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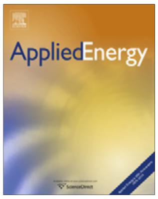

How can we improve biomethane production per unit of feedstock in biogas plants?

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ABSTRACT

Biogas production is one of the number of tools that may be used to alleviate the problems of global warming, energy security and waste management. Biogas plants can be difficult to sustain from a financial perspective. The facilities must be financially optimized through use of substrates with high biogas potential, low water content and low retention requirement. This research carried out in laboratory scale batch digesters assessed the biogas potential of energy crops (maize and grass silage) and solid manure fractions from manure separation units. The ultimate methane productivity in terms of volatile solids (VS) was determined as 330, 161, 230, 236, 361 L/kg VS from raw pig slurry, filter pressed manure fiber (FPMF), chemically precipitated manure fiber (CPMF), maize silage and grass silage respectively. Methane productivity based on mass (L/kg substrate) was significantly higher in FPMF (55 L/kg substrate), maize silage (68 L/kg substrate) and grass silage (45–124 L/kg substrate (depending on dry solids of feedstock)) as in comparison to raw pig slurry (10 L/kg substrate). The use of these materials as co-substrates with raw pig slurry will increase significantly the biomethane yield per unit feedstock in the biogas plant.

1. Introduction

In the latter 20th century, anaerobic digestion gained popularity as a solution to environmental and energy concerns. Anaerobic digestion embraces the concept of sustainability and proximity. Local wastes are used to generate local energy whilst minimizing pollution and substituting for mineral fertilizers. Twenty years ago the process was mainly used for treatment of wastewater sludge. More recently the farming sector, particularly in Denmark, Germany and Austria embraced biogas technology co-digesting farm wastes with some imported feedstocks [1–3].

Slurries due to their high water content (in excess of 90%) have relatively low specific methanogenic capacities, hence it is difficult to make the biogas plants economically viable. Large watery wastes require large reactors which are expensive. Parasitic demand is excessive due to the need to raise the temperature of this water (4.184 kJ/kg/°C). This is particularly true for thermophilic digesters (ca. 55 °C) which tend to dominate in Denmark. This leads to a demand for imported feedstocks with high energy content and potential to increase the gas production per unit of feedstock. The obvious feedstocks are residues with gate fees: fatty residues from food and pharma industry which are not negatively affected by the Animal by-products Regulation [4]. But these resources are limited; they are the low hanging fruit. New facilities particularly in Denmark and Germany may require alternative feedstocks. Germany in particular has pioneered the use of energy crops. These crops tend to be coarser biomass including for starches and lignocellulosic material such as beets, whole crop maize silage, grass silage and millet [5–9].

The transport of slurry from the farm to the biogas plant is a considerable logistical and financial cost for co-digestion facilities. It is one of the reasons for poor economical performance of biogas plants treating primarily slurry. The economic feasibility of such facilities can be improved through reduction of transport costs and increasing the digester efficiency [10–12]. Thus there are considerable advantages to reducing the water content of slurries through separation into a fibrous fraction (high in nutrient and in solids content) and a liquid fraction. Technologies available include for: mechanical screen separators, filter presses, sedimentation, centrifugation, biological treatment and reverse osmosis [13]. Substitution of raw pig slurry in a digester with the separated solid fraction can lead to an increase in biogas production per unit mass, whilst also reducing transport costs and parasitic thermal demands (less water to heat). The methane yields of the separated solids from different separation methods may be different because there are different processes involved and there is different particle size distribution in the solid biomass separated by different systems

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[13]. The carbon lost to the liquid fraction must also be considered. An ambition of this paper is to assess the biogas production from filter pressed and chemically precipitated manure fibers.

The context of this paper is based on the Centralised Anaerobic Digestion (CAD) system employed in Denmark. These facilities predominately treat feedstock consisting of 80% slurry with 20% imported feedstock at a scale of up to 80,000 t/a. Slurry produces of the order of 24 m^3 biogas/t and does not generate a gate fee. The imported feedstock is relied on to increase the biogas production per unit of feedstock. For example if the imported feedstock generates 150 m^3 biogas/t then the average biogas production for all the feedstock would be of the order of 50 m^3/t. This obviously makes the facility far more energy productive and improves the potential for financial viability.

This paper also is concerned with the myriad of results available in the scientific literature on biogas production per unit of feedstock. Nizami and Murphy [11] in reviewing the literature on biomethane from grass silage found values in the range 0.197–0.47 m^3 CH_4 per kg VS added. Feedstocks can be specific to their geographical situation. Korres et al. [7] highlight the fact that biomethane yield from grass silage is dependent on the age of the grass, the grass species, the fertilizer applied, the climate of the region, the length of the growing season, the time of the day the grass is cut. Similarly, yields of maize in Austria differ from Denmark and Ireland. It is not prudent to simply take data from the literature that may have been generated in one country and apply to another. Thus this paper takes all its feedstock from a region in Denmark.

In brief the aim of the research is to investigate the biogas and methane potential of different substrates all from the same region, namely whole maize silage, grass silage, pig slurry and separated manure fiber fractions from manure separation units. The application of this is to improve the biomethane production per unit of feedstock in Centralised Anaerobic Digestion facilities plants designed primarily for slurries.

2. Material and methods

2.1. Materials used

The materials used in the study are maize silage, grass silage, FPMF (filter pressed manure fiber) and CPMF (chemically precipitated manure fiber). Biogas potential from the raw pig slurry is used as a reference to facilitate comparison. Maize silage, grass silage, raw pig slurry and filter pressed manure fiber fraction (Samson Bimatech, Denmark) are collected from Asdal biogas plant located near Hørring, while chemically precipitated solid manure fiber fraction is obtained in the laboratory by using a precipitant and polymer. The chemically precipitated fibers were obtained from the same slurry from the same site to avoid any special variation in the composition of the slurry. The chemical precipitant and polymer used for this experiment was selected from Kemira technology (Kemira Miljø; Esbjerg, Denmark), which provides separators at pig farms for solid liquid separation. The polymers used were super floc C-2260 and precipitant used was iron chloride hexahydrate ($FeCl_3{\cdot}6H_2O$).

Polymer solution (0.5%) was prepared in a 500 ml flask and mixed thoroughly for 45 min on a shaker. Three liters of raw pig slurry were collected in a container and continuously mixed by means of a stirrer while adding 30 ml of 1 M iron chloride solution (10 ml/L manure). 450 ml (150 ml/L manure) of polymer solution was added to the manure in small intervals to avoid the formation of large flocs. After floc formation the mixture was transferred into two 1 L columns and left to settle for about 3 h. The supernatant was removed and the flocs were transferred into containers. All the materials were stored at 5 °C until use. Anaerobically digested sewage sludge was used as inoculum. The inoculum was collected from Aalborg East waste water treatment plant and stored in a closed container at room temperature for 10 days before use.

2.2. Dry Matter and volatile solids

Dry matter (DM) content was determined by drying a known weight of sample (W_s) in an oven at 105 °C for 24 h and measuring the weight after drying (W_{DM}). The dry matter content was calculated by the expression DM (%) = 100 × W_{DM}/W_s. To determine the volatile solid, dried samples were combusted at 550 °C for 12 h and the weight of ash (W_{ash}) was measured. The volatile solid content (VS) was calculated by the expression $(100 \times (W_{DM} - W_{ash})/W_{DM})$. The volatile solid content of the substrates (VSs) was determined at the beginning of the experiment. After 56 days of digestion, the volatile solid content of the digested mixture (VSm) and inoculum (VSi) were determined.

2.3. Preparation of samples for biogas production

After measurement of dry matter (DM) and volatile solids (VS) the different raw materials (substrates) were placed in the serum bottles together with inoculum and wash water in accordance with the substrate to inoculum ratios. The five feedstocks (maize silage, grass silage, pig slurry, FPMF and CPMF) plus the blank (inoculum without feedstock) were each tested twice. Thus in total there were 12 samples. These 12 samples were tested for gas production during the laboratory scale batch experiment. The results presented in the figures and tables are the mean of these two replicates.

2.4. Measurement and analysis of biogas production

In a 1 L serum bottle 500 g of inoculum was added followed by the addition of 50 g of substrate and 50 g of tap water. The air in the bottles was flushed out with nitrogen gas before closing the bottles tightly with rubber stoppers and metal screw-on caps with a gap provided for gas collection. To estimate the effect of inoculum on gas production, a blank sample was prepared in a serum bottle by adding 500 g of inoculum and 50 g of tap water. As mentioned all the samples were prepared in duplicates. These samples were then transferred in the oven at 53.5 °C (thermophilic anaerobic digestion).

The volume of biogas produced was measured by the syringe method every day in the first week. The frequency was reduced to 2 days in the second week; two times in the third week and then once a week for the remaining period (5 weeks). The quality of the biogas was tested every week in the first three weeks by collecting biogas samples and testing them in a Gas chromatograph (Chrompack CP9001) for methane and carbon dioxide content. For the remaining period, the quality of biogas produced was analysed once in 2 weeks.

3. Results and discussion

3.1. Methane productivity from fractions of manure

The characteristics of all the substrates with respect to DM and VS before biogas production are given in Table 1. The methane production calculated for manure fibers and raw pig slurry is presented in Fig. 1 and Table 2. The ultimate methane yield per kg VS is lower for FPMF (filter pressed manure fiber) (average 161 ± 19 L/kg VS) and CPMF (average 230 ± 51 L/kg VS) than for raw pig slurry (average 330 ± 49 L/kg VS) as shown in Fig. 1 and Table 2. The methane potential of organic particulate matter

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Table 1
Dry matter and volatile solid content of substrates before anaerobic digestion.

Materials (substrates)	% Dry solids	% VS before digestion
Filter pressed manure fiber	38	89
Chemically precipitated manure fiber	7	76
Raw pig slurry	4.3	73
Maize silage	30	96
Grass silage	15	86
Anaerobically digested sewage sludge (inoculum)	3.5	49

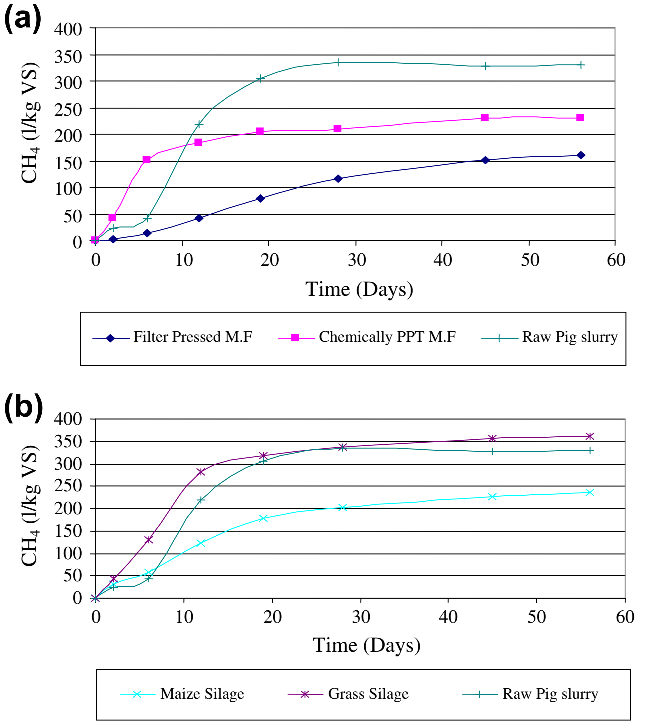

Fig. 1. (a) Cumulative methane production from the manure fibers and raw pig slurry (b) from maize silage and grass silage.

Table 2
Methane production from different substrates obtained from batch experiment.

Materials (substrates)	Methane potential	
	(L/kg VS)[a]	(L/kg substrate)
Filter pressed manure fiber	161 ± 19	54.5 ± 6.4
Chemically precipitated manure fiber	231 ± 51	12.3 ± 2.7
Raw pig slurry	330 ± 49	10.4 ± 1.5
Maize silage	236 ± 52	68.0 ± 15.0
Grass silage	361 ± 8	46.5 ± 1.03

[a] Results are mean of duplicate tests and values in ± shows the standard deviation.

increases with decreasing particle size because of increase in surface area [7]. The particle size in the filter pressed manure is more than 1 mm, whereas chemically precipitated manure has smaller particles due to the addition of precipitant during the precipitation process. Therefore, the chemically precipitated manure has a higher methane yield in terms of VS as in comparison to filter pressed manure, and the raw pig slurry has even more yield than the other two. Obviously in separating fibers from the slurry some of the readily biodegradable soluble carbon content follows the route of the liquor reducing the potential for methanisation. These values are in line with the findings of Møller et al. [14]. He observed the methane potential from centrifuged manure fibers (average 194 L/kg VS), chemically precipitated manure fibers (average 247 ± 25 L/kg VS) and raw manure (356 L/kg VS). Andara and Esteban [15] observed methane potential of 165 L/kg VS in the solid fraction of manure derived from filtration.

Moreover, the volumetric methane yield of filter pressed manure (54.5 ± 6.4 L/kg substrate) is considerably higher (Table 2) in comparison to raw pig slurry (10.4 ± 1.5 L/kg substrate) and chemically precipitated manure fibers (12.3 ± 2.7 L/kg substrate). Filter pressed manure has almost 5 times higher volumetric methane productivity than raw pig slurry and chemically precipitated manure. This is because the filter pressed manure has high dry matter content (38%) when compared to chemically precipitated manure (7%) and raw pig slurry (4.3%). The chemical precipitation of manure performed in the laboratory yielded only 7% dry matter content. However solid–liquid manure separators (Kemira Miljø; Esbjerg, Denmark) used in farms can achieve a dry matter content of about 35%; thus yielding volumetric methane of the order of 55 L/kg substrate. The volumetric methane potential observed by Møller et al. [14] for different manure fractions was also between 50 and 55 L/kg substrate.

3.2. Methane productivity of maize silage and grass silage

The ultimate methane yield per kg VS is lower for maize silage (average 236 ± 52 L/kg VS) than grass silage (average 361 ± 7 L/kg VS) as shown in Fig. 2 and Table 2. The grass silage is easy to digest by anaerobic bacteria [7] in comparison to maize silage. Additionally, the plateau phase during the batch digestion is reached faster with grass silage than maize silage (Fig. 2), indicating that retention time seems to be longer during digestion of maize silage. Mähnert et al. [16] investigated the methane potential of different grass species (fresh and silage) and found the methane yield range from 310 to 360 L/kg VS. Nizami et al. [7] and Nizami and Murphy [11] also reported the same range of methane potential from grass silage.

According to Braun and Wellinger [17], 200–300 L of methane can be produced from 1 kg of maize (dry matter). The amount of methane produced in this study by maize (236 L/kg VS) lies close to the median of this range. The volumetric methane yield of maize silage (ca. 68 L/kg substrate) is considerably higher than grass silage (ca. 46 L/kg substrate). This is because in this study maize silage has higher dry matter content (30%) than grass silage (15%). This is not always true. Various researchers [18–23] have indicated that grass silage may have a dry solids content of up to 40% due to field wilting in climates with warm dry summers such as Austria [21]. In temperate climates such as Ireland grass silage may be baled (30% dry solids content) or clamped (from a pit at 20% dry solids) [19–21]. Using these three dry solids content the volumetric methane yield for grass silage would increase to:

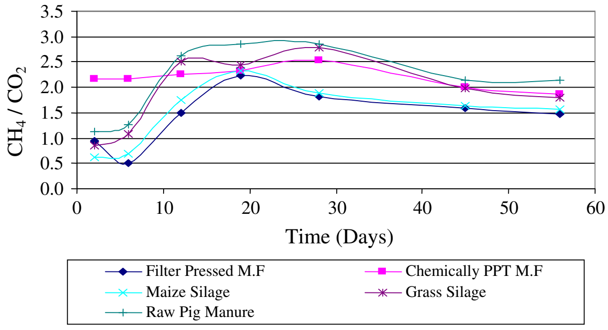

Fig. 2. CH_4–CO_2 ratio for different substrate with time.

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- 62 L/kg substrate at 20% dry solids (pit silage from Ireland);
- 93 L/kg substrate at 30% dry solids (bale silage from Ireland); and
- 124 L/kg substrate at 40% dry solids (field wilted silage from Austria).

3.3. Composition of biogas

It can be seen (Fig. 2) that except for chemically precipitated manure fibers, the ratio of CH_4/CO_2 is very low for all the materials initially due to slow start-up of methanogenic bacteria [7]. The reason for the higher CH_4/CO_2 for chemically precipitated manure could be due to the chemicals applied to slurry, which might have changed the carbon to a more reduced form. The CH_4/CO_2 ratio increased from 2.0 to 2.8 after 2 weeks and then stabilized between 1.8 and 2.0 for grass silage, raw pig slurry and chemically precipitated manure fibers, and around 1.5 for maize silage and filter pressed manure fibers. Typically biogas from grass and maize silage has methane content of the order of 54–60% [18–21] which corresponds with a CH_4/CO_2 ratio in the range of 1.2–1.5. Biogas from slurries would have a methane content of about 60–70% [24] which corresponds to a CH_4/CO_2 ratio of 1.5–2.3.

3.4. Degradability of substrates

Fig. 3 shows the overall mass flow of all the substrates used in the experiment. It can be seen from the figure that conversion of volatile solids to biogas for all the different substrates falls between 35 and 70%, which is almost in accordance with theoretical expectations. VS conversion in raw pig slurry is relatively high (55%) as in comparison to chemically precipitated manure (41%) and filter pressed manure (35%). This is in close agreement with Møller et al. [14] who observed that volatile solid conversion by anaerobic digestion for raw pig slurry was 62% and for centrifuged manure was 37%. This is due to the more biodegradable nature of raw pig slurry in comparison to solid manure fractions with associated lignin content. It can also be seen in Fig. 3 that almost 56% of VS in the case of filter pressed manure and 45% of VS in the case of chemically precipitated manure are present in serum bottles after 56 days of digestion. This leads to a discrepancy of between 10 and 15% of VS. These must be assumed to be either converted to other gases (other than CH_4 and CO_2) or lost as leakage during measurements. Moreover, the VS conversion to biogas in the case of grass silage is very high (almost 70%) in comparison with maize silage (54%). However, maize silage still has a higher conversion efficiency as compared to raw pig slurry and manure fibers. This indicates that grass silage and maize silage are more readily degradable than raw pig slurry and manure fibers. For grass silage 30% of VS are still present in the serum bottle after 56 days of the digestion period and only 1% is converted to other gases or lost. Residual volatile solids in the serum bottle may be an issue for greenhouse gas emissions in land application of digestate.

3.5. Effect of substrates on volumetric methane production from the raw pig slurry

The methane potential (measured in units of L/kg VS) is 1.4 times higher from chemically precipitated manure fiber than for filter pressed manure fiber (Table 2). However the volumetric methane yield (L/kg substrate) of chemically precipitated manure is low due to the low dry matter content (7%); the dry solids content is about 5 times less than the filter pressed manure fiber (Table 1). The combined effect is that the volumetric methane yield from chemically precipitated manure fiber is about 4.5 times less than filter pressed manure fiber (12.3 L/kg substrate vs. 54.5 L/kg substrate). Due to the higher VS content in the separated manure fiber (expressed as kg VS/kg substrate) its use as a co-substrate with raw pig slurry will increase the volumetric methane yield of the overall mixture.

What would this mean to a CAD facility based on raw pig slurry? If the investigated feedstocks comprised a portion of the total feedstock what would this do to the production of methane? Fig. 4a shows the *potential* increase of methane (L/kg feedstock) if 100 g of substrate is mixed with 1 kg of raw pig slurry. Fig. 4a is based on a simplified assessment using the results of the batch tests in Table 2. In essence Fig. 4a is extrapolated from the

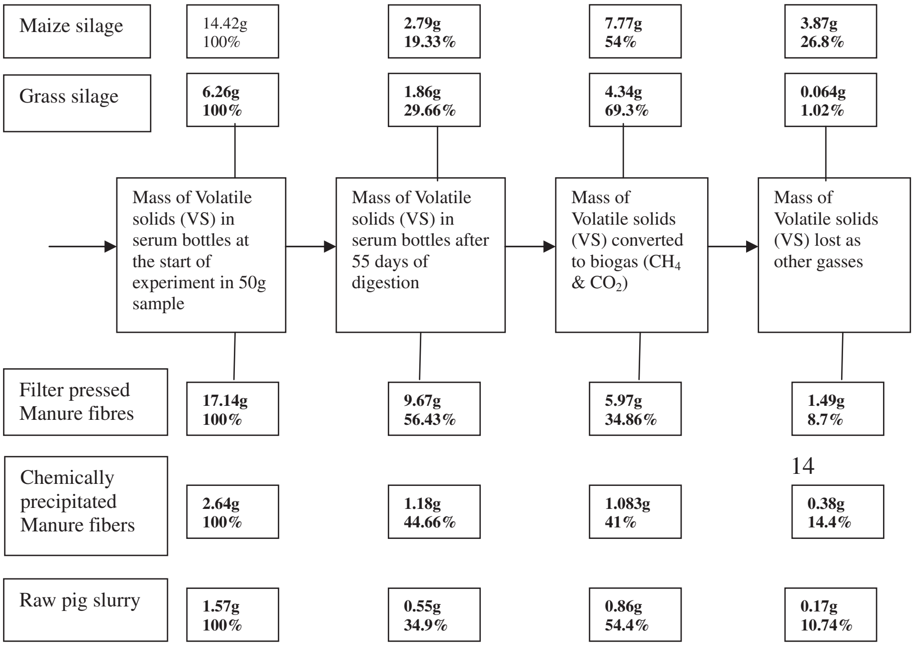

Fig. 3. Mass balance from batch experiment. The figure is a statement of conservation of mass. For each feedstock read across the 18 columns. The "column headings" are indicated by the arrows.

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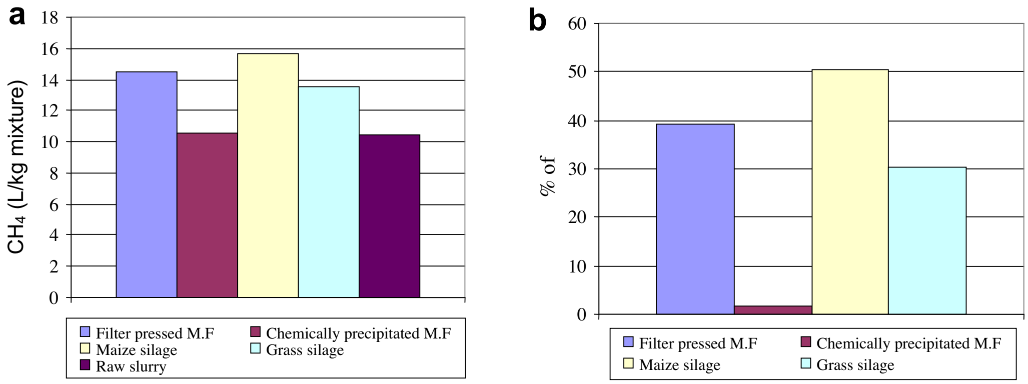

Fig. 4. (a) Increase in the volumetric methane yield (L/kg of mixture) from the raw pig slurry by adding different substrates in the ratio 1:10 substrate to raw pig slurry. (b) Increase in the volumetric methane yield expressed as a percentage increase of CH_4.

mono-digestion of the substrates to exemplify what these batch tests could actually mean to the gas production of a digester designed predominately for pig slurry. For example the addition of FPMF to pig slurry in the ratio of 10:1 equates to approximately 9.1% of the feedstock. Thus the methane production may be assessed as below:

- 90.9% (10.4 L CH_4/kg pig slurry) + 9.1% (54.5 L CH_4/kg FPMF) = 14.4 L CH_4/kg feedstock.

This corresponds to a 38% increase in methane (L/kg feedstock) for a substitution of 9.1% of pig slurry with FPMF. The increases in volumetric methane yield associated with the substitution of pig slurry with the four additional substrates can be seen expressed as a percentage increase in Fig. 4b. An increase in yield of ca. 50% can be achieved through use of 9.1% maize silage. If the substrate is mixed in the ratio 1:10 (substrate to raw pig slurry) the dry matter content of the mixture will not exceed 10% dry matter content. It must be noted that co-digestion tests were not carried out and there may be some potential for co-digestion effects; however given the C:N ratio of slurry this effect will not be significant.

3.6. Comparative analysis

The investigated substrates which are filter pressed manure fibers, chemically precipitated manure fibers, raw pig slurry, maize silage and grass silage were anaerobically digested at 53.5 °C and biogas production was measured and analysed for methane and carbon dioxide content. The cumulative biogas production as a function of time for raw pig slurry, chemically precipitated manure fibers, grass silage and maize silage mainly consisted of three different phases: an initial production at a faster rate followed by a moderate production and then ending up with a plateau phase. Much of the biogas production is in the initial phase showing that a retention time of 20 days is sufficient to generate the majority of the biogas for these substrates. This is not true for the filter pressed manure fiber, suggesting that longer retention time will be required for digestion of this material.

The ultimate methane productivity in terms of VS is found to be higher in the raw pig slurry (ca. 330 L/kg VS) as compared to filter pressed manure fibers (ca. 161 L/kg VS) and chemically precipitated manure fibers (ca. 231 L/kg VS). However the volumetric methane productivity was more than 5 times higher in filter pressed manure (ca. 54.5 L/kg substrate) as compared to raw pig slurry (ca. 10.4 L/kg substrate) and chemically precipitated manure fibers (ca. 12.3 L/kg substrate). On the other hand, ultimate methane production from maize silage is found to be ca. 236 L/kg VS and from grass silage is ca. 361 L/kg VS. The volumetric methane yield of maize silage is found to be higher (ca. 68 L/kg substrate) than the yield from the raw pig slurry and both the solid fiber fractions. Grass silage has also a considerably higher volumetric methane yield (45 L/kg substrate).

The higher volumetric methane yields from maize silage, grass silage and filter pressed manure fibers is due to higher VS content per unit mass of feedstock. Therefore, the use of these materials as co-substrates in biogas plants will increase the volumetric methane productivity as compared to a purely pig slurry facility. Mixing 100 g substrate/1 kg of raw pig slurry will increase the methane productivity by 38% using filter pressed manure fibers, 31% using grass silage and 50% by using maize silage as co-substrates. The biodegradation or conversion of volatile solids to biogas is relatively higher in raw pig slurry (55%) as compared to chemically precipitated manure (41%) and filter pressed manure (35%). The conversion of VS to biogas from maize silage is measured as 54% and 70% for grass silage; these values are in agreement with documented scientific data [25–28]. This indicates that grass silage and maize silage will be easily degraded in a biogas reactor as compared to manure fibers.

4. Conclusions

The methane potential of raw pig slurry, FPMF, CPMF, maize silage and grass silage was 330, 161, 230, 236, 361 L/kg VS respectively. Pre-treatment of manure by separation produces a solid fraction with significantly higher methane potential per unit of volume (55 L/kg FMPM) as compared to raw pig slurry (10 L/kg). The economic cost of such technology may be offset by reduced transportation costs coupled with the potential to increase livestock production by export of surplus nutrients. Effecting higher dry solids content of feedstock either through agricultural practice or separation techniques may lead to lower transportation costs, smaller facilities and lower thermal parasitic demand. Thus, more profitable renewable energy production is achievable at lower production costs. This is exemplified by considering the range of methane production levels from grass silage based on dry solids concentration:

- 45 L/kg at 15% dry solids (in this study);
- 124 L/kg at 40% dry solids (field wilted silage from Austria).

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