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Effect of Temperature Fluctuation, Substrate Concentration, and Composition of Starchy Substrates in Mixture and Use of Plant Oils as Antifoams on Biogas Production

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This work investigated the effect of temperature fluctuations, substrate concentration and composition on foaming in anaerobic co-digestion of matooke, cassava, and sweet potato peels for biogas production as well as the use of plant oils as antifoams. In general, obtained results show that temperature fluctuations from mesophilic (≥25°C) to psychrophilic range (<25°C) is the major factor behind foaming in anaerobic digestion. Specifically, at concentrations of 6 and 9 g VS/L with methane yield of 1228.69 and 735.55 Nml CH_4/g VS, respectively, for 2:0:1 ratio, there was no foaming until after 7.6 days compared with the other ratios where foaming generally started after 3.5 days. This indicated that, apart from high substrate concentration, foaming is also a function of substrate composition. Plant oils were found to suppress foaming only if temperatures were above 25°C, indicating temperature fluctuations to be the main factor in foaming even in the presence of antifoams.

Keywords: temperature fluctuation, biogas production, foaming, antifoams, substrate concentration

HIGHLIGHTS

- ZnO based transparent conductive oxide (TCO) films were prepared by electro-deposition anodization on n-type (100) silicon wafer
- Simulation with AFSORS-HIT exhibited enhancement in ZnO/a-Si:H/n a-Si:H/i-c-Si/p/Al BSF/Al solar cell performance compared to control cell with no ZnO.
- The simulations of a-Si/c-Si heterojunction solar cell were carried out to reveal the cell parameters.
- Inclusion of ZnO-based TCO enhances PEC performance of the HIT solar cell.

INTRODUCTION

Anaerobic digestion is a well-known technology for organic waste management producing biogas—a sustainable clean energy. The amount of biogas produced from any given organic waste depends on a number of parameters such as temperature, type of organic waste, concentration and composition, pH, hydraulic retention time, and availability of microorganisms. Researchers and biogas plant operators often set temperature to be constant as required. The set temperature is always taken to be the actual temperature inside the incubators or digesters. However, this is not always the case due to inefficiencies of the monitoring systems [1]. In some digesters or incubators, temperature continuously fluctuates in such a way that may hinder digester performance if certain limits are exceeded. This scenario of temperature fluctuation was observed in previous investigations on hydraulic retention time and methane production kinetics with incubator temperature set at mesophilic temperature of 37°C. However, the actual temperature inside the incubator fluctuated between 28°C and 46°C and at one point, the lower temperature limit abruptly went as low as 26°C, causing an abrupt sharp decrease in biogas production [2]. Nevertheless, biogas production recovered after this temperature shock as reported by other researchers [3–5] investigating temperature fluctuation effects on biogas production. Studies available have only investigated temperature shocks between 5 and 12 h [3,6]. However, it is still unknown whether the process can recover after several days under this temperature shock. Temperature fluctuations have also been associated with foam formation in some literature [4,7] but with no experimental evidence, yet others who also investigated temperature fluctuation effects on anaerobic digestion were silent on foam formation [3,5,6]. This is not to mention that foam formation is also attributed to high initial substrate concentrations [4,8–10] and easily hydrolyzed substrates like sugar/starch containing substrates [11]. Sánchez *et al.* [12] also observed that increasing initial substrate concentration at ambient temperature (16.8°C–29.5°C) caused a reduction in volatile solids removal rate than at mesophilic temperature (35°C). In another study by Chae *et al.* [13], it was observed that regardless of the digestion temperature, substrate concentration from 5% to 40% decreased biogas yield with no mention of foam formation. Subramanian and Pagilla [14] did not observe foam formation when looking at the effects of organic loading rates on digester foaming, but concluded that it was due to the absence of a primary foaming cause. Kougias *et al.* [15] attributed foaming to combination of substrate composition and mixing pattern. Given the inconsistency in prevailing literature over temperature fluctuation, initial substrate concentration, and substrate composition in relation to foam formation in biogas digesters, more research is needed to enable attaining optimal operating conditions for any given substrate in terms of temperature range and volatile solids concentration for the stability of anaerobic digestion. This work would also provide on the relationship between temperature fluctuation, substrate concentration, substrate composition, and foam formation to aid in monitoring biogas digestion processes. Therefore, the objective of this work was to investigate the effect of temperature fluctuation, variation of volatile solids concentration, and substrate composition on anaerobic digestion process for the selected substrate ratios of matooke peels (MP), cassava peels (CP), and sweet potato peels (SP). The order of the ratios investigated (MP:CP:SP) were: 0:1:1; 0:1:2; 1:1:1; 1:1:4; 2:0:1; and 2:1:0 as based on the previous study [2].

MATERIALS AND METHODS

Substrate Selection

Following Tumutegyereize *et al.* [2], 16 substrate ratios of matooke peels, cassava peels and sweet potato peels (1:0:0, 0:1:0, 0:0:1, 1:1:0, 1:0:1, 0:1:1, 1:2:0, 1:0:2, 0:1:2, 0:2:1, 2:1:0, 2:0:1, 1:1:1, 4:1:1, 1:4:1, and 1:1:4) had been tested at a uniform *VS* concentration for their methane production. In terms of fitting onto first order models, having a positive synergetic effect on hydraulic retention time (HRT), hydrolysis rate constant, lag phase and methane yield, withstanding temperature shock and maintaining a relatively favorable pH for methanogens, ratios 0:1:1, 0:1:2, 2:1:0, 2:0:1, 1:1:1, and 1:1:4 performed well compared with the rest. The ratios that performed were therefore selected for concentration and temperature fluctuation investigation. Inoculum was collected from a household biogas digester fed with household organic waste and analyzed as described in Tumutegyereize *et al.* [2]. The characteristics of the selected substrate ratios and inoculum are presented in Table 1 in addition to their parent substrate.

Description of Experimental Setup

Following preliminary trials, three volatile solids concentrations for each of the selected ratios were used, that is, 3 g *VS*/L, 6 g *VS*/L, and 9 g *VS*/L. Serum bottles of 250 mL with a working volume of 150 mL were used in duplicate for each substrate ratio in batch digestion. For each of the concentrations, 80 mL of inoculum and 70 mL of tap water were added. Two control serum bottles were also set up each with 120 mL of inoculum and 30 mL of tap water. The bottles were then flashed with nitrogen gas, sealed, and placed in an incubator set at an average temperature of 30°C. However, the actual temperature fluctuated in the range of less than 25°C to above 45°C. Temperature fluctuations were monitored using a Vision type data harvester. The incubator setting of 30°C was maintained for 10 days and thereafter raised to 37°C for which there were no temperature fluctuations below 25°C. The temperature was set in this way to investigate the effect of temperature fluctuations on biogas production process across the boundary of psychrophilic and mesophilic temperature ranges [6]. In addition, this was to assess the effect of prolonged temperature fluctuations below 25°C on biogas production. However, based on literature, temperature fluctuations and increased substrate concentrations are said to cause foam formation that inhibit anaerobic digestion [9]. Therefore, in order to avoid foam formation, two plant oils (olive oil and sunflower oil) were selected for use as antifoams based on their differences in composition and availability. Olive oil composition was 14% palmitic acid, 9% linoleic acid, and 68% oleic acid while that of sunflower oil composition was 11.6% palmitic acid, 23% linoleic acid, and 57% oleic acid. Olive oil was selected due to its high percentage of oleic acid compared with sunflower oil given that oleic acid has less inhibitory effect on anaerobic process [16]. Sunflower oil on the other hand was selected due its low cost and relative availability at household level compared with olive oil.

Foam Formation Monitoring and Antifoam Addition

From day one when the bottles with their content were put in the incubator, the experiment was monitored for foam formation. In which ever bottle foam formation occurred, 1 mL of either olive oil or sunflower oil was added once at random using a syringe through the rubber septum. The 1 mL equivalent to 0.4% of the digester volume was based on suggestions from literature that foam suppression is enhanced by increasing the amount of antifoam [10]. Monitoring of foam formation continued throughout to determine whether addition of plant oil caused foam disappearance and enhanced biogas production under temperature fluctuation and volatile solids concentration variation. Control serum bottles with inoculum and plant oils were also setup to determine the methane potential of the oils during antifoaming. Procedures already described by Tumutegyereize *et al.* [2] were followed for gas measurements and data processing.

RESULTS AND DISCUSSION

Composition of Substrate Ratios Under Investigation

Table 1 presents the characteristics of the substrate ratios used. Although there were no significant differences between the substrate ratios in terms of their organic fraction composition at 5% alpha level, ratio 2:0:1 showed to be low in terms of proteins having high C:N ratio of 45.6 compared with the rest. Conversely, the same ratio 2:0:1 together with 1:1:1 ratio were also high in terms of cellulose content compared with the rest.

Table 1. Characteristics of the substrate ratios used.

Substrate	C%	N%	%Ash	%TS	%*VS*	%ADL	%Cel	%Hem	%Fat	%Protein	% Car	C:N
1:0:0	40.75	0.9	8.48(4.1)	95.80	91.46(0.3)	1.86(15.3)	9.11	33.81	2.64	5.63	74.08	45.28
0:1:0	41.40	1.9	3.26(4.0)	95.61	96.62(0.1)	2.76(12.2)	20.52	7.23	0.76	11.88	63.45	21.79
0:0:1	41.11	0.9	6.41(0.9)	95.66	93.42(0.1)	2.68(5.0)	9.75	24.83	2.23	5.63	75.81	45.67
2:1:0	40.84	1.3	6.42(1.8)	97.35	93.51(0.2)	1.22(20.9)	7.45	27.73	2.18	8.13	75.75	31.42
2:0:1	41.08	0.9	8.17(1.6)	95.55	91.40(0.3)	2.55(22.1)	13.02	23.85	2.35	5.63	70.41	45.64
0.1:2	41.05	1.1	5.47(1.5)	96.12	94.30(0.3)	1.63(14.0)	10.69	18.60	2.31	6.88	74.42	37.32
0:1:1	41.06	1.3	4.70(4.3)	97.18	95.29(0.2)	3.19(15.7)	8.73	23.01	1.58	8.13	76.86	31.58
1:1:1	41.05	1.2	6.20(3.5)	97.46	93.67(0.3)	2.14(7.4)	13.40	19.89	2.24	7.50	70.53	34.21
1:1:4	42.16	1.1	6.31(1.7)	97.30	93.58(0.1)	2.83(14.4)	10.19	23.58	2.19	6.88	74.32	38.33
Inoculum	–	–	1.24(1.1)	–	65.25(0.3)	–	–	–	–	–	–	–

NB: Values in brackets represent the coefficients of variation (cv); C, carbon content; N, nitrogen content; TS, total solids; VS, volatile solids; ADL, acid detergent lignin; Cel, cellulose; Hem, hemicellulose; Car, degradable carbohydrates.

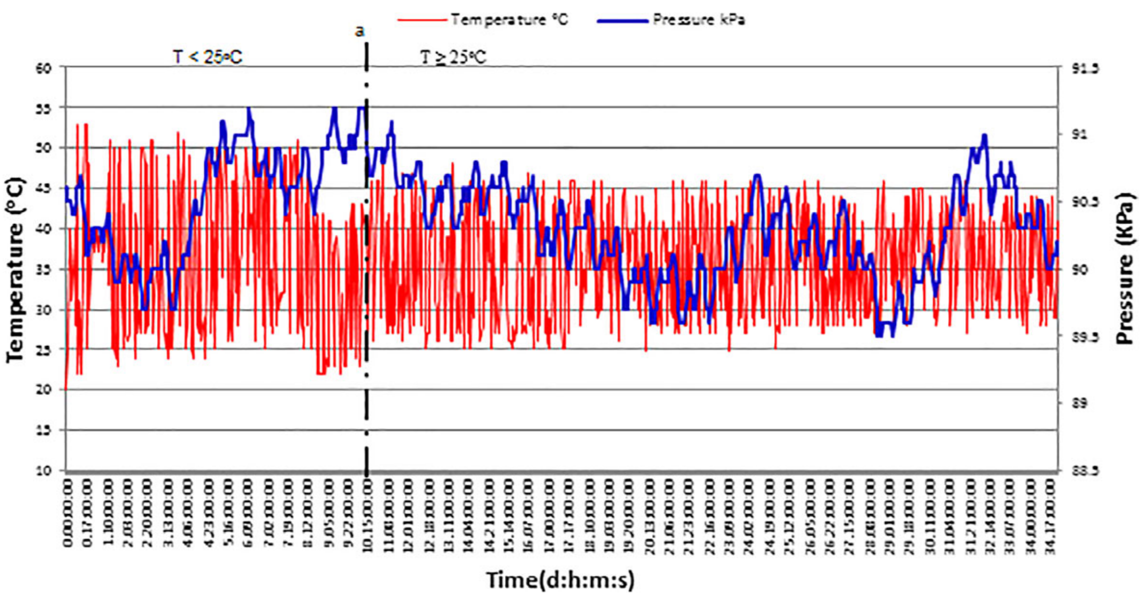

Figure 1. Temperature fluctuation profile below 25°C for 10 days and above 25°C for 25 days. [Color figure can be viewed at wileyonlinelibrary.com]

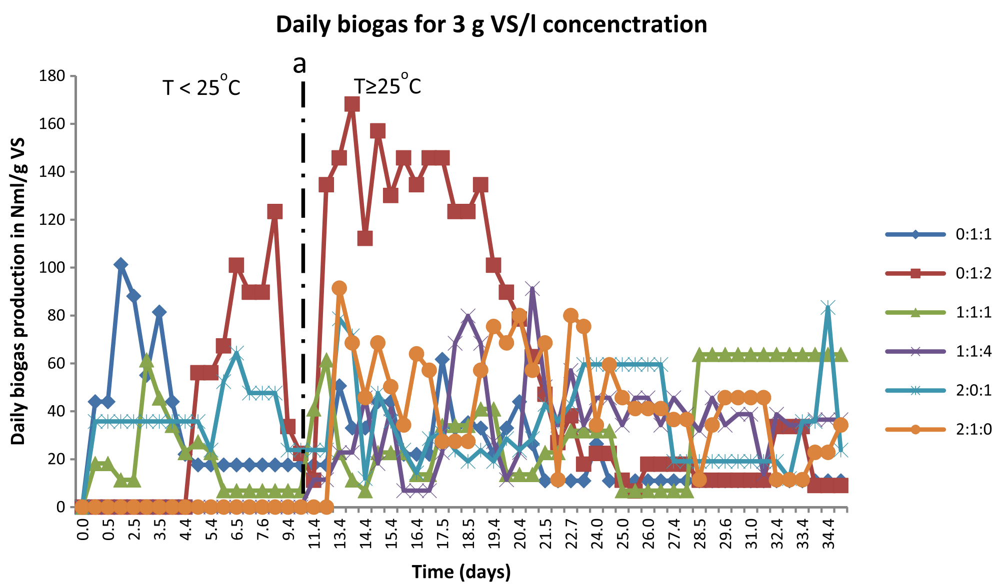

Figure 2. Daily biogas production under two temperature profiles for 3 g *VS*/L substrate concentration. [Color figure can be viewed at wileyonlinelibrary.com]

Ratio 0:1:0 had the highest cellulose content of 20.52%. Differences in organic fraction composition even when not significant, probably, bring about differences in performance when used as substrates for biogas production. If optimal biogas production is to be realized, there is need to pay attention to substrate ratio proportions of any new substrates.

Temperature Fluctuation Effect on Foam Formation and Biogas Production

Figure 1 shows two temperature profiles ($T < 25°C$ and $T \geq 25°C$) separated by the black vertical line labeled "a," under which the different substrate ratios in three different *VS* concentrations were anaerobically digested. For purposes of guiding the reader, Figures 2–4 are explained in relation to Figure 1. Temperature fluctuating below 25°C remained the limiting factor to biogas production. In the temperature profile $T < 25°C$, despite increase in concentration in Figures 2–4, there was a general trend for biogas production. That is, depending on the substrate ratio, biogas production either decreased or ceased. In other cases, no biogas was produced at all. For example, Figure 2 shows substrate ratio 0:1:2, at 3 g *VS*/L concentration which tended to withstand the low temperatures without foam formation. However, biogas production dropped sharply by 68.3% (from 120 mL to about 38 mL) after day eight. This is because after day eight, temperature was fluctuating below 25°C almost after every hour as evidenced in Figure 1. Given that $T < 25°C$ represented the psychrophilic temperature range with its own group of microbes that are favored, and $T \geq 25°C$ represented mesophilic temperature range with

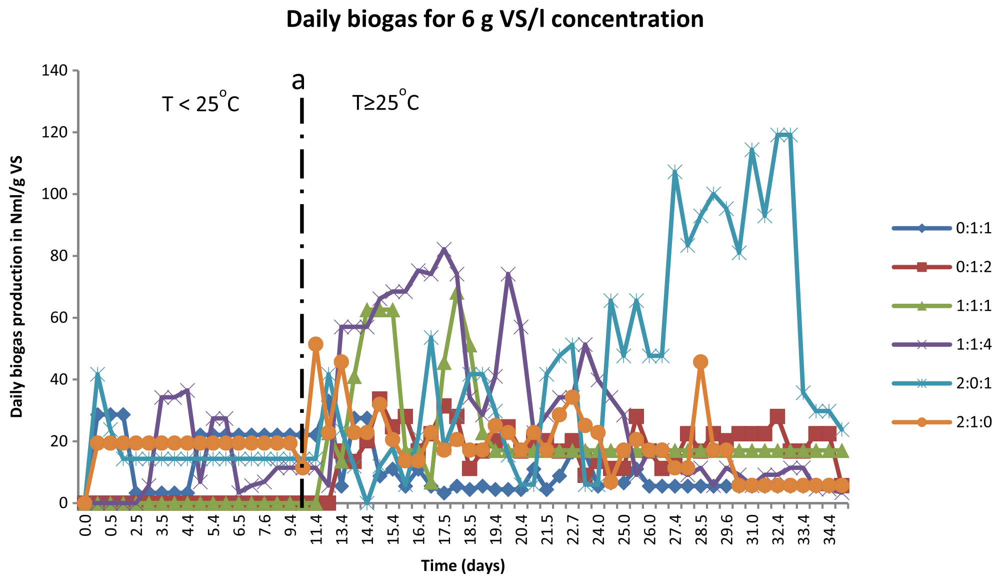

Figure 3. Daily biogas production under two temperature profiles for 6 g *VS*/L substrate concentration. [Color figure can be viewed at **wileyonlinelibrary.com**]

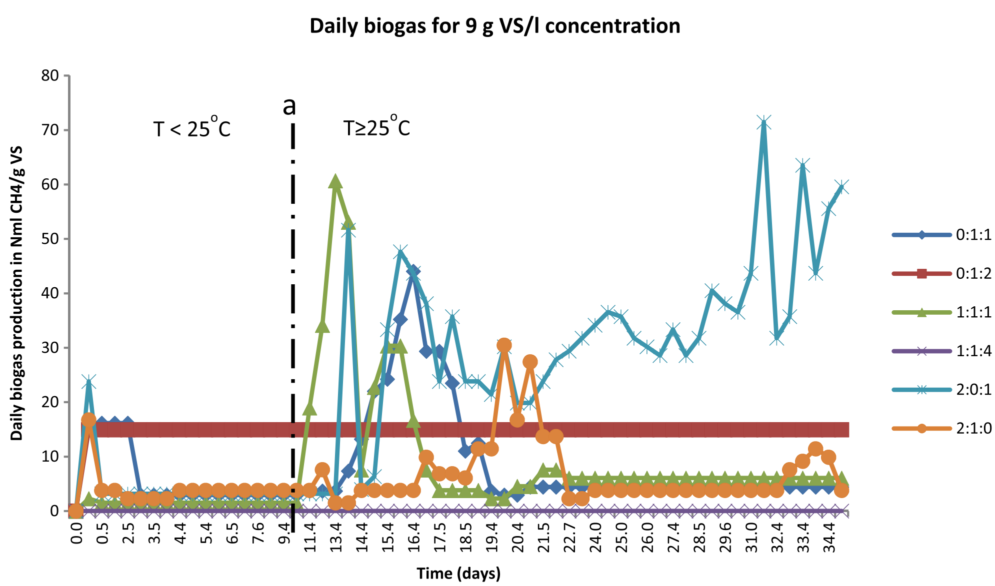

Figure 4. Daily biogas production under two temperature profiles for 9 g *VS*/L substrate concentration. [Color figure can be viewed at **wileyonlinelibrary.com**]

another group of microbes, probably microbes favored by the mesophilic temperature could have been affected when temperature changed to the psychrophilic range. In addition, the different substrate ratios at different *VS* concentrations responded differently to temperature fluctuations below 25°C in terms of biogas production and foam formation. Foam with large bubbles covering the whole slurry surface in the bottle started forming in the temperature region of $T < 25°C$ in almost all substrate ratios with 3 and 6 g *VS*/L loading concentration, but limited foaming was observed in the 9 g *VS*/L. This foam formation in these particular substrate ratios occurred on different days within the first 10 days. Fluctuating temperature below 25°C probably favored increased solubility of carbon dioxide in the digestate. This could have caused acidic conditions in the substrate ratios 0:1:2 and 1:1:4 at 9 g *VS*/L concentration where no biogas and no foam were generated. Therefore, biogas production probably plays an important role in foam formation during anaerobic digestion.

However, when a temperature shift was made to $T \geq 25°C$ region as seen in Figure 1, biogas production either started or increased for almost all the substrate ratios irrespective of the *VS* concentrations as shown in Figures 2–4. This confirms that biogas production is a function of temperature. Foaming on the other hand ceased after 1 or 2 days for all the substrate ratios with the exception of 2:0:1 and 2:1:0 ratios at 9 g *VS*/L concentration that took more than 3 days for foam to disappear after temperature change to $T \geq 25°C$. This suggests that once foaming has occurred in substrate ratios with higher *VS* concentrations, more time is needed for it to disappear even at the favorable

Table 2. Results of anaerobic digestion process of MP:CP:SP substrate ratios under varying substrate concentrations with addition of plant oils as antifoams.

Substrate ratio (MP:CP:SP)	Antifoam (plant oil)	Biogas (Nml/g *VS*)	Specific Methane (Nml CH_4/g *VS*)	Methane content (%)	pH	%*VS* reduction	Lag before foaming (days)	Foam ceasing day
			3 g *VS*/L concentration					
0:1:1	Sunflower	1046.12	601.52[b]	57.5	6.98	55.20	5.9	13.4
0:1:2	n/a	3060.29	1774.97[a]	58.0	7.06	69.24	No foam	n/a
1:1:1	Olive	701.10	431.88[c]	61.6	6.42	56.16	6.5	11.4
1:1:4	Sunflower	1418.87	813.02[b]	57.3	7.82	66.06	3.5	12.4
2:0:1	Olive	1227.17	674.95[b]	55.0	6.89	52.82	3.5	12.4
2:1:0	Olive	1572.45	924.60[b]	58.8	6.80	57.65	3.5	13.4
1:0:0	n/a	620	280a	50.5	6.11	66.40	No foam	n/a
0:1:0	n/a	260	180[b]	50.2	6.01	64.06	No foam	n/a
0:0:1	n/a	290	140[c]	48.8	5.94	64.37	No foam	n/a
			6 g *VS*/L concentration					
0:1:1	n/a	298.42	183.53[d]	61.5	6.78	66.96	No foam	n/a
0:1:2	Sunflower	778.53	456.22[c]	58.6	6.67	63.13	6.5	13.4
1:1:1	Olive	368.76	224.94[d]	61.0	6.58	70.97	3.5	12.4
1:1:4	Olive	1843.17	1008.21[a]	54.7	7.12	72.98	2.46	13.4
2:0:1	Olive	2186.28	1228.69[a]	56.2	7.11	68.27	7.6	12.4
2:1:0	Sunflower	751.94	432.37[c]	57.5	6.75	59.75	3.5	11.4
			9 g *VS*/L concentration					
0:1:1	n/a	289.24	155.03[d]	53.6	7.71	69.30	No foam	n/a
0:1:2	n/a	14.96	ND	ND	5.6	*ND*	No foam	n/a
1:1:1	n/a	323.99	183.06[d]	56.5	6.85	64.56	No foam	n/a
1:1:4	n/a	0.00	ND	ND	4.71	ND	No foam	n/a
2:0:1	Olive	1374.12	735.15[b]	53.5	7.13	87.83	7.6	13.9
2:1:0	Sunflower	232.36	128.73[d]	55.4	6.15	64.97	3.5	16.9
			Controls					
Inoculum	Olive	352.76	199.44	57.9	7.01	ND	No foam	n/a
Inoculum	Sunflower	534.43	327.70	60.5	6.31	ND	No foam	n/a

NB: MP, matooke peels; CP, cassava peels; SP, sweet potato peels; VS, volatile solids; n/a stands for not applicable while ND stands for not determined; values with different subscripts show significant difference at 5% alpha value.

temperature. However, proof of the influence of higher *VS* concentrations on foam formation is required. From these observations, it can also be concluded that temperature fluctuations across two temperature ranges (mesophilic to psychrophilic) seem to be the primary cause of foam formation at specified *VS* concentrations. It is interesting to note that Lienen *et al.* [17] attributed foaming to addition of fat, oil and grease (FOG) in the digester whose operating temperature was fluctuating between 38°C and 42°C, which is also a boundary of mesophilic and thermophilic according to El-Mashad *et al.* [6]. Most probably temperature fluctuation across the two ranges could have been the cause of foaming given that in other cases where FOG had been added, foaming did not occur [18]. In general, results show that the process of anaerobic digestion is able to recover regardless of substrate concentration and how long it takes under low temperature, provided there is no buildup of intermediary compounds to cause toxicity.

Effect of Plant Oils in Antifoaming and Biogas Production

Table 2 presents the time in days that elapsed before foam formation started in different substrate ratios in relation to *VS* concentrations. It also presents the day when foaming ceased after temperature change and introduction of antifoam. It was observed that addition of antifoams alone in the substrate ratios where foam formation had occurred; did cease foam formation until there was a shift in temperature profile from $T < 25°C$ to $T \geq 25°C$. Moreover, neither olive oil nor sunflower oil affected biogas production while the temperature was fluctuating below 25°C. However, when temperature changed to $T \geq 25°C$, biogas production generally doubled in all substrate ratios at 3 g *VS*/L concentration regardless of the type of plant oil that had been added. Only results of the mono-substrates (1:0:0, 0:1:0, and 0:0:1) have been reproduced in this work (Table 2) from Tumutegyereize *et al.* [2] as reference substrates to show the effect of co-digestion and plant oil addition on biogas production. This confirms that addition of plant oils as antifoams has a synergetic effect on biogas production which agrees with findings of Kougias *et al.* [10]. Nevertheless, olive oil proved superior to sunflower oil at higher *VS* concentrations as methane yield values were significant at 5% alpha value. This could be attributed to the high percentage of mono-saturated fatty acids of 68% in olive oil compared with 57% in sunflower oil. Since these plant oils especially sunflower oil are readily available and used in almost every household for daily cooking activities, it is worth looking into the economic and environmental use of adding plant oils into domestic household biogas digesters to enhance biogas production.

Volatile Solids Concentration Effect on Foam Formation and Biogas Production

Table 2 further presents the amount of biogas and methane generated from the individual substrate ratios with respect to *VS* concentrations. With the exception of 1:1:4 and 2:0:1 ratios that performed well at 6 g *VS*/L loading concentration in terms of either biogas or methane yield, the rest of the substrate ratios showed favorable results when loaded at 3 g *VS*/L. The 2:0:1 ratio performed well even at 9 g *VS*/L concentration with 735.15 Nml CH_4/g *VS* methane volume. This suggests that *VS* concentration is substrate ratio-specific when favoring or hindering biogas/methane production.

In addition, foaming generally occurred in all substrate ratios with 3 and 6 g *VS*/L loading concentration. This is in close agreement with Ganidi *et al*. [8] who established that 2.5 kg *VS* m^{-3} was the critical loading rate for foam initiation and that at 5 kg *VS* m^{-3}, foam was persistent. However, as *VS* concentration increased to 9 g *VS*/L, foaming appeared only in 2:0:1 and 2:1:0 ratios. This observation points to foaming in digesters being a function of substrate ratio composition as was observed by Kougias *et al*. [14]. On the other hand, substrate ratios in which there was no foaming with 9 g *VS*/L loading concentration, suggested that high concentration leads to fall in pH making the environment around microbes acidic and hence inhibiting the anaerobic digestion process. This is supported by the low or no biogas production in the substrate ratios where there was no foam formation for the 9 g *VS*/L concentrations and in particular, 0:1:2 and 1:1:4 ratios with pH of 5.6 and 4.71, respectively. Therefore, contrary to observations in literature [9], foam formation may not be an inhibitor itself, but an indicator that certain factors such as temperature fluctuation below a threshold lower limit in combination with substrate composition are hindering the process of biogas production.

CONCLUSIONS

Substrate ratio of 2:0:1 at 6 and 9 g *VS*/L proved superior with 1228.69 and 735.55 Nml CH_4/g *VS,* respectively. This ratio took longer time (7.6 days) before foaming compared with the other ratios at 3 g *VS*/L concentration, implying that foaming depends on substrate composition. In addition, temperature fluctuation from mesophilic (≥25°C) to psychrophilic range (<25°C) was found to be the major factor influencing foaming in anaerobic digestion. In the presence of antifoams, foaming could not be suppressed until temperature ≥25°C was maintained. Further investigation may, however, be required at the mesophilic–thermophilic boundary for confirmation.

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