this type of co-digestion as a mixture, as the liquid cannot circulate within the solid phase and hence provides few or no benefits.

The high buffer capacity of the initial *inoculum* of around 10 $g.L^{-1}$ allowed it to handle the quantity of volatile fatty acids present in the medium. This characteristic, associated with the very ligneous and dry nature of the roadside grass of this series, allowed the respective anaerobic digestions to not undergo an acidification phase. For reactors 2 and 3, the quantities of volatile fatty acids decreased considerably over the course of the anaerobic digestion, going from approximately 6 $g.L^{-1}$ to less than 2 $g.L^{-1}$ (Fig. 3d). Conversely, the buffer capacity of these two reactors increased by 10 $g.L^{-1}$ to 12 $g.L^{-1}$ (Fig. 3e). These parameters are representative of stable anaerobic digestions (Fig. 3f).

A decrease in the content of TS and VS was also noted for the second series of measurements (Table 3). A higher content of TS was noted for the digestate of reactor 1. The content of TS decreased from reactor 1 to reactor 3; this phenomenon is normal as the quantity of roadside grass was highest in reactor 1 and lowest in reactor 3. Conversely, the content of VS increased for reactor 1 to reactor 3. This is likewise normal, as reactor 3 contained the largest quantity of cattle manure (Table 3).

The contents of lignin, hemicellulose, and cellulose were similar in the three reactors. Comparison of the initial substrates presents the same issue as previously. Be that as it may, as it was not possible to separate the digestates of grass and manure in this second series, it is

difficult to conclude which substrate was degraded the most (Fig. 4c). Filling as a mixture did not allow good percolation of the liquid phase within the bulk material to be achieved. The draining of liquid was very slow due to the low permeability of the roadside grass. No acidification phase was observed for this series of measurements. The blend mode allowed acidification of the medium to be avoided. Nonetheless, percolation, which is an essential point of anaerobic digestion as a dry phase, could not take place properly and the liquid was not well distributed. Moreover, the very dry roadside grass absorbed the liquid phase and this impacts directly on industrial management of this type of co-digestion.

3.2.2.2. *Liquid and mass balances of the reactors*

The liquid and mass balances of the reactors allow the phenomena involved in methanisation by dry batch digestion to be understood (André et al., 2015; Shewani et al., 2015). The characteristics of filling and emptying of the reactors are summarized in Table 5. The reactors were filled in the same manner for series 1 only. Nonetheless, as shown in Figure 5, the immersion did not take place in the same manner. Indeed, areas of retention created local areas of immersion, and these were not at the bottom of the reactor as in the first series of measurements. Moreover, it is difficult to pinpoint these areas and their representations within the reactors involved. The weights of the liquid and solid phases allow it to be established whether water was produced or absorbed. This point is essential for conducting such an undertaking by dry digestion. It can be seen that reactor 1, composed of 50% roadside grass, needed a greater liquid phase input. Absorption of water was noted and calculated to be 13%. For the other two reactors with a higher proportion of cattle manure (60% and 75%, respectively), water releases of 4 and 8%, respectively, were observed (Table 5). Calculation of the loss of weight of the solid phase allowed the degradation taking place over the course of the anaerobic digestion to be translated. A small loss of weight was observed for reactor 1, composed mainly of roadside grass with a ligneous nature. Reactor 3 exhibited the greatest

loss of weight, with a loss estimated at 44.8%. This considerable loss of weight was due particularly to degradation of the cattle manure.

One of the hydrodynamic characteristics of fluid transfers and settling over the course of the process that allows its progression to be observed is the height of the bulk material before and after anaerobic digestion. Despite the differences in weight loss, the change in height was identical for the three reactors (Table 5). This phenomenon can be explained by the initial composition of the reactors and their progression. The smallest amount of digestate obtained corresponded with reactor 3, due to the quantity of cattle manure used in this reactor (Table 5). As stated previously, the contents of TS and VS of the digestates obtained changed inversely from reactor 1 to reactor 3 and for the same reasons outlined in series 1 (Table 3). The quantity of VS consumed amounted to 43% for reactor 1, 50% for reactor 2, and 53% for reactor 3. These values are a clear reflection that the "mixed" mode is not ideal for carrying out anaerobic digestion with this type of substrate. Fifty percent of the organic matter remained in the digestate and did not allow biogas to be produced. The methane productions could be optimized in this series of measurements by changing the way that the reactors were loaded (Table 5).

3.2.3. Comparison of the two series of measurements

The conclusions that can be drawn from these different operating conditions employed for co-digestion are listed below and they are also shown in Figure 5. These two series of measurements, involving substrates and *inocula* with different seasonalities and two modes of conducting the process, allowed the consequences of each of these parameters on dry batch anaerobic digestion to be discerned. The roadside grass was a highly fermentable substrate and could induce acidification of the medium following the accumulation of volatile fatty acids in the medium. The roadside grass exhibited low permeability and a sponge-like nature. It has a low structuring capacity and does not allow good structuring of the medium to be obtained, thereby leading to poor percolation of the liquid phase. This substrate nonetheless

exhibited a good methanogenic potential, although it did require adjustment in order to be methanised in co-digestion. The seasonality impacted the composition of this substrate and hence its methanogenic capacity. The cattle manure was a less fermentable substrate, but it provided a buffer capacity to the medium as well as the methanogenic populations. This substrate also varied with the seasonality and its storage. The presence of straw in its constitution provided it with a structuring capacity. This structuration lead to a good level of permeability and percolation of the liquid phase. The *inocula* used corresponded to manure derived from the same farm, albeit obtained at different times. It was noted that this manure changed over the course of the year. It also emerged from this study that the buffer capacity is a key point for conducting such processes. It is, therefore, recommended at 10 $g.L^{-1}$ in order to be able to handle acidifications. Degueurce et al. (2016a) have shown that the production of methane by the dry process with straw-based manure is influenced by the origin of the *inoculum* and that it can be multiplied by two by using an *inoculum* or agricultural origin (~ 114 $Nm^3_{CH4}.t_{VS}^{-1}$) compared to a phosphate buffer (~ 60 $Nm^3_{CH4}.t_{VS}^{-1}$). The *inoculum* is a key parameter for optimization of the production of methane.

Conducting the process of methanisation by the dry process: the so-called "mixed" mode led to problems in terms of management of the recirculation and the permeability of the medium. The absence of acidification with this mode of loading can be attributed to this fact alone. Indeed, the *inoculum* had a buffer capacity that was considerably higher than that for the other series of measurements. This mode is not representative of what takes place at industrial sites. The "layered" mode exhibited a good level of percolation of the liquid phase and good repartition thereof in the reactor. The methane potentials obtained in 60 L with this modality were low and due to the fact that the *inoculum* had a poor buffer capacity and to the presence of an acidification phase. This acidification was the consequence of the very rich nature of the roadside grass used. As a reminder, in the second series, this roadside grass was very ligneous and less fermentable. The two modes of filling of the reactors allowed it to be

shown that loading as layers was the most suitable method for co-digestion of these substrates. Moreover, it was noted that the degradation of organic material was considerably greater with the layered mode than with the mixed mode. As a reminder, approximately 72% of the organic material was degraded in the layered mode while only 50% or the organic matter was degraded in the mixed mode. Although other parameters need to be taken into account, the difference is sufficiently large to be an issue. Considerably lower levels (9 – 20%) of degradation of organic material for two other types of thermophilic co-digestions in the presence of cow or poultry manure have been reported (Ahn et al., 2010). The levels of degradation obtained in these two series of measurements are, therefore, good, but there is room for improvement.

Studies have also highlighted the advantage of this layered mode by showing that the alternation of layers of fermentable and less fermentable layers leads to acceleration of the start-up of these reactors (Foster-Carneiro et al., 2004). The *inoculum* to substrate ratios were in favour of the mixed loading mode with a ratio equal to approximately 0.22, while it was 0.06 for the layered condition. This variation of the *inoculum* to substrate ratio is due to the loading mode as a function of the usable volume, and not the organic matter. This approach is representative of that used at the industrial level. Optimization of this co-digestion can be carried out, as 30% of the organic material (layered mode) was not degraded. This optimization can be carried out by adjustment of the frequency of recirculation, the quantity of liquid recirculated, and the level of immersion of the solid medium. Several studies have shown that increasing the frequency of recirculation and the quantity of liquid recirculated increases the production of methane (Klink and Ham, 1982; François et al., 2006; El-Mashad et al., 2006; Filipkowska, 2008; Kush et al., 2009; Shahriari at al., 2012; André et al., 2018). A doubling of the recirculation of the liquid manure in a 60 L reactor resulted in a 22% increase in the production of methane (André et al., 2015). With a 60 L batch reactor in dry

mode using cattle manure, two immersions of the solid bulk of 33% and 66% were tested, revealing a 13% increase in the production of methane (André et al., 2016).

In terms of the recipe of this co-digestion, there appears to be a clear limit for the proportion of roadside grass that can be used due to acidification that depends on the seasonality, the mode of operation, and the *inoculum*. Anaerobic digestion by the dry process is a key way to valorise agricultural resources. Optimization of this process can be achieved by management of the liquid phase during the process and by the way that the reactors are loaded. Trials of different co-digestions should be pursued to optimize and better manage these processes.

4 Conclusion

Co-digestion by the dry process at a pilot scale (60 L) of RSG and CM is achievable, but the input of RSG needs to be controlled. Based on two series of measurements, the condition exhibiting the best compromise between inhibition and production was the layered mode with 40% v/v RSG and 60% v/v CM. The production of methane from this co-digestion can be optimized by the filling mode, the *inoculum*, the level of immersion, and the rate of recirculation. The seasonality of the input alters their composition and hence their hydrodynamic properties, the inhibitions, and the methane production.

E-supplementary data for this work can be found in e-version of this paper online.

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Figure and table captions

Table 1. The BMP of fresh and dried crushed matter substrates, expressed in $Nm^3_{CH4}.t_{VS}^{-1}$.

Table 2. Methane production during the two series of dry batch anaerobic digestion in 60 L.

Table 3. The TS and VS contents of the substrates and dry digestates at the start and at the end of the experimentation.

Table 4. The liquid and mass balance of the dry batch reactors (60 L) for the first series.

Table 5. The liquid and mass balances of the dry batch reactors (60 L) for the second series.

Figure 1. a. The dry batch anaerobic reactor (60 L), b. Experimental synopsis for the 60 L reactors.

Figure 2. The cumulative methane production of the dry batch anaerobic reactors in (a) the first series and (b) the second series. Black: reactor 1, 50% CM – 50% RSG; dark grey: reactor 2, 60% CM – 40% RSG; light grey: reactor 3, 75% CM – 25% RSG.

Figure 3. Monitoring of the volatile fatty acids, buffer capacity, and pH during anaerobic digestion in a dry batch pilot at 60 L: a,b,c: for the first series; d, e, f: for the second series; Black squares: 50% CM – 50% RSG; white diamonds with a continuous outline: 60% CM – 40% RSG; grey triangles: 75% CM – 25% RSG.

Figure 4. The fibre contents of RSG, CM, and digestates. a,b: for the first series; c: for the second series; black: hemicellulose, dark grey: cellulose; light grey: lignin.

Figure 5. Summary of the liquid transfer during the dry anaerobic digestion in the first and the second series.

Table 1. The BMP of fresh and dried crushed matter substrates, expressed in $Nm^3_{CH4}.t_{VS}^{-1}$.

Substrates	First series		Second series	
	Fresh matter	Dried crushed matter	Fresh matter	Dried crushed matter
Cattle Manure	194 ± 22	222 ± 29	216 ± 4	173 ± 13
Roadside grass	203 ± 8	193 ± 22	168 ± 3	216 ± 22

Table 2. Methane production during the two series of dry batch anaerobic digestion in 60 L.

	Reactor 1 50% CM – 50% RSG	Reactor 2 60% CM 40% RSG	Reactor 3 75% CM 25% RSG
First series			
Filling condition		Layers	
Methane production ($Nm^3_{CH4}.t_{FM}^{-1}$)	44.4 ± 0.9	39.8 ± 0.8	34.8 ± 0.7
Methane production ($Nm^3_{CH4}.t_{VS}^{-1}$)	200 ± 4.0	186 ± 3.7	170 ± 3.4
Second series			
Filling condition		Blend	
Methane production ($Nm^3_{CH4}.t_{FM}^{-1}$)	39.9 ± 0.8	39.5 ± 0.8	35.4 ± 0.7
Methane production ($Nm^3_{CH4}.t_{VS}^{-1}$)	217 ± 4.3	232 ± 4.6	229 ± 4.6

Table 3. The TS and VS contents of the substrates and dry digestates at the start and at the end of the experimentation.

			Reactor 1 50% CM 50% RSG		Reactor 2 60% CM 40% RSG		Reactor 75% CM 25% RS
s	Roadside grass	Cattle Manure	Digestate RSG R1	Digestate CM R1	Digestate RSG R2	Digestate CM R2	Digestate RSG R3
	34.8 ± 0.2	22.1 ± 0.1	13.4 ± 0.1	12.2 ± 0.2	13.2 ± 0.1	11.9 ± 0.1	18.3 ± 0.1

	88.9 ± 0.1	87.8 ± 0.2	71.3 ± 0.1	82.1 ± 0.1	73.5 ± 0.1	81.3 ± 0.1	76.3 ± 0.1	8

ries	Roadside grass	Cattle Manure	Digestate blend R1	Digestate blend R2	Digestat blend R3
	52.2 ± 0.1	15.6 ± 0.1	14.8 ± 0.1	13.2 ± 0.2	12.5 ± 0.
	88.0 ± 0.1	87.9 ± 0.2	73.1 ± 0.1	74.0 ± 0.1	77.5 ± 0.

Table 4. The liquid and mass balance of the dry batch reactors (60 L) for the first series.

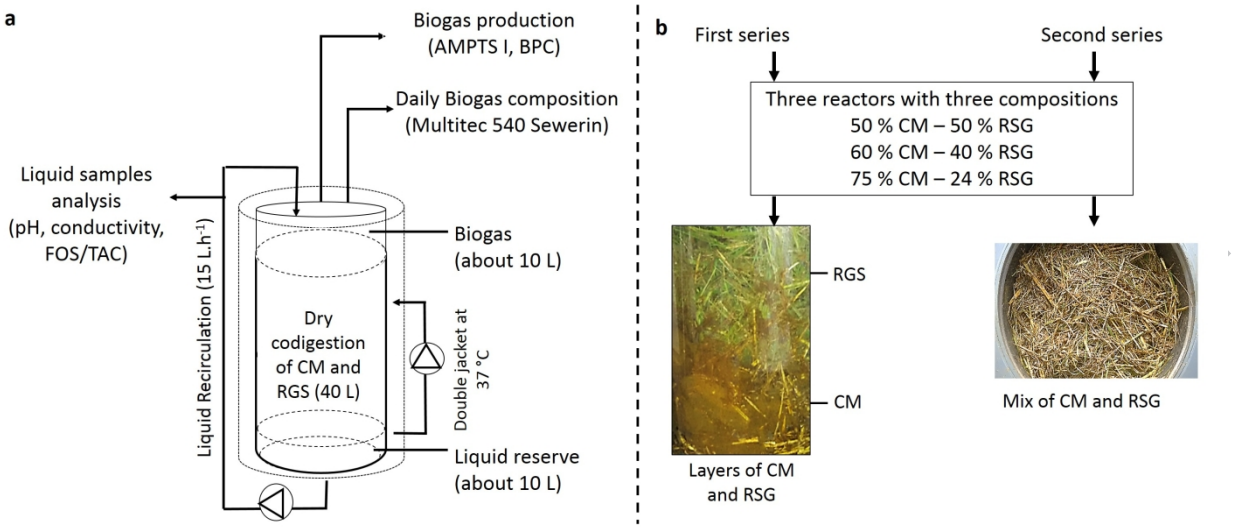
First series		Reactor 1 50% CM – 50% RSG	Reactor 2 60% CM 40% RSG	Reactor 3 75% CM 25% RSG
Initial *inoculum* weight	*kg*	20.00	20.00	20.00
Initial CM weight	*kg*	12.52	14.33	17.91
Initial RSG weight	*kg*	4.06	3.10	1.94
Inoculum/Substrate ratio	$g_{VS}.g_{VS}^{-1}$	0.06	0.06	0.06
CM digestate weight	*kg*	6.41	6.35	7.64
TS	*%*	12.21 ± 0.33	11.89 ± 0.19	8.84 ± 0.98
VS	$\%_{TS}$	82.14 ± 0.51	81.35 ± 1.32	80.67 ± 1.14
RSG digestate weight	*kg*	5.20	4.50	3.10
TS	*%*	13.43 ± 0.55	13.21 ± 0.63	18.33 ± 1.74
VS	$\%_{TS}$	71.32 ± 1.27	73.47 ± 3.32	76.33 ± 1.55
Final *Inoculum* weight	*kg*	20.84	20.89	21.20
CM weight loss	*%*	48.80	55.68	57.34
Increase in RSG weight	*%*	21.92	31.12	37.42
Increase in water produced	*%*	4.22	4.47	6.00
Total weight loss of solid phase	*%*	29.98	37.47	45.89
Decrease in media height	*%*	60.00	60.00	54.29
Initial VS weight	*kg*	3.68	3.74	4.07
Final VS weight	*kg*	1.14	1.05	0.98
Methane production	$Nm^3_{CH4}.t_{VS}^{-1}$	200 ± 4.0	186 ± 3.7	170 ± 3.4

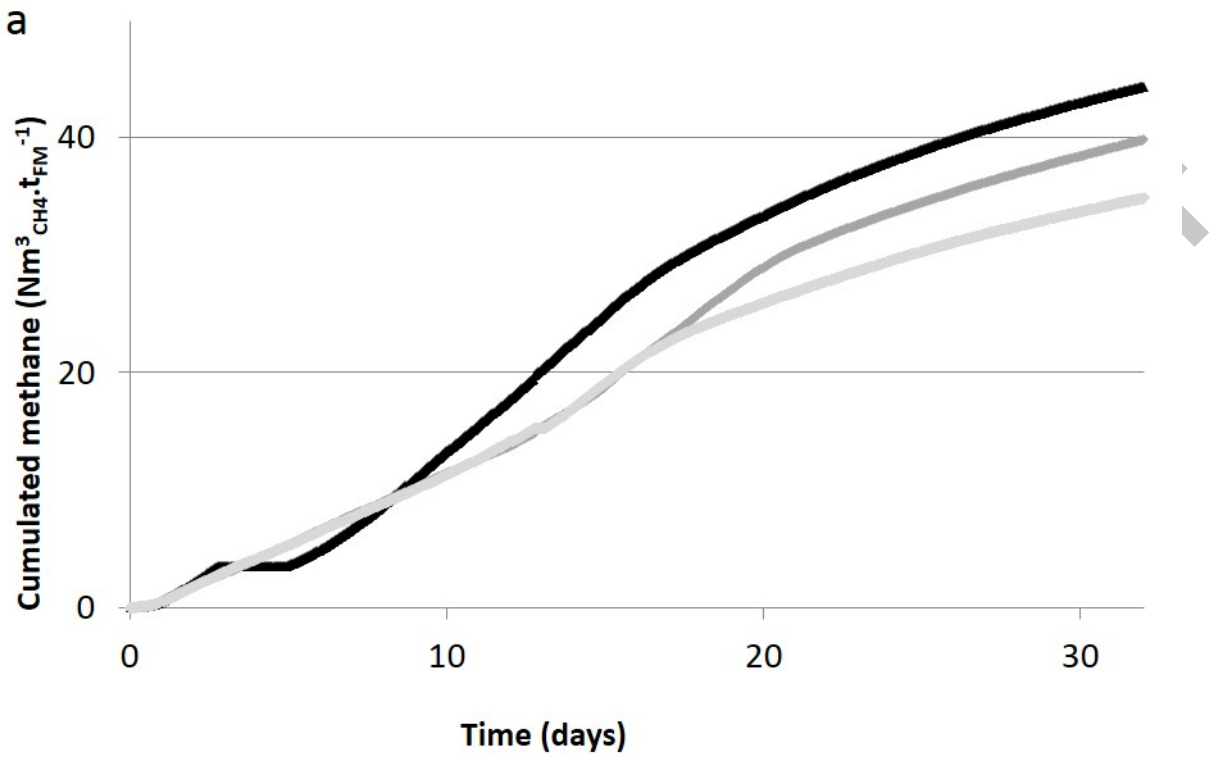
Table 5. The liquid and mass balances of the dry batch reactors (60 L) for the second series.

Second series		Reactor 1 50% CM – 50% RSG	Reactor 2 60% CM 40% RSG	Reactor 3 75% CM 25% RSG
Initial *inoculum* weight	*kg*	20.00	20.00	20.00
Initial CM weight	*kg*	11.89	14.27	17.83
Initial RSG weight	*kg*	2.05	1.64	1.02
Ratio *inoculum*/substrate	$g_{VS}.g_{VS}^{-1}$	0.25	0.24	0.22
Digestate weight	*kg*	13.50	13.86	10.40
TS	*%*	14.89 ± 3.21	13.17 ± 1.33	12.49 ± 0.05
VS	$\%_{TS}$	73.13 ± 3.17	74.01 ± 0.07	77.49 ± 0.63
Final *Inoculum* weight	*kg*	17.40	19.20	21.90
Increase in water produced	*%*	-13.00	4.00	8.70
Total weight loss of solid phase	*%*	3.16	12.88	44.85
Decrease in media height	*%*	45.71	45.71	42.86
Initial volatile solid weight	*kg*	2.57	2.71	2.91
Final volatile solid weight	*kg*	1.46	1.35	1.36
Methane production	$Nm^3_{CH4}.t_{VS}^{-1}$	217 ± 4.3	232 ± 4.6	229 ± 4.6

Highlights

- Dry co-digestion of roadside grass with cattle manure was performed in batch.
- Filling as layers or as a blend impacted the hydrodynamics and CH_4 production.
- A seasonal impact of methane production was observed.

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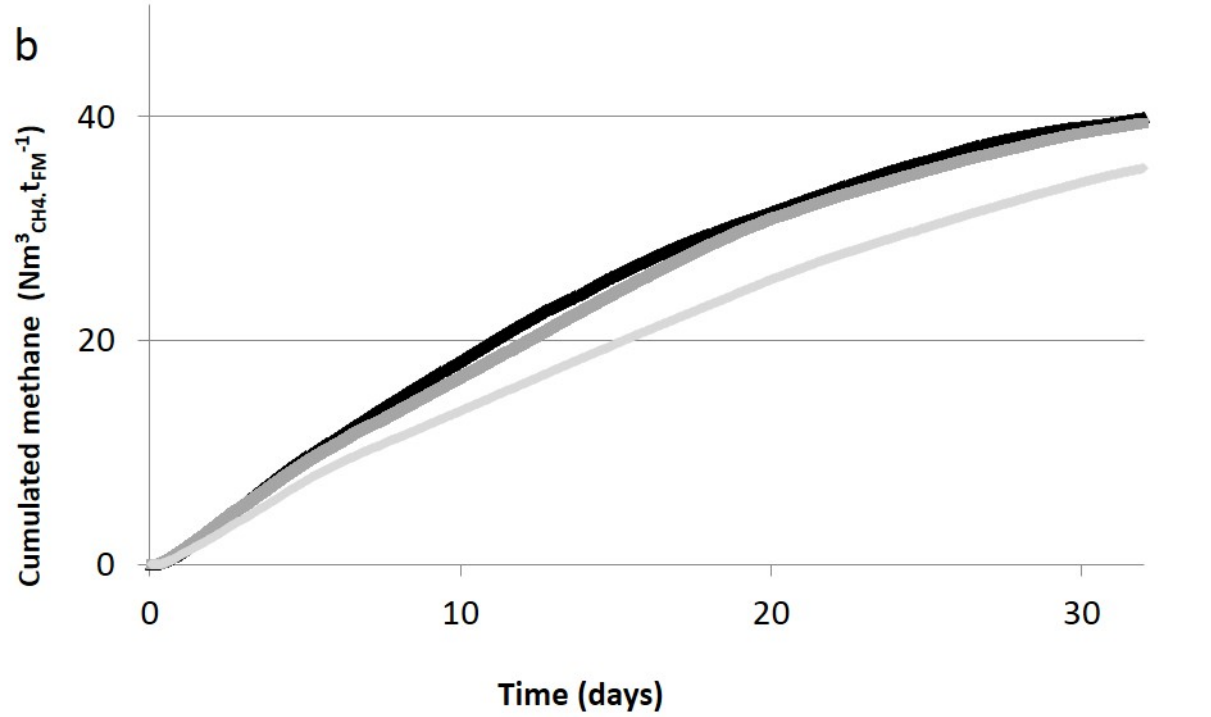
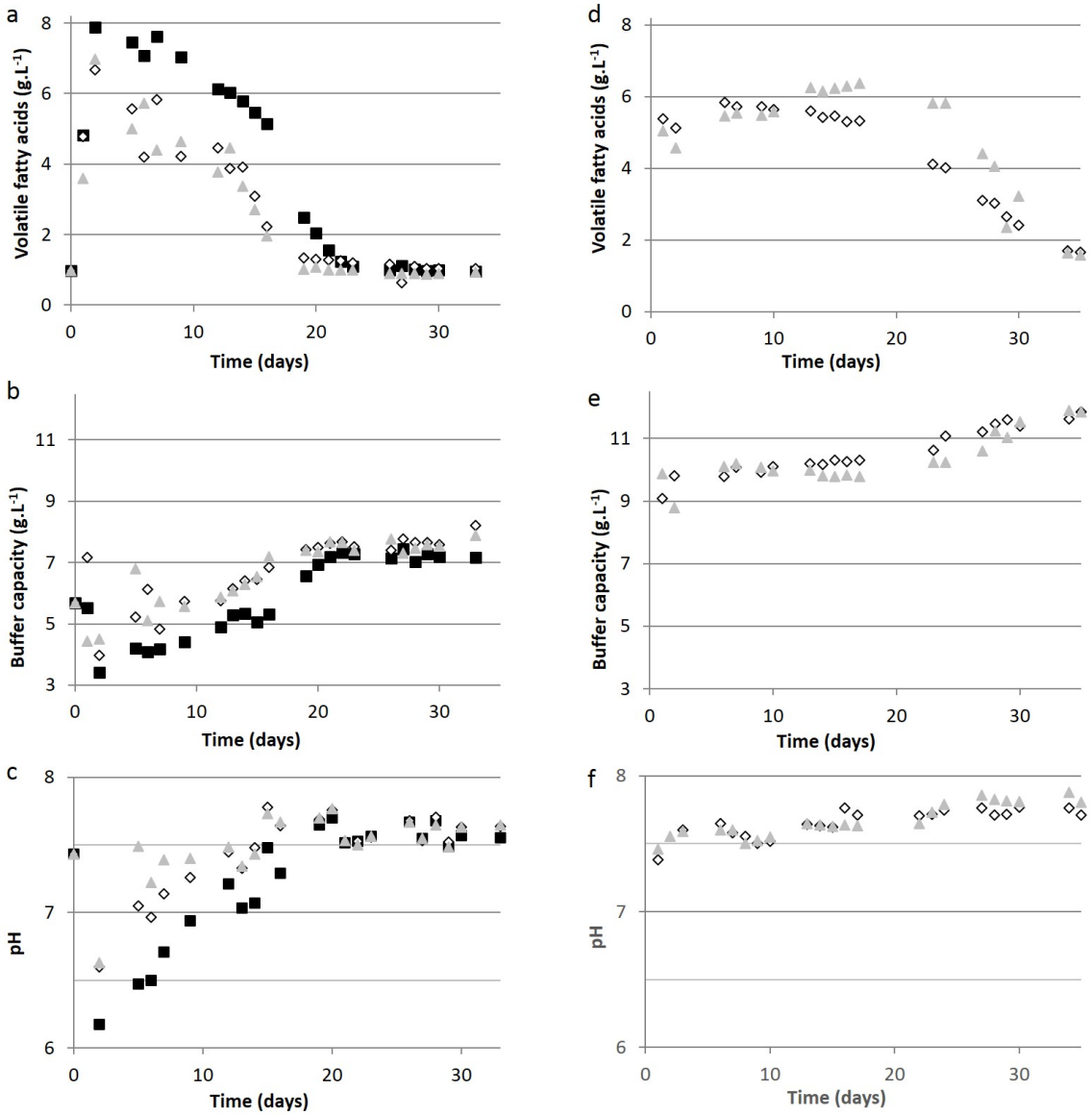
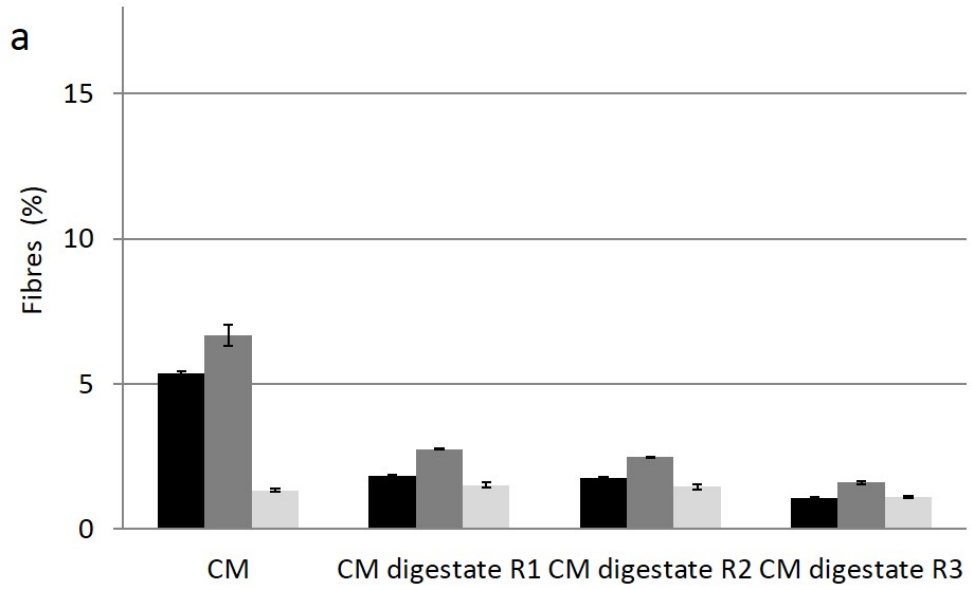
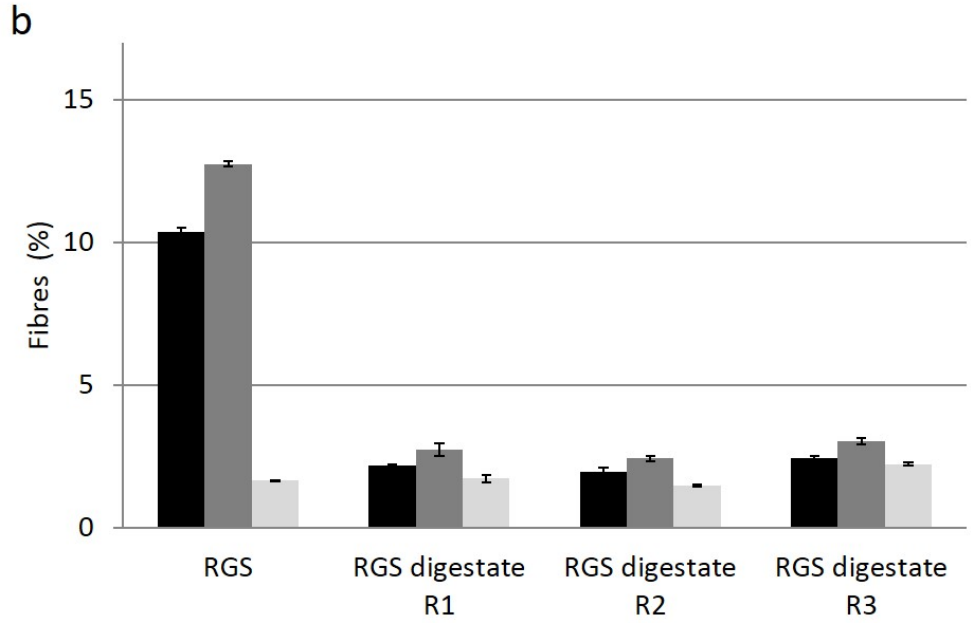
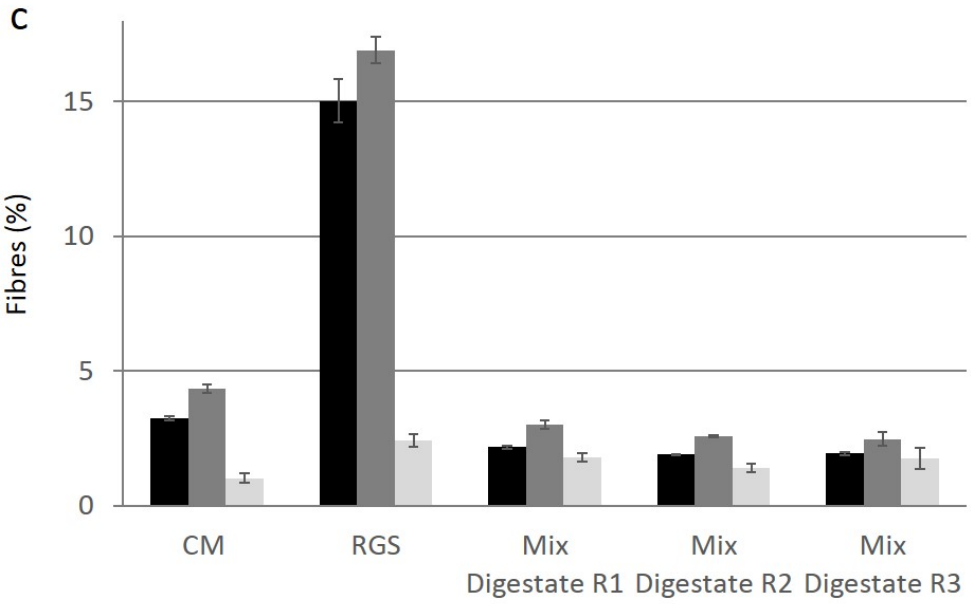
Cumulated methane ($Nm^3_{CH4}.t_{FM}^{-1}$)
0
20
40
0
10
20
30
Time (days)


b
Cumulated methane ($Nm^3_{CH4}.t_{FM}^{-1}$)
0
20
40
0
10
20
30
Time (days)

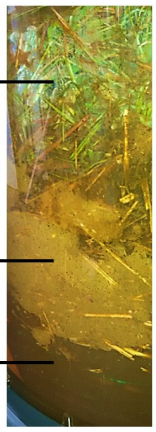

a
Volatile fatty acids (g.L-1)
Time (days)
b
Buffer capacity (g.L-1)
Time (days)
c
pH
Time (days)
d
Volatile fatty acids (g.L-1)
Time (days)
e
Buffer capacity (g.L-1)
Time (days)
f
pH
Time (days)


a
Fibres (%)
15
10
5
0
CM
CM digestate R1
CM digestate R2
CM digestate R3


b
Fibres (%)
15
10
5
0
RGS
RGS digestate R1
RGS digestate R2
RGS digestate R3


c
Fibres (%)
15
10
5
0
CM
RGS
Mix Digestate R1
Mix Digestate R2
Mix Digestate R3

First series



Filling method " in layers "

- Liquid phase was drained during AD process
- High percolation
- High recirculation

Second series

Filling method " in mixed "

- Liquid phase was not drained during AD process
- No percolation
- No/low circulation