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Horse Manure and Lignocellulosic Biomass Characterization as Methane Production Substrates

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Abstract: This paper aimed to study the value of horse manure through anaerobic digestion. The study involved characterization of different components of horse waste and the evaluation of their biochemical composition, physicochemical characterization and the influence of the composition of horse waste on biochemical methane potential. More specifically, two bedding mixtures were studied: the first one was composed of wheat straw (WS), wood chips (WC) and horse manure (HM) with a volumetric composition of 85%, 14% and 1%, respectively; and the second one was a mixture of WS and HM with a volumetric composition of 99% and 1%, respectively. The analysis was carried out on the two bedding mixtures and on each substrate separately with 406 samples from May 2017 to October 2019. Biochemical methane potential tests conducted on these samples showed that the composition and structure of the substrate influenced the BMP. WS had the highest mono-digestion methane production with 176.1 $NmL \cdot g_{VS}^{-1}$. The second bedding mixture (99% WS, 1% HM) showed a production of 189.4 $NmL \cdot g_{VS}^{-1}$ compared to 127 $NmL \cdot g_{VS}^{-1}$ by bedding mixture 1 (85% WS, 14% WC, 1% HM). The difference was due to a dilution effect on methane production caused by the presence of WC rich in lignin.

Keywords: horse waste; biomass characterization; anaerobic digestion; biochemical methane potential (BMP); lignocellulosic biomass

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1. Introduction

The French energy transition law for green growth, which was established on 17 August 2015, aims to preserve resources, assure the energetic independence of France and limit climate change. This law also focuses on the reduction in energy consumption by 50% between 2012 and 2050. This focus encourages the use of existing materials and resources to generate energy and preserve resources. The valorization of organic waste and, specifically, the anaerobic digestion process can play an important role to achieve these goals. The waste production in France in 2018 represents 342 millions of tons, including 39 millions of tons of municipal waste, 8.7 millions of tons were organic waste, including sludge from wastewater treatment [1]. When digested anaerobically, the biodegradable natural part of the biomass produces green, clean gas that can be used as biofuel. The most common biomass types used in AD are sewage sludge from wastewater treatment plants, animal waste and their byproducts, agricultural waste and the organic fraction of the municipal solid waste. The waste can be digested raw or pre-treated. To evaluate different types of biomass, a general characterization and a methane evaluation are needed. Lignocellulosic biomass usually refers to agricultural waste mainly composed of cellulose, hemicellulose and lignin. It is the most abundant and renewable material in the world [2]. A profound understanding of the physicochemical and biological properties of this biomass is essential for AD facilities' design and operation. The agricultural waste used in this research was horse waste, a mixture of feces, urine and bedding. The French National Groupement Hippique reported that the production of manure by an average horse farm is about 330 tons annually [3]. The Ile-de-France region's production is a little above the average, where more than 360,000 tons of manure is produced by the region's horses per year (IRSD 2015). The equestrian center of Maisons Laffitte produces 20,000 tons of horse manure per year. The need to valorize this waste led to the signature of a partnership between "Maisons Laffitte" and the "Service publique de l'assainissement francilien" (SIAAP) on 10 February 2017, to start the Valoéquiboue project. This project is a research and development project that aims to valorize horse manure by co-digestion with urban sewage sludge or by mono-digestion, using the anaerobic digestion method. This project's duration was 3 years (2017–2020) and it included three axes. The first axis aimed to characterize the different components of the horse waste (horse manure (HM), wheat straw (WS), wood chips (WC)) regarding their biochemical composition, biochemical methane potential (BMP) and physicochemical characteristics (density, humidity, total solids and volatile solids). The second axis focused on the production of biogas and transformation of the organic matter using two types of anaerobic co-digestion depending on the percentage of total solid (TS): wet anaerobic digestion for TS < 15% and solid-phase anaerobic digestion for TS > 15%. The third axis focused on metrological innovation including microbial diversity and 3D fluorescence/NIRS coupling. This paper treated the first axis regarding the characterization of horse waste that will be used in future studies as a substrate for anaerobic digestion. This topic brings interest to the scientific and industrial community at the same time. The other two axes of the project will be treated in future articles. Therefore, this study's primary objective was to investigate the potential of this raw agricultural waste. It also included a material characterization at laboratory scale, and an evaluation of the biological and physicochemical properties.

2. Materials and Methods

2.1. Substrates

The agriculture biomass considered in this study was generated from a horse stable in France (Eq'invest, Maisons-Laffitte), generating 1500 m^3 of waste material per year. Two types of bedding were selected: (1) a mixture of wheat straw (WS), wood chips (WC) and horse manure (HM) with a volumetric composition of 85%, 14% and 1%, respectively; and (2) a mixture of WS and HM with a volumetric composition of 99% and 1%, respectively.

The horse stall bed was cleaned daily, and the mixture of waste was stored in a container for a week. A sample was collected from the container surface to acquire the newest material. It was then manually sorted to separate each type of substrate. The method of sampling used for each type was quartering. This technique involved dividing the sample into quarters. The two opposite quarters were discarded, while the other two were combined and constituted the reduced sample. This method was repeated several times to obtain the final sample size needed. The samples were collected over the course of a year to evaluate internal and seasonal changes.

Prior to testing, each sample collected was homogenized and dried at 60 °C for 48 h to achieve proper workability. Then, all the material was passed through a shredder of 4 mm sieve for size reduction.

2.2. Physicochemical Analytical Methods

Physical, chemical and biochemical characterizations were completed to determine the AD process's stability, the composition of biogas generated and the biodegradability of the material.

The substrate's chemical and physical composition included proximate analysis tests (TS, VS and moisture content) and elemental analysis tests (carbon, nitrogen, phosphorus, potassium, magnesium and calcium content). Dry matter TS showed the total value of organic and inorganic compounds, VS content represented the organic compounds in the sample and macro-elements content confirmed the availability of necessary nutrients for a well-functioning AD process.

The biochemical composition included cellulose, hemicellulose and lignin content. It indicated the biodegradability and the structure of the sample.

All tests were executed in accordance with the standard methods listed in Table 1 below.

Table 1. Analytical methods used to characterize horse waste.

Parameter	*Unit*	*Standards Used*
Density	$kg \cdot m^{-3}$	mass/volume
Moisture content	%	Weight loss after water evaporation at 105 °C
TS	%	Heating 105 °C NFISO 11465
VS	%TS	Ignition 550 °C NTU44-160
TOC	%TS	NFEN15936
N	%TS	NFEN16168
P	%TS	NFENISO11885
K	%TS	NFENISO11885
Mg	%TS	NFENISO11885
Ca	%TS	NFENISO11885
Cellulose	$g \cdot g_{VS}{}^{-1}$	Van Soest method
Hemicellulose	$g \cdot g_{VS}{}^{-1}$	Van Soest method
Lignin	$g \cdot g_{VS}{}^{-1}$	Van Soest method

2.3. Biochemical Methane Potential

The BMP tests were conducted to estimate the biomethane potential and the biodegradability rate of the various substrates. The experiment was conducted using an automatic methane potential test system (AMPTS II, by Bioprocess Control) that showed the methane kinetic and the volumes of methane generated. Each type of biomass WS, WC and HM was analyzed alone, followed by two different bedding mixtures. The mono-substrate and the two separate mixtures were analyzed in four repetitive sets of experiments. All tests were performed in triplicates.

The inoculum used was sewage digested sludge taken from a full-scale mesophilic anaerobic digester of a municipal wastewater treatment plant (SIAAP-Achère).

A homogenous slurry of each sample was prepared and fed to a 500 mL bioreactor, leaving a 100 mL gas space. The bio-digesters were then placed in a thermostatic water bath creating a mesophilic environment of 37 °C (Aqualine AL 18, LAUDA). The substrate to inoculum ratio used was 0.3 (grams of VS of substrate per gram of VS of inoculum).

The biogas generated passed through a CO_2 trap, which allowed for exclusive methane measures to be taken, and the methane production was monitored for 27 days. Blank assay (only inoculum) was also carried out to determine the endogenous methane potential of the inoculum. It was later subtracted from the methane production obtained in the tested samples.

3. Results and Discussion

3.1. Characterization of the Biomass

Quantitative results of different analyses of biomass are shown in Table 2. Elemental analysis and biochemical compositions are represented based on the percentage of dry weight.

3.1.1. Proximate Analysis

Figure 1 shows that the density of different biomass feedstock showed extreme variations from lows of 115 and 195 $kg \cdot m^{-3}$ for WS and WC, respectively, to highs of 566.4 $kg \cdot m^{-3}$ for HM (Table 2).

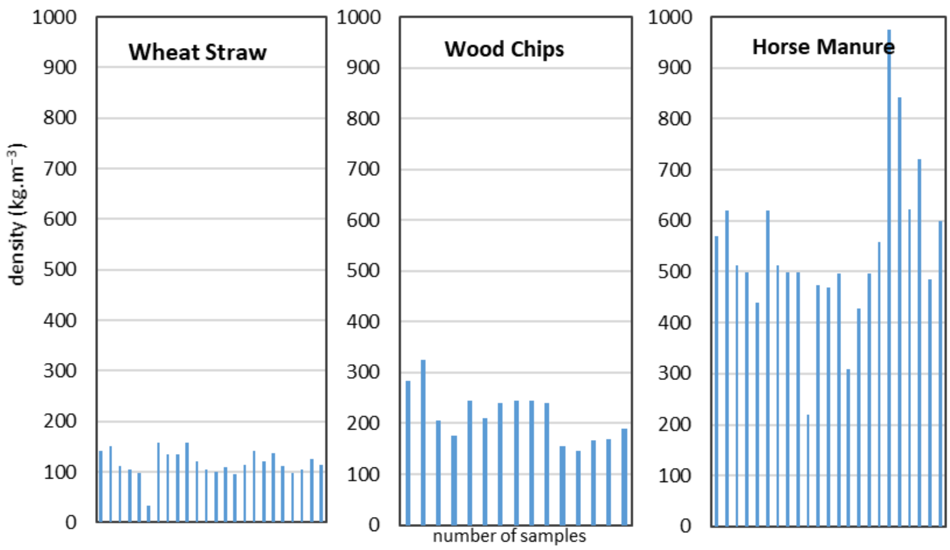

Figure 1. Density of the horse waste components.

Table 2. Characterization of the different types of biomass (values are presented as average ± standard deviation).

	Wheat Straw (WS)	*Wood Chips (WC)*	*Horse Manure (HM)*
Density ($kg \cdot m^{-3}$)	115.1 ± 25.4 (n = 21)	195.2 ± 92.2 (n = 12)	566.4 ± 138 (n = 21)
Humidity (%)	50.3 ± 17.7 (n = 22)	51.7 ± 12.1 (n = 13)	66.1 ± 11.4 (n = 22)
TS (%)	49.7 ± 17.7 (n = 22)	48.3 ± 12.1 (n = 13)	33.9 ± 11.4 (n = 22)
VS (%TS)	89.2 ± 5 (n = 22)	94.5 ± 2.3 (n = 13)	72.3 ± 21.2 (n = 22)
TOC (%TS)	38.1 ± 4.8 (n = 9)	29.9 ± 3 (n = 4)	30.2 ± 5.7 (n = 9)
TN (%TS)	0.9 ± 0.1 (n = 9)	1 ± 0.3 (n = 4)	1 ± 0.5 (n = 9)
P (%TS)	0.3 ± 0.1 (n = 9)	0.5 ± 0.2 (n = 4)	1.3 ± 0.6 (n = 9)
K (%TS)	1.6 ± 0.3 (n = 9)	1.4 ± 0.7 (n = 4)	1.3 ± 0.6 (n = 9)
Mg (%TS)	0.2 ± 0.01 (n = 9)	0.4 ± 0.1 (n = 4)	0.4 ± 0.2 (n = 9)
Ca (%TS)	0.9 ± 0.3 (n = 9)	1.4 ± 0.3 (n = 4)	1.1 ± 0.7 (n = 9)
C/N	46.4 ± 7.7 (n = 9)	33.8 ± 11.3 (n = 4)	32.3 ± 9.5 (n = 9)
Cellulose (%TS)	43 (n = 3)	49.27 (n = 3)	39.97 (n = 3)
Hemicellulose (%TS)	24.97 (n = 3)	17.45 (n = 3)	28 (n = 3)
Lignin (%TS)	17.7 (n = 3)	22.83 (n = 3)	9.19 (n = 3)

n: number of characterized samples.

The method used to determine the apparent density of the different feedstock was to measure the mass of a beaker of a specific volume (1 L), fill the beaker with the substrate until it reached the gauge mark and measured the difference in weight to determine the mass of 1 L of the substrate. This method was operator-dependent because it varied with the force that the operator applied to pile the substrate (especially the wheat straw). The errors can be reduced when the same operator measured the density of the substrate and applied approximately the same piling force, this same operator filled the reactors and, therefore, the same force was applied. It can be seen that for WS and WC, the apparent density's variation was not important; therefore, the operator assured the repeatability of the protocol; regarding the horse manure, and the density's variation did not depend on the operator.

Generally, the density of lignocellulosic biomass was relatively low compared to the density of other biomass (organic fraction of municipal waste 513 kg·m^{-3} and sewage sludge 1054 kg·m^{-3}) [4]. The density of biomass was also dependent on the degree of fill tightness. Packed fill can have a higher density than the loose fill. Density is an important parameter as it affects the residence time in the industrial digestion units. A low density will lead to a drop in HRT. The WS, WC and HM moisture average content were 50.3%, 51.7% and 66.1%, respectively. Similar results were shown in the organic fraction of municipal solid waste and sewage sludge with average content of 59.83% [5] and 96.8% [4], respectively. Moisture plays an important role in the degradation processes, and the values obtained indicate that the substrate had adequate moisture for the AD. However, moisture in lignocellulosic biomass can reside outside the wall or can be absorbed within the cell wall. Water in the cell wall interacts with hydroxyl groups of the constituting polymers (cellulose, hemicellulose and lignin) by hydrogen bonding [6].

Total solids is an important parameter in the design of anaerobic digestion processes. It plays a significant role in determining the organic loading charge and the methane yield per volume of the digester. Additionally, total solids were one of the significant characteristics influencing the choice of digestion method. High TS (>20%) favored the solid-state AD, a range of 10 to 20% of TS favored the hemi-solid AD and lower TS ($\leq$10%) in the liquid state AD [7,8]. The biomass showed high TS percentages ranging from 34% to 50%. Researchers showed that the TS content varied from about 30% to 35% in horse manure [9] and 50% to 70% in bedding material [10].

WS and WC substrates showed high VS (%TS) ranging from 89% to 95%, ensuring high solid organic content. Those percentages were similar to the ones found in the literature of around 80% to 99% corresponding to bedding material [10]. However, they showed a very high percentage when compared to other types of biomass (animal manure 72%, organic fraction of municipal waste 77% and sewage sludge 65%) [11–13]. The results indicate that the biomass was rich in degradable matter, making it suitable for a biological process aimed at converting the organic material into biomethane.

3.1.2. Elemental Analysis

As shown in Table 2, carbon was found to be the dominant element. The TOC content of the WS, WC and HM were 38.1% TS, 29.9% TS and 30.2% TS, respectively. [14] showed that the carbon content of a typical biomass was around 45%.

In addition to the carbon source, substrates should also meet the nutritional requirements of the microorganisms in terms of energy source and should include various macroelements essential for the growth of the microbial activity [15]. Based on the values obtained in Table 2, all three substrates contained various nutritional elements, such as nitrogen, phosphorus, potassium and magnesium. Macronutrient requirements were mainly assessed based on C: N: P: S ratio. The optimum ratio for methane yield enhancement was reported to be 600:15:5:3 [16]; the biomass showed similar ratios (Table 2). Another important indicator was the C/N ratio. Studies showed that the optimal C/N ratio differs among substrates, and a C/N ratio between 15 and 50 indicates a well-functioning biogas process, as microorganisms use carbon 25–30 times faster than nitrogen. [17]. As shown, the C/N ratio of the biomass ranged from 32 to 46 (Table 2).

Previous studies also reported that macronutrients such as calcium have significant effects as additives during AD [18,19]. This element was also present in the substrates, as shown in Table 2.

3.1.3. Biochemical Composition

Table 2 shows that 85% of WS, WC and HM biomass carbon sources consisted of carbohydrates material that fell under the polysaccharides group. They were composed primarily of cellulose, hemicellulose and lignin.

WS had 43% TS of cellulose, 25% TS of hemicellulose and 17.7% TS of lignin. The highest lignin and cellulose content were found in WC with 17.45% TS and 49.3% TS, respectively. HM showed the lowest lignin content with 9.2% TS. The results obtained were similar to the numbers seen in the literature that said that the basic structure of lignocellulose biomass was comprised of cellulose from 40 to 50%, hemicellulose from 25 to 35% and lignin from 15 to 20% [20].

Cellulose, hemicellulose and lignin are composite polymers with complex structures and these polymers are associated with each other [21]. Researchers showed that cellulose and hemicellulose can be slowly biodegraded in an AD and can reach 60% of biodegradability [22]. However, lignin had little value for bioenergy production and can serve as both a physical and biochemical barrier that inhibits most biomass-to-bioenergy conversion processes [23].

3.2. Biochemical Methane Potential of the Biomass

3.2.1. BMP Test Results

Different components of organic material can provide varying quantity and quality of biogas, depending on their energy content and digestibility in an AD. Biodegradability of biomass feedstock was determined by the content of easily degradable compounds such as sugars, low-degradable compounds such as hemicelluloses and cellulose and compounds such as lignin, usually passing undigested through the biogas reactor [24].

As shown previously, the primary energy sources of those complex substrates were cellulose, hemicellulose and lignin. Researchers showed that the biodegradation of cellulose was affected by the content of lignin that acts as a strong structural material. It is also very resistant to microbial degradation, thus making the access of microorganisms to the abundant polysaccharides energy source difficult [21].

Biochemical methane potential (BMP) is a measure of the amount of methane that can be produced through the anaerobic digestion of substrates. It is usually expressed as the methane yield per unit mass of volatile solids (VS). The cumulative methane yields obtained are presented in Figure 2. WS had the highest mono-digestion production with 176.1 $\mathrm{NmL \cdot g_{VS}}^{-1}$, the methane yield obtained was in the range of the typical values reported in the scientific literature (168.8 $\mathrm{NmL \cdot g_{VS}}^{-1}$ for WS after a pretreatement at 175 °C and 181.4 $\mathrm{NmL \cdot g_{VS}}^{-1}$ for unpretreated WS) [25]. WC was the substrate with the highest percentage of lignin and it presented the lowest production of methane with 77.2 $\mathrm{NmL \cdot g_{VS}}^{-1}$. According to [26], wood is resistant to microbial degradation and WC bedding resulted in low methane yield in comparison with other bedding materials. The literature also showed that methane production is known to be less from lignocellulosic substrates, which are high in lignin content [27]. HM showed a production of 147.4 $\mathrm{NmL \cdot g_{VS}}^{-1}$, which was very similar to the production of animal manure shown in the literature [26].

The use of straw as a bedding material offers a value for energy recovery. The results of BMP demonstrated that wheat straw exhibited the highest methane yield among the three bedding materials studied. This finding aligns with previous studies comparing straw to other bedding materials, such as softwood chips or pellets, where straw was shown to outperform them in terms of methane production [28].

The HW bedding mixture 1 and the HW bedding mixture 2 showed a production of 127 $\mathrm{NmL \cdot g_{VS}}^{-1}$ and 189.4 $\mathrm{NmL \cdot g_{VS}}^{-1}$, respectively. When comparing the mixtures to WS digestion, bedding 1 showed a lower methane production indicating a diluting effect on methane production caused by the presence of WC. This was also shown in [28].

The theoretical BMP of a substrate is the weighted average of theoretical BMP of biochemical components (cellulose, hemicellulose and lignin). Knowing the formula of these components, their biochemical methane potential was calculated based on the Buswell and Mueller formula [9].

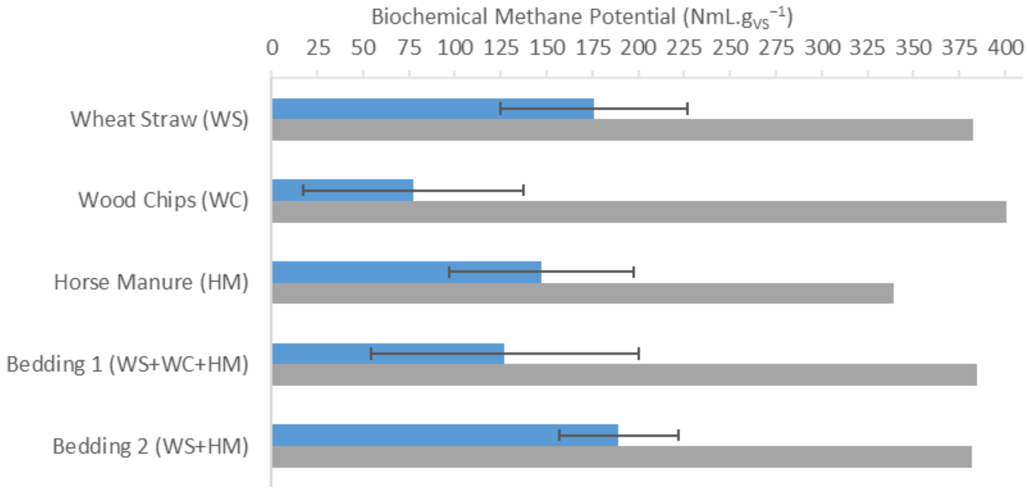

Figure 2. BMP from the different biomass studied (average ± standard deviation) (blue: experimental BMP, gray: theoretical BMP).

By comparing the cumulative methane yields obtained to theoretical BMP, it can be seen that the theoretical value cannot be attained. This can be due to several reasons, such as the variation of biodegradability of the biochemical components [9] and the use of components for the development of microorganisms [11,29,30].

Additionally, the substrates containing wood chips represent a less important yield than the other substrates which can be explained by the high content of lignin present in wood chips.

When compared to other biomass, raw bedding material showed low BMP values. Based on several studies, sewage sludge and organic waste (OFMSW, FVWs (fruits and vegetables solid waste), household waste) showed BMP values of 250 NmL·g_{VS}^{-1} [13] and around 400 NmL·g_{VS}^{-1}, respectively [11,29,30].

The addition of wheat straw to horse manure in bedding mixture 2 resulted in a methane yield of 189.4 NmL·g_{VS}^{-1} ± 32.2. This value represents the weighted average of methane production when each bedding material (wheat straw and horse manure) was digested separately. According to [31], when straw is mixed with animal feces, it creates a dilution effect on the properties of the feces. The specific properties affected by this dilution effect included total solids, volatile solids, C/N ratio, fiber composition and BMP. Therefore, the characteristics of bedding mixture 2, which consisted of horse manure and wheat straw, would exhibit properties that were intermediate between those of pure feces and pure straw. The exact values of these characteristics would depend on the proportions of the two components in the mixture and how they interact during the digestion process.

On the other hand, the literature demonstrated that when animal feces and straw ae mixed, it can lead to a decrease in the total amount of nitrogen. This reduction is advantageous in anaerobic digestion processes and can have an inhibitory effect. Additionally, the mixture tends to increase the C/N ratio to reach optimal values for efficient anaerobic digestion [32]. This effect was observed for both bedding mixtures, where BMP was slightly higher compared to the average BMP of each material digested separately.

Furthermore, horse manure provides a favorable environment for the growth of bacterial and fungal organisms responsible for the degradation of cellulose and hemicellulose present in bedding materials.

3.2.2. Kinetic Study

Figure 3 below shows the average methane production yield during the experiments. It can be seen that WS showed the highest value of methane yield. The curve of WS showed longer logarithmic phase compared to WC, which indicated additional growth of microorganisms that can be correlated to the biodegradability of the waste (lower lignin content).

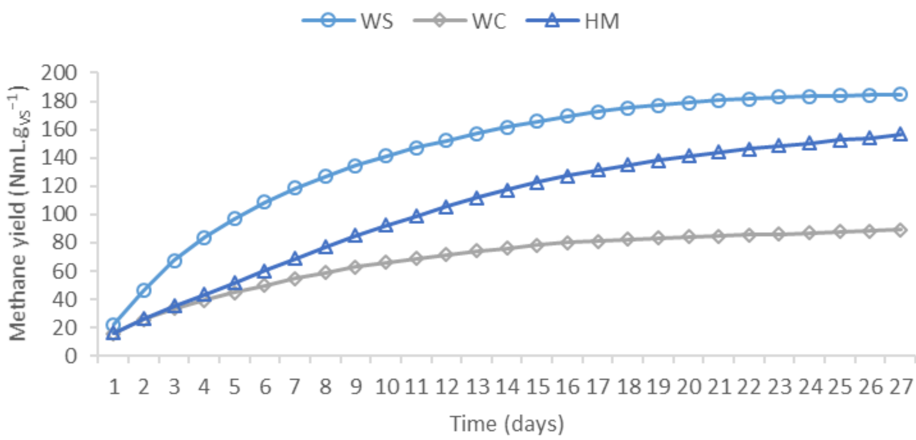

Figure 3. Average production yield of the different fractions of HW (calculated average of 4 sets of experiments).

The figure also showed that the lowest values of methane yield were reached by WC and this, as explained earlier, could be correlated to the relatively high amount of lignin content that affects the enzymatic conversion of lignocellulosic biomass. Lignin is a major contributor to biomass recalcitrance due to its protective structure and hydrophobic nature [33]. The existence of this component led to a low biomass degradation. The curve of WC also showed a shorter logarithmic phase, showing that the microorganisms are running out of substrate food since the biomass is very hardly biodegradable.

HM had a steeper slope but a lower methane production, showing less food substrate content (a lower VS% when compared to WS), but a higher biomass degradation (a low lignin content). The curve of HM also showed that the biomass did not reach the saturation phase.

A total of 80% BMP of the biomass was reached on days 12, 9 and 14 for WS, WC and HM, respectively. Longer time was needed for the degradation of lignocellulosic waste showing longer hydrolysis rate [34]. Additionally, according to [22], hemicellulose was the easiest to decompose, then came the cellulose, and finally, the lignin that was resistant to breakdown.

4. Conclusions

Individual bedding materials and two bedding mixtures of horse waste were studied, and their physicochemical properties were compared.

Wheat straw, wood chips and horse manure exhibited different densities, TS, VS, C/N ratio and biochemical compositions. WS yielded the highest methane production with a value of 176.1 NmL·$g_{VS}{}^{-1}$, followed by HM with a BMP of 147.4 NmL·$g_{VS}{}^{-1}$. WC had the lowest BMP of 77.2 NmL·$g_{VS}{}^{-1}$, likely due to its high lignin content (22.83%), which acts as a resistant structural material.

Furthermore, two bedding mixtures were studied. Bedding 2, composed of 99% wheat straw and 1% horse manure, achieved a higher methane yield compared to bedding 1, which consisted of 85% WS, 14% WC and 1% HM with respective biochemical methane potentials of 127 NmL·$g_{VS}{}^{-1}$ and 189.4 NmL·$g_{VS}{}^{-1}$.

The results obtained demonstrate that the horse waste can be considered a suitable substrate for AD, offering a significant energy recovery potential through methane production.

Overall, the research provides valuable insights into the characterization of horse waste as a substrate for anaerobic digestion. Understanding the physicochemical and biological properties of horse manure and bedding is crucial for the design and operation of anaerobic digestion facilities. Moreover, these substrates showed an energy contribution, emphasizing the importance of considering agricultural waste as a valuable resource rather than waste.

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