Effects of volumetric dilution on anaerobic digestion of food waste

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Despite the increasing number of small scale digesters operating, there remains a lack of information with regards to performance optimization from an everyday user's standpoint. The objective of this study was to determine the effects of volumetric dilution and food waste composition on digester performance. Batch experiments utilizing food waste majoring in carbohydrate, protein, lipid, and cellulose, subjected to five concentrations of volumetric dilution (3.7%–17.1% total solids (TS)), were conducted. Irregardless of volumetric dilution, all assays achieved substrate degradation higher than 82.5% and did not suffer methanogenic inhibition, when provided with retention times comparable to those used in small scale digesters. Protein rich and cellulose rich waste achieved the highest methane potential varying between 0.410–0.539 m^3/kg volatile solids (VS) and 0.450–0.535 m^3/kg VS, respectively. Protein rich assays were also observed to be the first to achieve 50% of its Bo irregardless of concentrations, followed by carbohydrates and cellulose, and lipids having a considerably longer methanation time. Results saw an increase in total methane generated but a decrease in specific yield as % total solid increased. To successfully digest lipid rich waste a dilution no lesser than 1:4 was required. © *2012 American Institute of Physics*. [http://dx.doi.org/10.1063/1.4764935]

I. INTRODUCTION

Currently, there are over 30 million small scale anaerobic digesters around the world, most of which are located in developing nations such as China and India. The adaptation and expansion of low cost small scale digesters in developing countries have had enormous benefits. The benefits of such micro scale digesters have been discussed by many (van Groenendaal and Wang[1] and Bond and Templeton[2]). Unfortunately, despite the large number of existing systems, there has been a paucity of quantitative research regarding low cost digester's performance (Lansing *et al.*[3] and Linderoth[4]). In addition, most available information with regards to small scale digesters pertain to manure based digesters and not food waste digesters. As the interest of food waste digesters increases, there is a pressing need to further our knowledge with regards to the operation of small scale food waste digesters (Battistoni *et al.*[5] and Chanakya *et al.*[6]).

An area of uncertainty with regards to the operation of food waste digester is knowing the appropriate water addition in order to achieve optimal digester performance. The amount of water added to a digester can directly affect the degradation efficiency and biogas generation. Adding too little water can lead to overfeeding and/or accumulation of inhibiting substances such as fatty acids (Linke[7]) which is a common cause of digester failure (RISE-AT[8]). Excessive water may also lead to flush out and the need for a large digester tank. Therefore, it is crucial to determine the influence of influent concentrations (determined through water addition) on digester performance.

The effects of organic loading rates (OLR) and percentage total solids (%TS) on methane (CH_4) generation have been studied using substrates such as the co-digestion of organic fraction of municipal solid waste (OFMSW) and return activated sludge (Fongsatitkul *et al.*[9]),

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co-digestion of MSW and domestic sewage, (Elango *et al.*[10]), co-digestion of fruit and vegetable waste and primary sludge (Gómez *et al.*[11]), fruit canning effluent (Trnovec and Britz[12]), olive mill solid residues (Rincon *et al.*[13]), piggery waste (Sanchez *et al.*[14]), and municipal solid waste (Igoni *et al.*[15] and Rao and Singh[16]). All of these studies adjusted their organic loading rate by either manually altering the %TS of the influent (Fongsatikul *et al.*[9] and Rincon *et al.*[13]), shortening the hydraulic retention times (HRT), or increasing the rate of feeding (Sanchez *et al.*[17]). These parameter adjustments can be performed easily in centralized and industrialized plants where specialized operators with advanced training, knowledge, and monitoring devices are used to obtain the highest digestion output.

However, unlike operations in such plants, small scale digesters are operated by the local people, who operate the digester based on given general operational guidelines. Consequently, it is vital to be able to predict the performance of digesters based on user-friendly parameters. In this study, the effects of dilution will be determined by varying the volumetric water to waste ratio, which can be easily measured and operated by the everyday user (Eze *et al.*[18]). The influence of water addition on biochemical CH_4 potential (BMP) alongside with semi-batch experiment will be investigated in this study.

II. MATERIALS AND METHOD

A. Waste characteristics and preparation and inoculent

Carbohydrate, protein, lipid, and cellulose rich mixed food wastes were prepared by mixing food groups representative of, carbohydrate, protein, lipid, and cellulose in the ratio 2:1:1:1, 1:2:1:1, 1:1:2:1, and 1:1:1:2, respectively. A mixture of potatoes, bread, rice, and pasta was used to represent carbohydrates; chicken, beef, and pork was used to represent proteins; vegetable oil and animal fat to represent lipids; and a mixture of carrots, spinach, and lettuce was used to represent cellulose. Nutrient data for each of these majoring food groups are detailed in Table I. For BMP test, each majoring macronutrient group was then subjected to five dilutions with a volumetric waste to water ratio of 1:2, 1:3, 1:4, 1:5, and 1:6.

Each feedstock coupled with its assigned dilution was then blended using an electric blender and stored in a cool room at $-1\,^{\circ}C$ until use. Each waste component was removed from the freezer to thaw overnight prior to use.

Anaerobic sludge collected from the Woodman Point Wastewater Treatment Plant (mesophilic) anaerobic digester in Perth, Western Australia was used as inoculent for all experiments. Inoculum was tested for active methanogens prior to use.

B. Experimental setup

100 ml serum bottles were used for the batch experiments, which were washed and soaked in 10% hydrochloric acid solution overnight and washed thoroughly with distilled water prior to use. With a working volume of 50 ml in each reactor, 40 ml of inoculum, 10 ml of the assigned feedstock (waste), and 120 mM of bicarbonate, to ensure an optimal *p*H, was added. Each serum bottle was purged with a mixture of 90% N_2 gas and 10% H_2 gas for 30 s before being sealed with a rubber septum seal and aluminium crimps to ensure an anaerobic condition.

TABLE I. Food composition (%) of majoring macronutrient groups used in experiments.[a]

Majoring macronutrient group	Moisture	Carbohydrates	Protein	Fats	Fibers
Carbohydrate rich FW	61.26	16.36	6.56	15.11	1.44
Protein rich FW	65.05	6.05	12.38	16.07	0.71
Lipid rich FW	44.38	6.05	6.79	42.34	0.71
Cellulose rich FW	70.85	7.85	5.77	14.81	1.40

[a]Values obtained by working out the average nutritional value from USDA[19] of carbohydrate, protein, lipid, and cellulose waste and the composition are listed.

Each test was performed in duplicate. In each experiment, blank reactors with 40 ml inoculent, 10 ml tap water, and 120 mM bicarbonate were also prepared to serve as the control. Serum bottles were placed in a 38 °C water bath. All reactors were depressurized to atmospheric pressure after the first hour of incubation.

C. Experimental procedures

All assays were tested for gas production and gas composition at regular intervals. Sampling was performed more frequently at the start of the experiments so as to avoid pressurization, and less frequently as cumulative gas production started to plateau. Testing was done while assays were still submerged in their respective water bath. Following gas testing, each reactor was swirled gently to mix the substrate and microbes. Gas testing was performed until all significant CH_4 production ceased.

D. Gas analysis

The biogas accumulated in the headspace of the serum bottles was sampled regularly and the CH_4 carbon dioxide (CO_2) concentrations were determined. Biogas composition was analysed for CH_4 and carbon dioxide percentage using a Varian Star 3400 gas chromatograph (GC) equipped with a thermal conductivity detector. The volume of gas produced was determined by displacement using a glass syringe. CH_4 production for each measurement was calculated using both the volume displacement and the percentage of CH_4 for any current reading and its previous reading as seen in the following equation:

$$CH_{4;t} = \left[\frac{(vol\,displaced + headspace) \cdot \%CH_{4;t}}{100}\right] - \left[\frac{headspace \cdot \%CH_{4;t-1}}{100}\right]. \quad (1)$$

E. Chemical analysis

Total and volatile solids (VS) were analysed through difference in mass at ambient temperature, after 105 °C heating and after 550 °C heating using a muffle oven. Effluent collected were filtered and centrifuged to obtain the supernatant for testing of volatile fatty acids (VFA) (g acetic acid/l) and soluble chemical oxygen demand (sCOD). VFA was determined using the esterification method using a spectrophotometer (HACH 2008) and sCOD was performed using a close reflux and spectrophotometer method (Jirka and Carter[20]). *p*H was measured using a microcomputer *p*H meter.

III. RESULTS AND DISCUSSION

A. Relationship between volumetric dilution and %TS

Characteristics of influent wastes are described in Table II with %TS tested varying between 3.70% and 17.11% depending on the dilution factor and the macronutrient group. Cellulose obtained the lowest %TS, and hence lowest load, when subjected to the same dilution factor, followed by protein, carbohydrate, then lipids.

As illustrated in Table II, *p*H of food waste is relatively acidic in nature. While different food waste differs in *p*H, when fresh, upon slight decomposition, *p*H falls, most likely exacerbated by the production of acid from acidogens. Hence, the addition of sufficient bicarbonate, such as addition of 120 mM of bicarbonate in these assays, would be required. Alternately, load conditions have to be regulated carefully and sufficient dilutions need to be enforced to ensure a healthy microbial population. The latter practice is more common with micro-scale digesters operated in the fields, i.e., *p*H regulation is performed by load control and not by chemical additions (Arias *et al.*[21] and Ding *et al.*[22]).

A power relationship between volumetric waste to water ratio and %TS can also be identified for all four macronutrient groups. The relationships for carbohydrate, protein, lipid, and cellulose rich waste can be described as follows, respectively ($R^2 > 0.99$):

TABLE II. Characteristics of influent waste tested for BMP analysis.

Majoring macronutrient group	Dilution factor waste: water	*p*H -	TS (g/l)	VS (g/l)	TS (%)	sCOD (g/l)	VFA (mg/l)
Majoring in carbohydrates	1:2	4.28	115.6 ± 6.4	114.3 ± 6.5	11.2 ± 0.00	76.5 ± 0.01	2224 ± 66
	1:3		90.9 ± 6.5	89.9 ± 6.3	8.2 ± 0.06	61.9 ± 0.11	1665 ± 71
	1:4		70.6 ± 1.8	70.0 ± 1.8	6.6 ± 0.01	51.6 ± 0.06	1235 ± 94
	1:5		57.9 ± 3.3	57.3 ± 3.3	5.5 ± 0.02	43.4 ± 0.01	955 ± 43
	1:6		51.4 ± 1.2	50.7 ± 1.3	4.8 ± 0.03	36.8 ± 0.18	720 ± 32
Majoring in proteins	1:2	3.96	112.2 ± 1.8	109.2 ± 1.0	10.9 ± 0.08	63.1 ± 0.02	1825 ± 111
	1:3		81.6 ± 0.8	80.2 ± 0.6	7.9 ± 0.01	47.6 ± 0.36	1173 ± 94
	1:4		68.5 ± 0.9	67.4 ± 1.0	6.5 ± 0.04	35.5 ± 0.09	750 ± 47
	1:5		56.2 ± 1.2	55.6 ± 1.4	5.3 ± 0.07	28.9 ± 0.03	626 ± 55
	1:6		46.4 ± 2.2	45.8 ± 2.2	4.5 ± 0.04	23.2 ± 0.03	528 ± 29
Majoring in lipids	1:2	3.78	182.6 ± 8.0	181.2 ± 8.0	17.1 ± 0.47	60.2 ± 0.02	1950 ± 69
	1:3		123.4 ± 1.8	122.6 ± 2.4	12.4 ± 0.17	45.0 ± 0.15	1118 ± 87
	1:4		103.9 ± 0.5	103.0 ± 0.6	10.2 ± 0.05	34.0 ± 0.04	800 ± 24
	1:5		88.6 ± 1.4	87.9 ± 1.7	8.4 ± 0.02	26.1 ± 0.38	622 ± 8
	1:6		76.9 ± 4.3	76.0 ± 4.2	7.3 ± 0.07	19.5 ± 0.17	510 ± 31
Majoring in cellulose	1:2	3.94	90.1 ± 1.9	88.0 ± 2.0	8.5 ± 0.14	53.1 ± 0.00	980 ± 74
	1:3		66.8 ± 2.6	65.3 ± 2.5	6.5 ± 0.01	37.2 ± 0.12	746 ± 35
	1:4		54.4 ± 3.0	53.1 ± 2.9	5.2 ± 0.04	21.6 ± 0.28	605 ± 51
	1:5		42.7 ± 1.7	44.6 ± 1.2	4.3 ± 0.25	19.8 ± 0.06	534 ± 23
	1:6		38.8 ± 0.6	38.0 ± 0.6	3.7 ± 0.08	17.2 ± 0.33	480 ± 12
Inoculent	…	7.37	40.9 ± 0.6	30.4 ± 0.7	4.2 ± 0.04	3.7 ± 0.09	86 ± 7.4

$$\%\,TS = 19.04 \cdot \left(\frac{Waste}{Water}\right)^{0.77}, \tag{2}$$

$$\%\,TS = 19.13 \cdot \left(\frac{Waste}{Water}\right)^{0.80}, \tag{3}$$

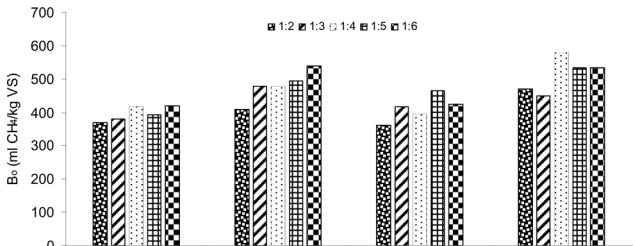
$$\%\,TS = 29.06 \cdot \left(\frac{Waste}{Water}\right)^{0.77}, \tag{4}$$

$$\%\,TS = 14.84 \cdot \left(\frac{Waste}{Water}\right)^{0.77}. \tag{5}$$

B. Maximum CH_4 generation potential (Bo)

Maximum CH_4 yield for all assays is listed in Figure 1 and cumulative CH_4 yields plotted against time, corrected for their inoculums' CH_4 yield, for all assays are shown in Figure 2.

In general, irregardless of the majoring macronutrient present, assays with a higher dilution factor obtained a higher Bo varying between 0.363 and 0.579 m^3 CH_4/kg VS (Figure 1). Protein rich and cellulose rich achieved the highest Bo interchangeably between the dilution concentrations, with increasing disparity as the %TS decreased (Figure 1). Successful CH_4 conversion of protein rich waste at 1:2 dilution suggests, given sufficient HRT, solid concentration up to 10.92%TS will not lead to sulphide inhibition of the methanogens or ammonia inhibition of VFA consuming methanogens (Table III), which is a common concern with regards to the digestion of protein rich substrate (Ek *et al.*[23] and Chen *et al.*[24]).



	Carbohydrate	Protein	Lipid	Cellulose
1:2	369.98	409.91	362.72	468.92
1:3	380.76	477.66	418.07	449.93
1:4	417.81	477.48	397.04	579.3
1:5	392.41	494.41	463.79	532.74
1:6	419.16	538.39	425.18	534.79

FIG. 1. Bo achieved for carbohydrate, protein, lipid, and cellulose rich food waste subjected to five volumetric dilutions.

Successful conversion of lipid rich and carbohydrate rich waste at high concentrations (11.18%TS and 17.11% TS, respectively) also suggest that with sufficient HRT, both substrates are capable of bio-methanization without significant inhibition from hydrogen and long chain fatty acids (LCFA), respectively (Table IV). Neves *et al.*,[25] also studying mixed food waste stream of majoring macronutrients, reported a lower range of Bo, ranging from 0.36 to 0.43 m^3/kg VS. In contrary to current findings, Neves *et al.*[25] reported waste streams with an excess

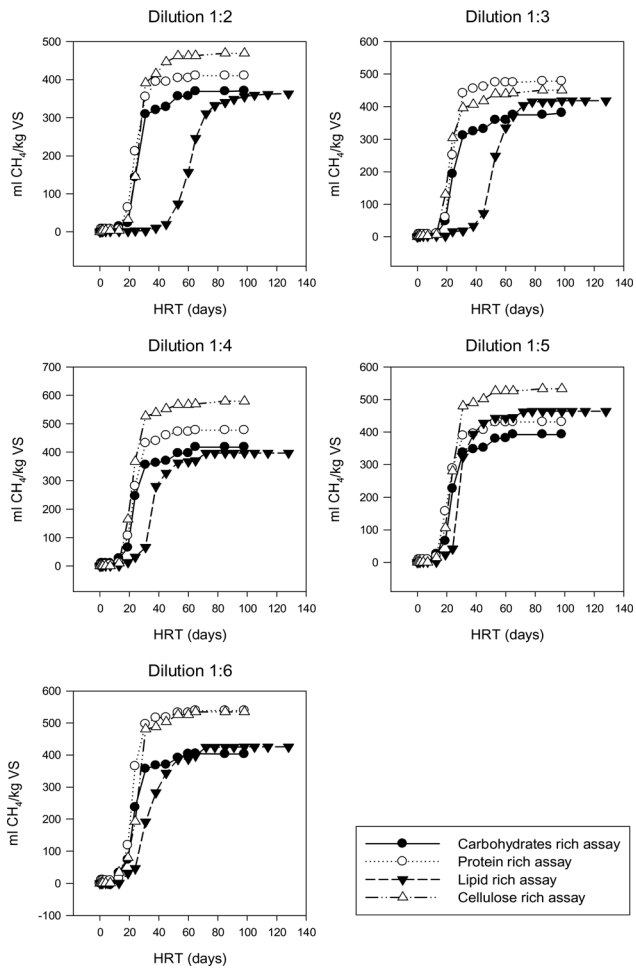


FIG. 2. Cumulative CH_4 generation profile for carbohydrate, protein, lipid, and cellulose rich waste under five volumetric dilutions.

TABLE III. VFA degradation efficiency (%) of assays under mesophilic and ambient conditions.

Majoring FW	1:2	1:3	1:4	1:5	1:6
Carbohydrate	97.9–98.0	96.8–97.2	96.3–96.8	95.6–97.0	94.6–95.3
Protein	97.8–98.0	96.0–97.0	95.9–95.9	94.7–959	93.6–94.5
Lipid	96.1–96.9	94.6–96.2	90.5–91.9	91.3–91.4	91.6–92.2
Cellulose	96.0–96.9	94.2–95.3	89.4–92.4	90.3–93.3	87.1–90.0

in lipids to have the highest CH_4 yield and waste streams in excess of carbohydrates or cellulose to have the lowest CH_4 yield. Studies by Cho and Park[26] found a wider range of Bo from 0.294 to 0.482 m^3/kg VS for waste streams varying in macronutrients. The current study agreed with the findings from Cho and Park[26] in that carbohydrates had the lowest Bo but protein was found to have the highest achievable Bo. Table IV shows the comparison of this study with previous studies. It should be considered that the cellulose referred here comprises of low lignin content cellulose such as vegetable and fruit waste, as opposed to high lignin green waste such as leaves and bark. The associated %TS yielded from these results was 1.8%TS for Neves *et al.*[25] and 3.0%TS for Cho and Park.[26] Although Bo values are within range of this study, the %TS tested are much lower than that tested.

C. Lag phases

Lag phase for carbohydrate, protein, and cellulose rich waste varied between 14.9 and 19.9 days and between 20.2 and 48.7 days for lipid rich waste. Lag phase decreased significantly for lipid rich waste, and slightly for carbohydrate rich waste, but no obvious differences were noticeable for protein and cellulose rich waste. The large differences in lag time between lipid rich waste and the other assays are most prominent under lower dilution between 1:2 and 1:3 (Figure 2).

The effects of lipid inhibition only become less noticeable with dilutions exceeding 1:3. This is most probably due to the initial inhibition by lipids (Hanaki *et al.*[27] and Kuo and Chen[28]). The rapid breakdown and release of LCFA caused a bottleneck effect, resulting in CH_4 being produced at a later time. It can be seen that the lag time for CH_4 generation for lipid rich assay decreased with increased dilution together with the increase in CH_4 yield as the inhibition effects are diminished. The effects of lipid inhibition became negligible after 1:5 dilution. Further dilutions beyond 1:6 would need to be tested to determine the %TS needed for lipid's lag phase to be equal to the other macronutrients. Irregardless of the initial lag phase, all substrate degraded effectively with 87.08%–98.04% degradation of VFA.

D. Rate of methanogenesis

The number of days for each substrate to achieve 50% of total CH_4 produced is summarized in Table V. With few exceptions, protein rich assays were the first to achieve 50% of its Bo irregardless of concentrations, followed by carbohydrates and cellulose, and lipids having a considerably longer methanation time. This coincides with results from Neves *et al.*[25] who

TABLE IV. Comparative B_o of carbohydrate, protein, lipid and cellulose rich food waste from similar studies (ml CH_4/kg VS).

	Neves *et al.*[25]	Cho and Park[26]	This study
Carbohydrates rich	370	294	370–419
Protein rich	390	482	410–538
Lipids rich	430	…	355–464
Cellulose rich	360	356	500–579

TABLE V. Number of days required to achieve 50% maximum CH_4 potential.

	Dilution factor				
	1:2	1:3	1:4	1:5	1:6
Carbohydrate rich	26.6	24.7	23.3	22.9	23.6
Protein rich	25.4	20.8	20.9	21.1	22.3
Lipid rich	61.3	50.1	35.9	29.5	32.7
Cellulose rich	26.9	21.9	22	23.6	25.7

reported, assays with an excess in protein were the first to achieve 85% methanization after 23 days, followed by cellulose (24 days), carbohydrate (30 days), and lipids (57 days). Generally, as the dilution factor increased, the time required to achieve 50% Bo decreased. While differences were slight for carbohydrate, protein, and cellulose rich wastes, lipid rich waste saw a substantial drop, approximately 41% when dilution increased from 1:2 to 1:4. This suggests that with regards of anaerobic digestion of food waste, lipids should be the main food waste component of concern that may affect digester's efficiency. The long lag phase and time required by lipid rich waste stream to achieve 50% of methanogenesis also suggest either an implementation of a two stage digester to improve the efficiency or a single larger digester as compared with other macronutrient in order to accommodate the longer retention time needed.

E. Total CH_4 generated and CH_4/kg VS

Results showed a general increase in total CH_4 production with increases in %TS for all substrate (Figure 3). The increasing CH_4 yield can be simply attributed to the higher concentration of TS available for bioconversion. Similar results were observed by Ignoi *et al.*[15] and Ignoi *et al.*[29] who studied the effects of total solids concentration of MSW, from 4% to 10%, on biogas produced in an anaerobic continuous digester under mesophilic conditions. Despite the increased total CH_4 yield with increased %TS, specific CH_4 production (SMP) increased as %TS decreased (Figure 3). This may be because the lower moisture content in waste with higher %TS resulted in a reduced level of microbial activity such as methanogenesis (Igoni *et al.*[15]).

This result is supported by studies by Fongsatikul *et al.*,[9] Igoni *et al.*,[29] and Igoni *et al.*[15] who suggested inhibition or overloading as possible causes to the low CH_4 yield experienced by substrates of high %TS. Fongsatikul *et al.*[9] observed a 26.0% increase in specific gas production when %TS decreased from 15% to 8% (from 0.54 to 0.73 m^3/kg VS for the OFMSW), while Lansing *et al.*[30] reported a reduced SMP with in increasing %TS/VS – 0.31, 0.18, and 0.12 m^3/kg VS/day for 2.5%, 5%, and 10% TS, respectively) for three field pilot plants co-digesting a mixture of swine manure and cooking grease. Previous studies by Itodo and Awulu[31] showed that substrates of higher %TS were more prone to acidic conditions, while Igoni *et al.*[15] argued that in both batch and continuous systems, a continual increase in TS at some point becomes immaterial to the increasing volume of biogas produced. Therefore, higher precaution should be practiced when limiting the amount of water in favour of a drier digestion process. This may be particularly relevant to a water scarce nation such as Australia.

F. Substrate degradation

Once no significant CH_4 generation was detected, effluent characteristics for each assay were determined. All assays achieved high substrate degradation with regards to TS, VS, COD, and VFA (Figure 4). Final *p*H levels varied between 7.7 and 7.9 which helped to assure the digestion process was able to proceed under optimal conditions for methanogenic activities. Degradable fractions of waste, representative by the amount of VS, was degraded effectively with VS concentrations varying from 5.0 to 0.0 g/l, which amounted to 86.6%–100% removal efficiency within approximately 100 days incubation time (Figure 4). This implied a very high

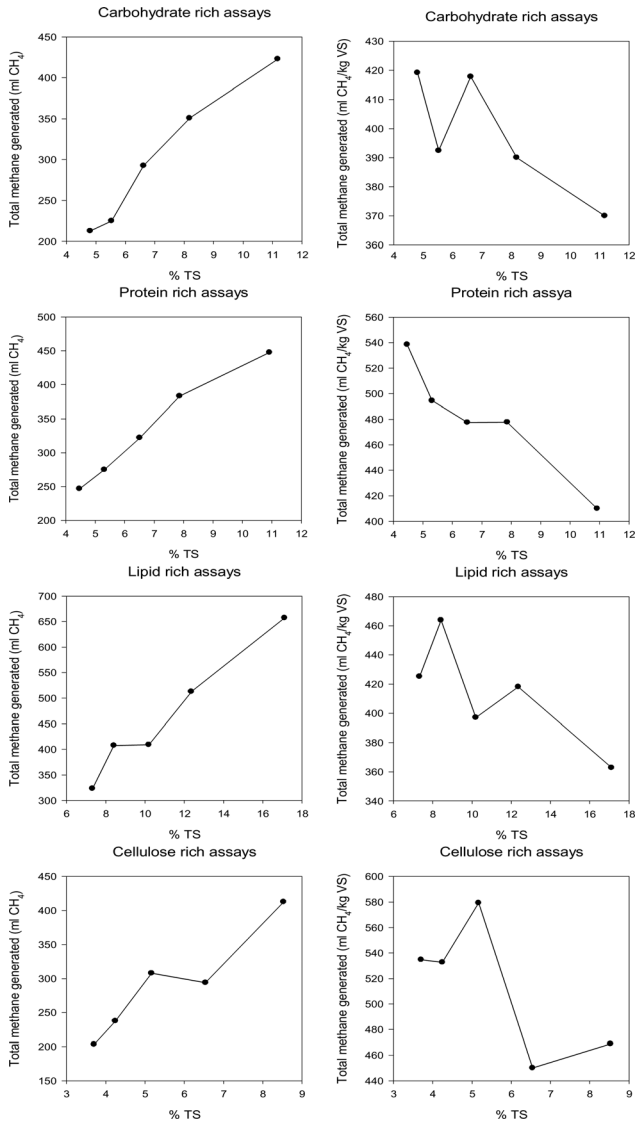


FIG. 3. Effects of %TS on total CH_4 generated (ml CH_4) (left hand side) and CH_4 yield per kg VS added (right hand side).

substrate conversion to biogas, which confirmed the total cumulative yield to be that of the B_o of each substrate. With a retention time of up to 100 days, a significant reduction in volumetric waste of between 64.7% and 96.0% to a TS concentration of between 16.7 and 1.7 g/l could be assured. sCOD and VFA were degraded between 87.1% and 98.0% with residue values of 4.0–0.9 g/l and 77.0–4.0 mg/l, respectively (Figure 4).

Final VFA levels for all food waste samples were low displaying no permanent inhibition by VFA which is an intermediately product of anaerobic digestion. Despite the high reduction in COD, COD values in all effluents remained significantly higher as compared to the recommended wastewater quality requirements (<20 mg/l BOD) for release (EPA[32]). Therefore, instead of direct disposal of the effluent, application potential should be explored. This high level of COD and the volume of effluent generated each day should be taken into consideration when considering the implementation of such a system. This is especially so if the digester is planned in an area close to any water source. The issues with regards to effluent management in biogas plants were highlighted in Orlebeke.[33]

Substrates of higher dilution factors achieved lower degradation efficiencies as compared to drier samples with regards to TS, VS, COD, and VFA concentrations (Figure 4). Despite the

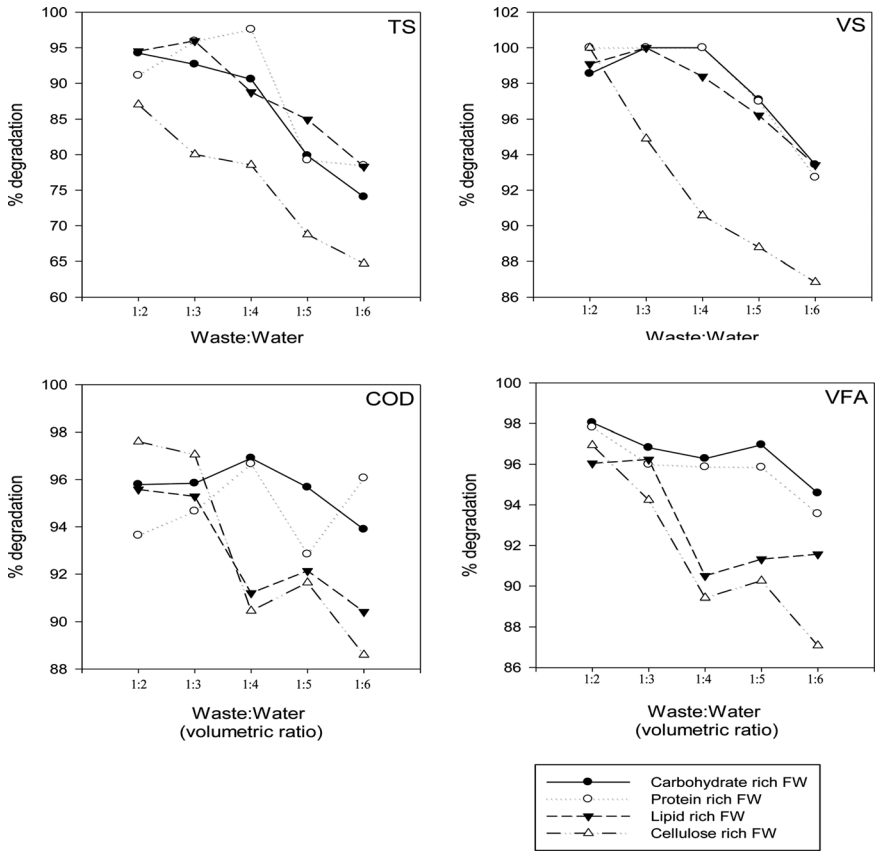


FIG. 4. Substrate degradation for carbohydrate, protein, lipid, and cellulose rich waste across five dilutions.

lower percentage conversions, the quantitative concentrations of COD and VFA were generally lower for assays of higher dilution factors. Comparing the performances of COD and VFA between the dilution factors, dilution 1:4 appeared to offer the lowest residue VFA and TS/VS for almost all samples (besides cellulose). 1:4 dilution would thus serve as the most appropriate rule of thumb dilution factor that would ensure efficient degradation.

IV. CONCLUSION

The amount of water added to food waste can influence the performance of a digester significantly with respect to CH_4 generation and substrate degradation. Despite achieving a higher total amount of CH_4 for waste with a high %TS, the SMP gradually decreased with increasing %TS. Batch studies showed that with sufficient retention time, waste high in %TS (1:2 dilution) can still achieve high substrate degradation for all macronutrient groups. Lipid rich waste required a longer retention time, and a dilution exceeding 1:4 is recommended. However, despite achieving high substrate degradation efficiency, effluent from the digester remains significantly higher than the standard required for its safe release into waterways irregardless of the %TS. While this study provides an introductory study with regards to performance evaluation of food waste digesters based on user friendly parameters, further studies were required to validate the conclusions drawn in this study, especially for the purpose of investigating different feedstocks.

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