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Dry Anaerobic Digestion of Food and Paper Industry Wastes at Different Solid Contents

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Abstract: A large volume of food is being wasted every year, while the pulp and paper industry also generate a large amount of solid wastes on a daily basis, causing environmental challenges around the world. Dry anaerobic digestion (AD) of these solid wastes is a cost-effective method for proper management. However, dry digestion of these waste streams has been restricted due to their complex structure, the presence of possible inhibitors and inappropriate operating conditions. In light of this fact, dry digestion of food waste (FW) and paper wastes (PW) was conducted at different total solid (TS) concentrations of reactor mixtures of 14%, 16%, 18% and 20% TS, corresponding to substrate to inoculum (S/I) ratio of 0.5 and 1; investigating the optimum operating conditions for effective dry digestion of these complex wastes. The highest methane yields of 402 NmlCH₄/gVS and 229 NmlCH₄/gVS were obtained from digestion of FW and PW, respectively at 14%TS corresponding to an S/I ratio of 0.5. Increasing the S/I ratio from 0.5 to 1 and thereby having a TS content of 20% in the reactor mixtures was unfavorable to the digestion of both substrates.

Keywords: dry anaerobic digestion; food waste; paper waste; biogas; digestate

1. Introduction

Organic wastes from municipalities, agricultural and industrial activities degrade over a period of time, having a negative impact on the environment [1]. Landfilling of these wastes is still the most common practice in the world [2], resulting in pollution of water and soil with leachate, and air with emission of methane and other greenhouse gases [3,4]. Anaerobic digestion to produce biomethane has been a successful treatment option for these wastes; it is effective in solving environmental, health and energy challenges [3–5]. Additionally, the digestate residues from biogas plants are rich in macro- and micronutrients and also contain phosphorus and ammonium nitrogen [6], which make them suitable as biofertilizers [7].

Anaerobic digestion (AD) can be divided into wet (Wet-AD) and dry anaerobic digestion (Dry-AD) depending on the total solids (TS) of the bioreactor content. Wet-AD has normally a feedstock TS content below 15% [8], and dry-AD has a feedstock TS content above 20% [9,10]. Wet-AD of wastes has been a common practice, but recently researchers and industries have focused on dry-AD in order to reduce the water content in the reactor and the digestate, thereby making the process more economical. Several countries in the world suffer from water scarcity and the priority of water usage is an important issue [11]. Since wet-AD for biogas production has a high-water content, it demands a large water consumption and also requires treatment of the digestate after biogas production by

processes such as a denitrification process. Consequently, wet-AD could be energy demanding and also result in a loss of nutrition. From that perspective, the dry-AD might be a better technology with lower water consumption and reduced water content in the residue (digestate), resulting in reduced reactor size [12,13]. Additionally, new substrates for biogas production have a growing market. These substrates or wastes from households, restaurants and agriculture contain high TS and consequently could be a suitable feedstock for dry digestion processes rather than for wet digestion. However, dry-AD needs a longer retention time during the startup for microorganisms to adapt to the higher concentration of some toxic compounds which might be present [14]. In addition, the pumping of feedstocks and mixing in the reactor can be more challenging in Dry-AD compared to wet digestion processes [13,15].

Paper wastes (PW) are solid wastes generated from the pulp and paper mill industry (P&P). Recovered fibers used for P&P manufacturing have increased during recent years [16]. Pulp and paper production involve the preparation of feedstock materials, pulping, bleaching, washing/ filtering, screening, and the last step is pulp drying or paper making to get pulp or paper. From these processes, a large amount of wastes, such as wastewater and solid wastes is generated, depending on the manufacturing process and the material used [17]. Dry digestion of these solid wastes fractions is a cost-effective method for proper management. The pulp and paper industry can apply AD processes to treat these solid wastes and to produce energy. However, the recalcitrant structure of PW makes it difficult for the microorganisms to breakdown this substrate and produce biomethane. Consequently the hydrolysis step is the rate-limiting step in the AD process when treating these lignocellulose-rich wastes [5]. In view of this point, there is a need to determine suitable dry AD process conditions for effective biogas production from PW.

On the other hand, a large volume of food is being wasted every year, which is estimated to be about 1.3 billion tons per year [18]. FW is a suitable feedstock for AD processes; it is nutrient-rich and easily biodegradable [19]. However, a major challenge with digestion of FW is obtaining suitable conditions for a stable process. Due to the easy degradability of these wastes, hydrolysis and acidogenesis would normally proceed well and quickly, while the methane production would be the rate-limiting step. Therefore, the AD process needs to be operated with controlled loading rates to avoid the accumulation of intermediary degradation products [20]. Instability in the process occurs often with a high concentration of volatile fatty acids (VFAs), ammonia and/or sulfide [21,22]. Moreover, under a mesophilic condition (37 °C) the process has better stability compared to that under a thermophilic condition (52 °C).

The main objective of this study, was to investigate the conditions for mesophilic dry-AD when using FW and PW as model-substrates. Wet anaerobic digestion (wet-AD) is a well-studied process [23–29], but very few experimental studies have been conducted on dry digestion of FW and PW for biogas production. The TS contents of total mixture in reactors were considered while investigating the dry digestion of these wastes, batch AD assays were performed at different initial TS concentrations as well as different organic loading. Additionally, the digestate residue was characterized to investigate its applicability as a bio-fertilizer.

2. Materials and Methods

2.1. Microorganisms and Substrates

Inoculum used in this study was obtained from a dry digester treating household wastes at Västblekinge Miljö AB (Mörrum, Sweden) and operating at mesophilic conditions. The inoculum was separated from plastics manually and then filtered through a 5-mm porosity sieve to remove other unwanted large particles. Thereafter, the microorganisms were acclimatized at 37 °C prior to use. The inoculum after acclimatization with TS content of 12% was used for the first batch series of the experiment while it was centrifuged at 8000× *g* for 15 min to obtain a TS content of 16% for the second batch assays. This inoculum, used for the digestion assays, as it mentioned above, was a digestate

from a large scale digester and as such contains essential bacteria and archaea for methane formation. The hydrolytic bacteria breaks the organic wastes down into sugars, fatty acids and amino acids, which are then converted into organic acids, alcohols, ammonia, carbon dioxide and hydrogen by the fermentative bacteria. Thereafter, the acetogens convert the obtained products into acetate, carbon dioxide and hydrogen, while the methanogens utilize the acetate or the carbon dioxide together with hydrogen to produce methane.

Two different types of substrates were used: paper wastes (PW) and synthetic food wastes (FW). Paper waste was collected from Södra AB (Varberg, Sweden) with a TS content of 27%. The synthetic food wastes used was prepared according to a previous work [3] with slight modification; i.e., more bread, rice and pasta were added to obtain a TS content of 22%.

2.2. Dry Anaerobic Digestion Assays

Experiments were carried out using either PW or FW as substrates, performed in accordance with the method described previously [30] but without additional water added to be able to provide high solids content within the assays [31]. Figure 1 shows a schematic overview of the experimental setup. The assays were carried out under mesophilic conditions (37 ± 1 °C) using 118 mL serum glass bottles as reactors. For the first experimental setup; substrates with loading of 1.5 g VS were used, and then inoculum was added without centrifugation keeping a VS or S/I ratio ($VS_{\text{substrate}}$ to VS_{inoculum}) at 1:2 and TS content of 14%. Furthermore, setups with VS ratio ($VS_{\text{substrate}}$ to VS_{inoculum}) 1:1 were also performed and thereby indicated a TS content of 16% in the reactors. The second batch series was prepared in the same manner as the previous one mentioned above, except that the inoculum with a higher TS content of 16% was used, thereby having a final TS content of 18% and 20% at VS ratios of 1:2 and 1:1, respectively, in the reactors. Experiments were carried out without any pH adjustment since the inoculum used had a high pH of 8.4. Inoculum and water instead of substrates was used as a blank to disclose any methane production by the inoculum itself. Microcrystalline cellulose of 50 μm particle size (Sigma Aldrich, Darmstadt, Germany) was used as control to check the activity of the inoculum. The reactors were sealed with a rubber septum and aluminum caps, then the headspace was flushed with a gas mixture of 80% N_2 and 20% CO_2 for 2 min, to achieve anaerobic conditions in all reactors. The reactors were then incubated at 37 ± 1 °C and they were shaken manually periodically during the incubation period of 112 days. All experimental setups were done in triplicates. Gas samples were taken every second and third day at the beginning and once a week towards the end of the digestion period from the headspace of each reactor using a 250- μL pressure-lock gas syringe (VICI, Precision Sampling Inc., Baton Rouge, LA, USA). Samples were analyzed using a gas chromatography (GC). Gas measurement and analysis were carried out as described previously [32] and all methane volumes are presented at standard conditions (0 °C and 1 atm).

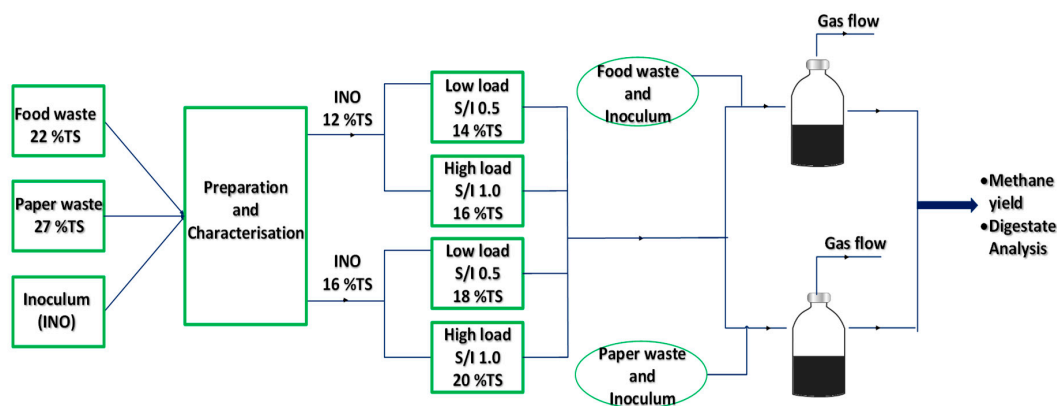


Figure 1. Schematic diagram showing the experimental set up for the batch digestion assays, resulting in different total solid (TS) concentrations of the mixture in reactors at a substrate to inoculum ratio (S/I) of 0.5 and 1.0.

2.3. Analytical Methods

Standard methods for water and wastewater [33] were used to determine the total solid, volatile solid, moisture content and pH for the substrates and the inoculum. The Kjeldahl method was used to analyze the concentration of the total nitrogen contents [34]. The total carbon was obtained by correcting the total dry weight carbon value for the ash content [22,35]. The compositions (methane, hydrogen and carbon dioxide) of the produced gas was determined using Gas chromatography or GC (PerkinElmer Clarus 590, PerkinElmer Inc., Waltham, MA, USA) equipped with a packed column (6' × 1.8" OD, 80/100, Mesh, PerkinElmer Inc., Waltham, MA, USA), and a thermal conductivity detector (PerkinElmer Inc., Waltham, MA, USA), with an inject temperature of 150 °C. The carrier gas was nitrogen operated with a flow rate of 20 mL/min at 60 °C. The European standard method for dry digestion sludge was used to determine the content of macro- and micro-nutrients, as well as trace elements in the digestate residue [36].

2.4. Digestate Analysis

An amount of 2 g of the digestate residue was diluted with milli-Q water to 10 mL. Dissolved samples were centrifuged at 3000× *g* for 10 min and the liquid fraction was centrifuged at 20,000× *g* for 10 min. Subsequently, the liquid fraction was passed through 0.2 μm filter prior to analysis. Samples were analyzed for ammonium nitrogen concentration using Ammonium 100 test kit (NANOCOLOR®, MACHEREY-NAGEL GmbH & Co. KG., Düren, Germany) and the concentration was measured using NANOCOLOR® 500 D Photometer (MACHEREY-NAGEL GmbH & Co. KG., Düren, Germany). Thereafter, the liquid fraction of the digestate was analyzed for individual and total VFA concentrations using HPLC (Waters 2695, Waters Corporation, Milford, MA, USA), which was equipped with an ion-exchange column (Aminex HPX-87H Bio-Rad Laboratories Inc., Hercules, CA, USA) and a Waters 2414 UV detector. The column temperature was set at 60 °C and 5 mM sulfuric acid was used as eluent with a flow rate of 0.6 mL/min.

2.5. Statistical Analysis

Anaerobic digestion experiments were designed in triplicates and standard deviations were analyzed for the set of experimental runs. The experimental data was analyzed using general linear model analysis of variance (ANOVA) with accumulated methane yield as the response variable. All error bars reported represent 95% confidence intervals.

3. Results and Discussion

3.1. Characterization of Synthetic Food Waste, Paper Waste and Inoculum

The most important characteristics of the inoculum and the substrates (food wastes and paper wastes) are shown in Table 1. The FW used contained 21.9%TS, while the PW contained 27.3%TS of which 96.8% and 99.5% were organic matter, respectively. The carbon to nitrogen (C/N) ratio for synthetic food waste was 18.67, which is in the suitable range to keep the anaerobic digestion in a stable condition. Optimal C/N ratios between 20:1 and 35:1 are normally mentioned [37] and even more wide-spread C/N ratios between 10:1 and 30:1 have been reported [37,38]. However, the paper wastes contain very high carbon content, resulting in a C/N ratio of 807, although this is non-easily degradable carbon.

The characterization of the inoculum with the macro- and micro/trace elements concentration is presented in Table 2. The C/N ratio of the inoculum was almost the same irrespective of the TS content. Inoculum with TS of 15.9% and 11.9% had a C/N ratio of 14.22 ± 1.34 and 12.80 ± 1.07 respectively. The C/N ratio was still within the acceptable range for a stable anaerobic digestion process [37,38].

Table 1. Characteristics of Substrate and Inoculum used for the dry AD experimental setup.

Parameters	Food Waste	Paper Waste	Inoculum, High TS	Inoculum, Low TS
Total solids (%)	21.90 ± 0.15	27.33 ± 0.05	15.91 ± 2.77	11.91 ± 0.26
Volatile solids (% TS) ^a	96.76 ± 0.10	99.46 ± 0.03	77.10 ± 2.35	69.42 ± 1.62
Moisture (%)	78.10 ± 0.15	72.66 ± 0.15	84.09 ± 2.77	88.09 ± 0.26
Ash (%) ^a	3.24 ± 0.10	0.54 ± 0.03	22.39 ± 2.35	30.58 ± 1.62
Total organic carbon (% TS) ^a	53.76 ± 0.06	55.26 ± 0.02	42.84 ± 1.30	38.57 ± 0.90
Kjeldahl Nitrogen (% TS) ^a	2.929 ± 0.38	0.069 ± 0.01	3.02 ± 0.11	3.02 ± 0.11
C/N ratio	18.67 ± 3.45	807 ± 25.46	14.22 ± 1.34	12.80 ± 1.07
Bulk density (kg/ m ³)	1090.4 ± 15.99	432.9 ± 6.35	1016.8 ± 17.65	1016.8 ± 17.65
Protein content (%) ^a	18.31 ± 0.38	0.43 ± 0.01	18.88 ± 0.14	18.88 ± 0.14
pH	5.34 ± 000	ND	8.42 ± 000	8.42 ± 000

ND = not determined; C/ N = carbon to nitrogen. ^a Dry basis.

The inoculum had a high concentration of calcium (Ca), an amount of 52.7 g/kg dry matter (DM), which is suitable to increase the buffer capacity of the digestion process [39], while potassium, magnesium and phosphorus are also present which are the major macronutrients needed. Additionally, it contained iron, copper, manganese and zinc which are important trace elements in anaerobic digestion processes [40]. The inoculum is also of high quality with very low concentrations of cadmium, cobalt, chromium, mercury, nickel and lead; and their concentrations did not exceed the quality standards [39,40].

Table 2. Macro-, and Micro/Trace Elements of the inoculum used in this work, which was a digestate of a dry-AD of municipal sorted food wastes.

Macro Elements	g/kg of Dry Matter	g/kg of Fresh Mass
Calcium	52.7	6.11
Potassium	19.2	2.23
Magnesium	2.55	0.30
Sodium	15.0	1.74
Phosphorus	10.8	1.25
Sulphur	5.33	0.62
Micro/Trace Elements	mg/kg of Dry Matter	mg/kg of Fresh Mass
Aluminum	1700	197
Arsenic	* < 15.0	* < 1.74
Boron	20.0	2.32
Barium	49.2	5.71
Cadmium	0.28	0.03
Cobalt	5.31	0.62
Chromium	5.95	0.69
Copper	30.2	3.50
Iron	4500	522
Mercury	* < 4.20	* < 0.49
Lithium	1.47	0.17
Manganese	201	23.3
Molybdenum	3.26	0.38
Niobium	* < 0.30	* < 0.03
Nickel	6.59	0.76
Lead	2.96	0.34
Selenium	* < 15.0	* < 1.74
Silicon	486	56.4
Tin	2.34	0.27
Strontium	142	16.5
Vanadium	2.27	0.26
Zinc	95.1	11.0

* Values marked with ‘ < ’ represent the quantification limit of the particular element. Macro-, micro/trace elements for dry digestion have a dry matter of 116 mg/ kg.

3.2. Dry Anaerobic Digestion of Food Waste

The cumulative methane yield from food waste at different TS contents during 112 days of anaerobic digestion is shown in Figure 2. The digestion process took a longer retention time due to

probably low mass transfer at higher solid content [8]. The reactor with mixture content of 14%TS (S/I ratio of 0.5) gave the highest methane yield of 402 NmlCH₄/gVS, which is 31% higher than the yield obtained at 18%TS with the same substrate to inoculum ratio. According to previous investigations, an increase in the TS content in the reactor mixture resulted in a reduction in methane yield [20]. This might indicate that the microorganisms need higher moisture for the methane production in the reactor [29].

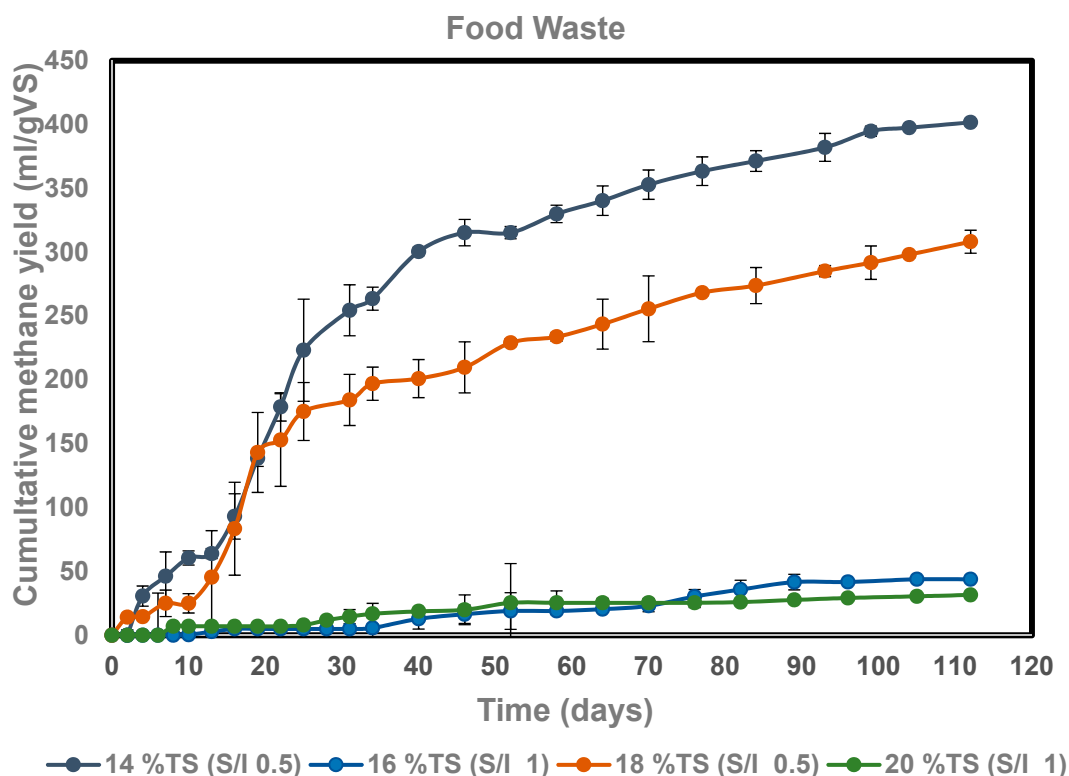


Figure 2. Cumulative methane yield of food wastes at different total solid (TS) concentrations of the mixture in reactors at substrate to inoculum ratios (S/I) of 0.5 and 1.

Increasing the S/I ratio from 0.5 to 1, thereby having a TS content of 16% and 20% in the reactors, was unfavorable to the production of methane. There was a fast hydrolysis of the organic fraction, which resulted into a process failure due to acidification as shown in Table 3. The methane yield was very low due to overloading and accumulation of volatile fatty acids (VFAs value of 7.5 g/L and 10.4 g/L), resulting in a decreased pH from 6.9 to 5.9. The microbial community structure has an important role regarding the tolerance to lower pH [41]. It was previously reported that the ratio between hydrogenotrophs and acetotrophs is particularly critical, where the hydrogenotrophs are more tolerant to inhibition [41,42]. However, the microbial community was not an area of focus in this work, but it would be interesting to study under such conditions. Effective biogas production has been reported in some cases with high S/I ratios (>1.77 VS/ VS) [19,41]. Though, most of the studies point out the requirement of lower S/I ratios (<0.5) to avoid inhibition [41,43,44].

Kawai et. al. [43] reported a drastic reduction in the methane yield from 425 to 257 mL/gVS as the S/I ratio was increased from 0.33 to 1 during wet digestion of food wastes.

The results obtained here in this study showed that the effect of S/I ratio is much higher than the effect of TS concentration during dry digestion of food wastes. The methane yield at 18% TS with a corresponding S/I ratio of 0.5 was 308 mL/gVS, which is 604% higher than the yield obtained at 16%TS with corresponding S/I ratio of 1. There was process instability as the total TS content in reactors were increased to 16% TS and 20% TS with a corresponding S/I ratio of 1. Accumulation of volatile fatty

acids (VFA) was observed with reduction in the pH value (Table 3), leading to digestion process failure as shown in Figure 2.

3.3. Dry Anaerobic Digestion of Paper Waste

Cumulative methane yields from paper wastes at different TS concentration of reactor mixtures corresponding to S/I ratio of 0.5 and 1 are presented in Figure 3. A methane yield of 229 NmlCH₄/gVS was obtained at 14% TS, which is 29% higher than the yield obtained at 18%TS with the same S/I ratio of 0.5. However, the drastic reduction in the methane yield was observed as the S/I ratio increased from 0.5 to 1; i.e., the methane yield reduced by 123% and by 78% at 16%TS at 20%TS, respectively. Consequently, the same trend was observed as for the digestion of food wastes with increased S/I ratio. This shows the significance of the S/I ratio as important parameter to be considered when performing dry digestion of food wastes and paper wastes. Rodriguez et al. [5] previously reported a reduction in the methane yield of untreated paper wastes from 132 mL/gVS to 107 mL/gVS as the S/I ratio increased from 0.5 to 0.7 under wet digestion process. Previous studies have shown methane yields between 40 and 200 mL/gVS [45] for wet digestion of untreated paper wastes. A methane yield of 229 NmlCH₄/gVS was obtained in this study at 14%TS of reactor mixture, which is slightly higher than previous obtained yield under wet digestion processes.

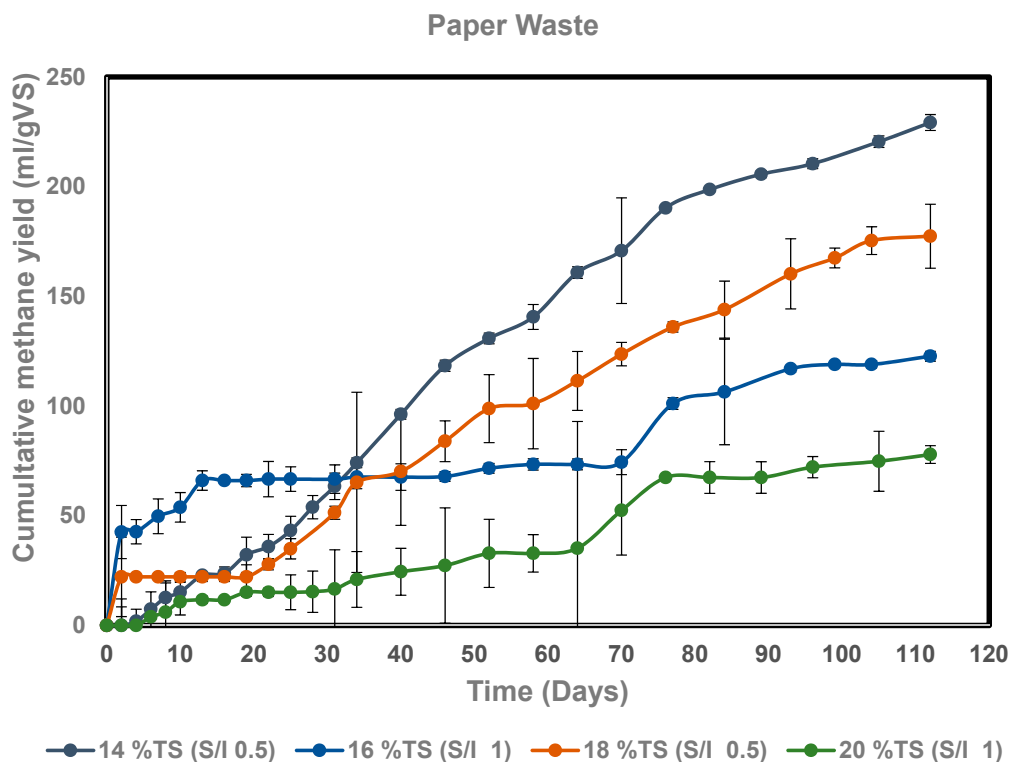


Figure 3. Cumulative methane yield of paper wastes at different total solid (TS) concentrations of the mixture in reactors at inoculum to substrate ratios (I/S) of 2:1 and 1:1.

During the digestion of PW, the methane production rate was low initially (during the first 30 days of digestion), then it increased gradually. This is probably because of the challenges in the hydrolysis of hemicellulose and cellulose and as such this process takes a longer time [5,14]. Therefore, a gradual acclimatization of the microorganisms to the substrate and condition in the reactor is needed to optimize the biogas production [5,14]. Moreover, PW has a low content of nitrogen (N), which results in a very high C/N ratio of 807. Therefore, the buffering capacity was low and as such the microorganisms needed an adaptation period to develop a diverse anaerobic microflora for a good anaerobic digestion process [46].

3.4. Digestate Quality

The digestate residue from dry anaerobic digestion of FW and PW was analyzed for its suitability as a bio-fertilizer, and is presented in Table 3.

Table 3. Characterization of the digestate residue from dry anaerobic digestion.

14%TS (S/I 0.5)	pH	VFA (g/l)	NH ₄ - N (mg/l)
Food waste	8.14 ± 0.03	1.03 ± 0.93	4200.00 ± 283.00
Paper waste	7.74 ± 0.43	0.38 ± 0.13	3250.00 ± 100.00
18%TS (S/I 0.5)			
Food waste	8.11 ± 0.08	0	540.00 ± 30.00
Paper waste	8.11 ± 0.07	0	363.33 ± 15.28
16%TS (S/I 1)			
Food waste	6.88 ± 1.19	7.48 ± 4.52	4600 ± 566.00
Paper waste	7.27 ± 1.27	0.29 ± 0.04	2900.00 ± 100.00
20%TS (S/I 1)			
Food waste	5.92 ± 0.07	10.42 ± 4.16	6700 ± 892
Paper waste	6.36 ± 1.60	0.52 ± 0.19	3400 ± 100

The pH of the residue after digestion of FW at 14% TS and 18%TS (S/I ratio of 0.5) was alkaline which is suitable for improving the buffering capacity of soil especially when the agriculture soil is more acidic [39]. It also contained a high concentration of ammonium nitrogen (NH₄- N) which can easily be taken up by the plants [39]. However, as the S/I ratio increased to 1, the digestate became slightly acidic due to the accumulation of VFAs and as such was not suitable as a bio-fertilizer. On the other hand, the residue after digestion of PW at all total solid concentration irrespective of the S/I ratio applied was found suitable for bio-fertilizers showing a high concentration of ammonium nitrogen (NH₄- N) without VFAs accumulation.

4. Conclusions

The batch dry digestion of FW and PW were carried out by successfully applying initial TS concentrations of 14%, 16%, 18% and 20% in the reactor mixtures. Comparing the dry digestion of FW with PW, FW performed better than PW, achieving 31% and 29% higher methane yield at 14%TS and 18%TS respectively; PW contains lignocellulose, where the hydrolysis is the rate-limiting step. However, increasing the S/I ratio from 0.5 to 1 had a more drastic effect on dry digestion of FW than on that of PW. Dry digestion of FW at higher organic load resulted in accumulation of VFAs. Therefore, a lower substrate to inoculum ratio of 0.5 is highly recommended for stable and effective dry digestion of food wastes and paper wastes.

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