The impact of thermal pretreatment on various solid-liquid ratios of Palm Oil Mill Effluent (POME) for enhanced thermophilic anaerobic digestion performance

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Abstract

An innovative treatment process for the treatment of palm oil mill effluent (POME) was proposed whereby a pretreatment technology and a dewatering device are introduced into the existing treatment process. Thermal pretreatment is a foolproof technique with the ability to enhance the rate and ameliorate the biogas production of anaerobic digestion. The dewatering device will infer a means of control on the digesters' load, allowing the removal of microbes and impurities as well as assist in the residual oil removal. The novel treatment process allows the removal of cooling ponds making the treatment process more sustainable in terms of the substantial reduction in the amount of greenhouse gas emission, improved residual oil removal efficiency in the waste stream, and better treated effluent quality. However, to be able to implement this innovative treatment method effectively, it is fundamental to know how thermal pretreatment effected the solid content of POME impacts on the anaerobic digestion process performance. To undertake the study mentioned above, POME was pretreated at 120℃ and was allowed to settle to separate the solid and liquid phases. The chosen method of anaerobic digestion was batch thermophilic anaerobic digestion, which was conducted on various solid: liquid ratios (i.e., 20S:80L, 40S:60L, 50S:50L, 75S:25L, and 100S). It was found that the optimal ratios were 20S:80L and 40S:60L, which generated approximately 9-fold and 6-fold higher methane yield, respectively, in contrast to their untreated counterparts. Thermally pretreated 40S:60L solid loading exhibited a higher removal efficiency in terms of chemical oxygen demand (COD), biological oxygen demand (BOD), total suspended solids (TSS), and oil & grease (O&G), a higher methane yield of 328.73 mL CH_4/g $COD_{removed}$ and biogas production of 1886.11±21.63 mL compared to all the other pretreated and untreated ratios.

Keywords: Palm oil Mill Effluent, Thermal Pretreatment, Solid loadings, Biogas Production, and Methane Yield.

Contents

1. Introduction

There are different categories of pretreatment techniques that have been studied over the years when it comes to wastewater treatment. Some examples of these pretreatment methods are thermal, biological, chemical, and mechanical pretreatment methods (Carrère et al., 2010). Amongst these pretreatment techniques, some have been commercialized and implemented on the full scale (e.g., thermal pretreatment: conventional heating) whilst others are still at research stages (e.g., chemical pretreatment: advanced oxidation) (Tyagi and Lo, 2011). Pretreatment technologies are known to significantly enhance the anaerobic digestion process as they prominently ameliorate the digestion rate, lessen the retention time, and increase the biogas production (Khadaroo et al., 2019b).

Palm Oil Mill Effluent (POME) is a waste associated with the production of palm oil. It is the primary waste contributor in Malaysia, which is the second-largest palm oil producer worldwide (Lam Man & Lee, 2010). The disposal and waste management of POME is an alarming concern for Malaysia since POME can cause pollution when discharged into watercourses due to the high amount of easily degradable organic matter, thereby referring to the high chemical oxygen demand (COD), biological oxygen demand (BOD) and the total suspended solids (TSS) contents which are of an average concentration of 50,000 mg/L, 25,000 mg/L, and 20,000 mg/L respectively (Choong et al., 2018; Iskandar et al., 2018). Another major drawback when it comes to the treatment process of POME is that the chemical characteristics of POME mentioned above typically depend on the efficiency of the palm oil extraction process as well as its chemical characteristics during the high and low crop seasons; owing to these fluctuating parameters, the chemical characteristics of POME tend to vary a great deal thus affecting the efficiency of the treatment process (Poh et al., 2010). Having such high COD and BOD values along with the inconsistency of these parameters, the anaerobic digestion process is subsequently hindered, and a lower anaerobic digestion rate causes the concentration of toxic substances produced in the process to rise (Li et al., 2019). Moreover, owing to the facultative and the cooling ponds, the current treatment process contributes to significant environmental burdens in terms of excessive greenhouse gas emission and water pollution, which is damaging to the aquatic ecosystem (Tajuddin et al., 2015).

In the currently implemented POME treatment process, raw POME is directed to a cooling pond where residual oil in POME is scraped off the surface of the cooling ponds to recover crude palm oil (CPO) lost into the waste stream. It is then sent for anaerobic digestion, which is the primary treatment, followed by a secondary treatment process known as the aerobic treatment (Bala et al., 2014). The aerobic treatment process is meant to reduce the COD and BOD further; however, the aerobic treatment process consumes an excessively high amount of energy (Singh et al., 2010). The drawback concomitant with the current process is that raw POME discharged at a temperature of 90°C is sent to the cooling ponds which are highly inefficient in terms of the residual oil extraction, and a significant amount of heat is lost to the atmosphere (temperature drops from 90°C to 60°C). POME is then further allowed to cool down to an adequate mesophilic temperature (35°C) before being sent for anaerobic digestion. Furthermore, often the treated effluent quality from the current treatment process is inadequate and does not conform to the stringent environmental standards stipulated by the Malaysian Government, stating that the COD concentration of the treated effluent should be no more than 50mg/L (Chin et al., 2013; Ahmed et al., 2015).

Subsequently, a substitute treatment process was recommended, which entails eliminating the cooling ponds and introducing a pretreatment technology coupled with a dewatering device such as a thickener prior to the anaerobic digestion process. The pretreatment technology will assist in enhancing the rate-limiting step of anaerobic digestion namely the hydrolysis step while the dewatering device will contribute to making the anaerobic digesters more efficient by inferring a means of control on the digesters' load during the treatment process of POME by regulating the amount of solids and liquid required in the system to ensure best conditions for the consortium of bacteria to thrive (Li et al., 2019). Another advantage of the dewatering device is that it will aid in controlling the load of the digesters making the anaerobic digestion process comparatively more stable when there is a drastic change in the chemical characteristics of POME as it varies from high crop season to low crop season (Khadaroo et al., 2019a). The pretreated POME would then be directed to the anaerobic digesters, which will operate at a higher thermophilic temperature (55°C) since thermophilic anaerobic digestion can achieve a higher solids reduction and biogas production, better resistance to foaming, better pathogens destruction and enhancement of the energy balance of the whole treatment process (Dohányos et al., 2004). The treated effluent quality will, therefore, improve considerably, reducing the load on the aerobic treatment, which will, in turn, reduce the energy consumption of the latter (Appels et al., 2011).

Khadaroo et al. (2019b) have thoroughly studied the effect of different pretreatment techniques on the anaerobic digestion of POME from various literature sources and assessed which method was most suitable in terms of treated effluent quality, biogas production, environmental encumbrance as well as the technical and economic feasibility. They found that for the treatment of POME, thermal pretreatment was the most adequate option as the energy consumption could be easily compensated by the amount of biogas produced, and the treated effluent quality can be significantly improved. Thermal pretreatment is also known to enhance digestion efficiency, and increase process performance as well as stability (Farhat et al., 2018). Thermal pretreatment can improve dewaterability, which will further assist in the solid: liquid separation (Higgins et al., 2017). These alterations in the POME treatment process will notably enhance the anaerobic digestion process, upsurge the amount of biogas produced, provide better treated effluent quality, all while drastically reducing the greenhouse gas emission to the atmosphere via the ponds. The biogas generated will be captured and utilized as per the mill requirement. This substitute treatment process makes the process more sustainable in terms of reduction in greenhouse gas emission and waste produced since with a better treated effluent quality, the treated solids can be used as A-grade fertilizers in palm tree plantations.

However, to comprehend how the thermal pretreatment of different solid loadings affects anaerobic digestion of POME and to find out at which solid loading to operate the digesters for optimal anaerobic digestion performance, it is fundamental to methodically understand how these two factors influence anaerobic digestion of the POME. Owing to a distinctly restricted amount of data on the topic, this paper aims to bridge the gap on the influence of the various solid-liquid ratios of POME and the impact of thermal pretreatment of these ratios on the anaerobic digestion performance.

2. Materials and Methods

2.1 Materials

The substrate for anaerobic digestion used is POME. POME was sampled at the Sime Darby East Oil Mill, Malaysia. The POME collection site lies within coordinates 2.8843° N, 101.4369° E. The temperature of POME at the sampling location was recorded to be 65°C. Anaerobic seed sludge, which was used as inoculum, was also collected from the same mill.

2.2 Experimental Set up

2.2.1 Solid-Liquid Separation

To separate the solid from the liquid phase, 5L of POME was carefully poured in a settling column of height 0.7 m with multiple sampling ports to allow the removal of each phase. POME at 65°C was used in the process for the suspension to settle effectively as a study conducted by Khadaroo et al. (2019a) indicated that POME at room temperature does not settle but instead tends to form lumps of solid flocs that float along the settling column. The solid flocs in hot POME were left to settle for approximately 24hrs to ensure the distinct separation of the phases. Samples of settled suspension and clear liquid were hereafter designated ``solid" and ``liquid" in this study.

After the settling of raw POME into solid and liquid phases, they were recombined to achieve the desired ratios by volume for this study (i.e., for a feed volume of 100 ml, the 20S:80L ratio consists of 20 mL of settled suspension and 80 mL of clarified liquid). The opted solid: liquid ratios were 20S:80L, 40S:60L, 50S:50L, 75S:25L, and 100S:0L (100S). Anaerobic digestion experiments on both thermally treated and untreated POME of the ratios mentioned above were undertaken to gain an insight into how significant the anaerobic digestion performance was improved across all ratios. These specific ratios were selected to allow the study over the full range of solids ratios for a complete picture of the performance of anaerobic digester.

2.2.2 Thermal Pretreatment

Prior to the anaerobic digestion process for the pretreated experiments, the solids from POME suspension was thermally treated after the separation of the solids from the liquid phase, as described above. The thermal treatment was undertaken by heating the solids at 120°C for an hour in an oven. Thermal pretreatment temperatures can range from 100 to 190°C, and thermal pretreatment effected at temperatures below 150 °C tends to be a more cost-effective process performance as compared to other pretreatment methods (Ariunbaatar et al., 2014). However, 120°C was chosen to ensure the compensation of energy input through the expected increase in biogas production, which can be used for heating purposes in the mill. Since the temperature is required to be raised by 30°C only, the boost in the amount of biogas produced can easily compensate for the energy input into the system. To ensure homogenous heating, the medium was stirred several times during the thermal treatment. Once the solid was pretreated, the appropriate volume of the liquid phase was added to the treated solid. The pH of the system was brought to 7.20 by dosing with 1M $NaHCO_3$. After pretreatment and recombining of the specific ratios, the samples were stored in a refrigerator below 4 °C (Wood et al., 2009).

2.2.3 Anaerobic Digestion experimental procedure

Thermophilic batch anaerobic digestion was the chosen mode of anaerobic digestion. The requisite ratio was placed in a 250ml Schott bottle with two outlets. The working volume of the system was set as 100mL. The anaerobic seed sludge (inoculum) was acquired from the anaerobic treatment ponds at the mill site. The inoculum volume was taken and maintained as 20% of the working volume throughout the experiments. The inoculum was cultivated and acclimatized in a reactor heated at 55°C under anaerobic conditions for one month to allow the bacteria to be accustomed to the conditions under which the anaerobic digestions experiments were undertaken as described in a study by Poh and Chong. (2010). The inoculum volume was kept constant in all the tested conditions to be able to gauge how thermal pretreatment affects the anaerobic digestion performance of the different solid: liquid ratios, particularly as inoculum to substrate ratio, has proven to affect the anaerobic digestion performance (Cappai et al., 2015). The digesters were heated and continuously stirred using a hot plate magnetic stirrer for appropriate homogenization of the medium.

Each digester was connected to a water displacement column via a pipe to enable the biogas generation volume measurement. As per ASTM D5511, the pH of the water in the displacement column was adjusted to 2.0 using 1M H_2SO_4 to prevent carbon dioxide from dissolving into the water. The latter ensures that the biogas measurement via the water displacement method is carried out more accurately (Müller et al., 2004). The pH of the medium consisting of the mixture of the solids and the liquid was kept between 6.8 to 8.0 by dosing 1M $NaHCO_3$ to ensure optimum conditions for the methanogenic bacteria to convert the available substrate into methane gas. To ensure that the pH was within the mentioned range, the pH was tested daily. The composition of biogas was measured using the Binder

COMBIMASS Gas Analyzer. The experiments were brought to an end when the Binder COMBIMASS Gas Analyzer detected no more methane in the digesters (Khadaroo et al., 2020).

Three assays for each ratio (20S:80L, 40S:60L, 50S:50L, 75S:25L, and 100S) were undertaken for which each test consisted of three batch anaerobic digesters. Triplicates were also used to conduct physico-chemical analyses. This set-up produced a total of 9 sets of data for each condition, which presented sufficient data for an efficient experimental analysis for this study.

2.3 Physico-chemical Analysis

The chemical characterization tests such as the COD, BOD, TSS, and O&G experiments were carried out before and post anaerobic digestion to determine the quality of the effluent discharged from the anaerobic digesters. The COD, BOD, and TSS tests were performed as per the HACH Standard Methods 8000, 8043, and 8006, respectively, while the O&G test was done as per the ASTM method D7066-04. The most probable number (MPN) was carried out as per ASTM STP695. Total solids test was undertaken as per the standard methods for the examination of water and wastewater to measure the solid content present in each tested condition (APHA, 1998). All the tests mentioned above are approved by the United States Environmental Protection Agency (USEPA), which is in accordance with ASTM standards as well as the APHA. The methane yield was calculated using the volume of methane produced and the mass of $COD_{removed}$ (Heidrich et al., 2011; Jingura and Kamusoko, 2017). The mass of $COD_{removed}$ was used for this study for better accuracy instead of Volatile Solids removed ($VS_{removed}$) as often used in other literature; since POME consists of 95-96% of water with only 4-5% of total solids (Iskandar et al., 2018; Khadaroo et al., 2019b).

3. Results and Discussion

The results obtained for the anaerobic digestion of untreated and thermally pretreated POME at 20S:80L, 40S:60L, 50S:50L, 75S:25L, and 100S solid loadings are presented below. The error bars in Figures 1,2,3, and 4 were calculated based on the standard deviations from the 9 sets of data obtained. Figure 1 illustrates the daily cumulative biogas production for untreated POME, while Figure 2 shows the daily cumulative biogas production for the thermally pretreated conditions. It can be observed that the anaerobic digestion durations of the thermally treated samples are shorter than that of the untreated conditions, as shown in Tables 1 and 3. An explanation for this occurrence may be due to the thermal pretreatment of the effluent, the microbial cell walls holding the intracellular matter rupture during pretreatment releasing the intracellular substances in the liquid phase which allowed the microorganisms to break down the latter faster as it is more easily and readily available (Pilli et al., 2015a, 2015b). This phenomenon, in turn, noticeably ameliorates the digestion rate, lessens the retention time, and augments the biogas production (Appels et al., 2008, 2011).

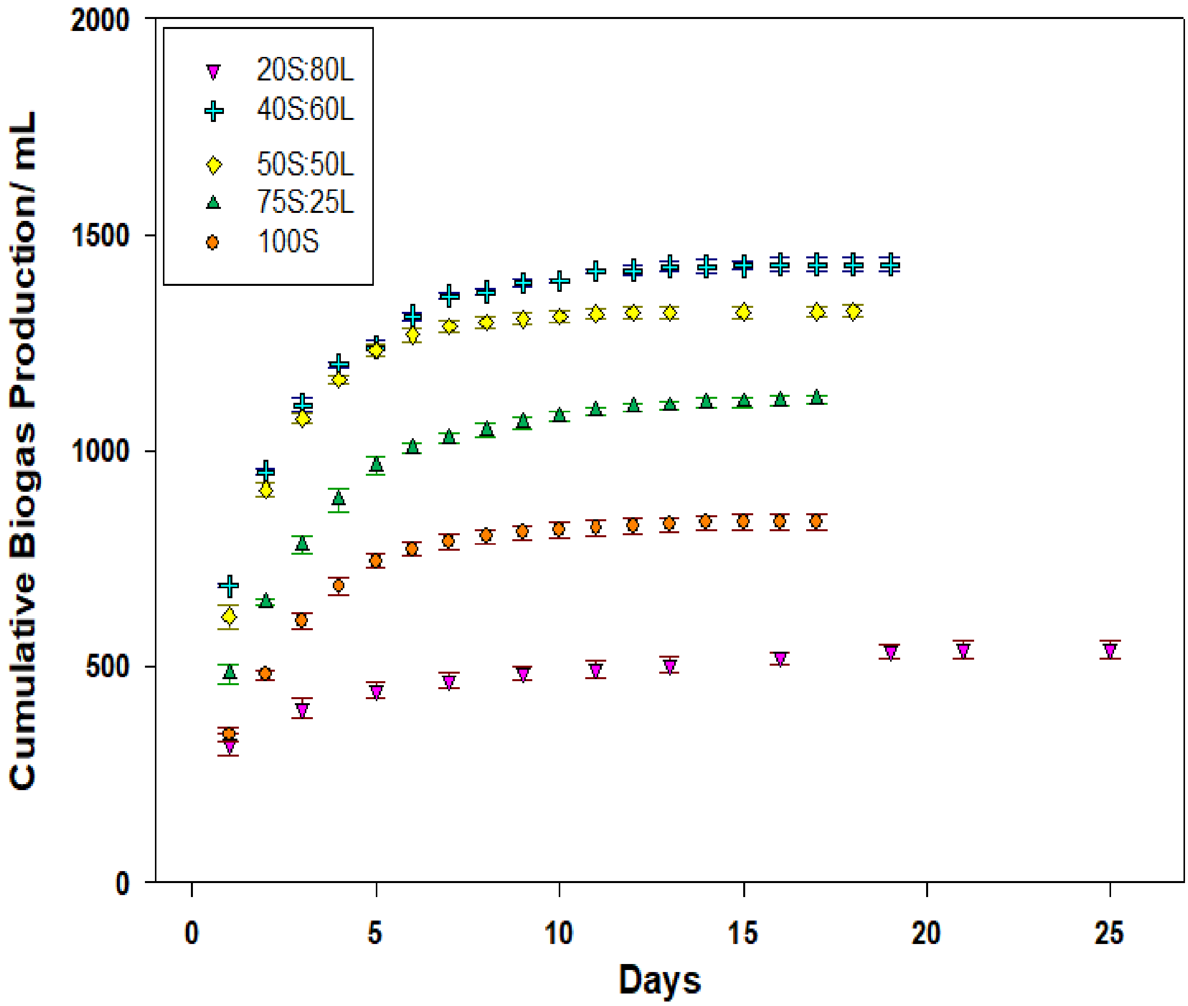

Figure 1: Combined cumulative biogas production graphs for untreated POME at different solid: liquid ratios

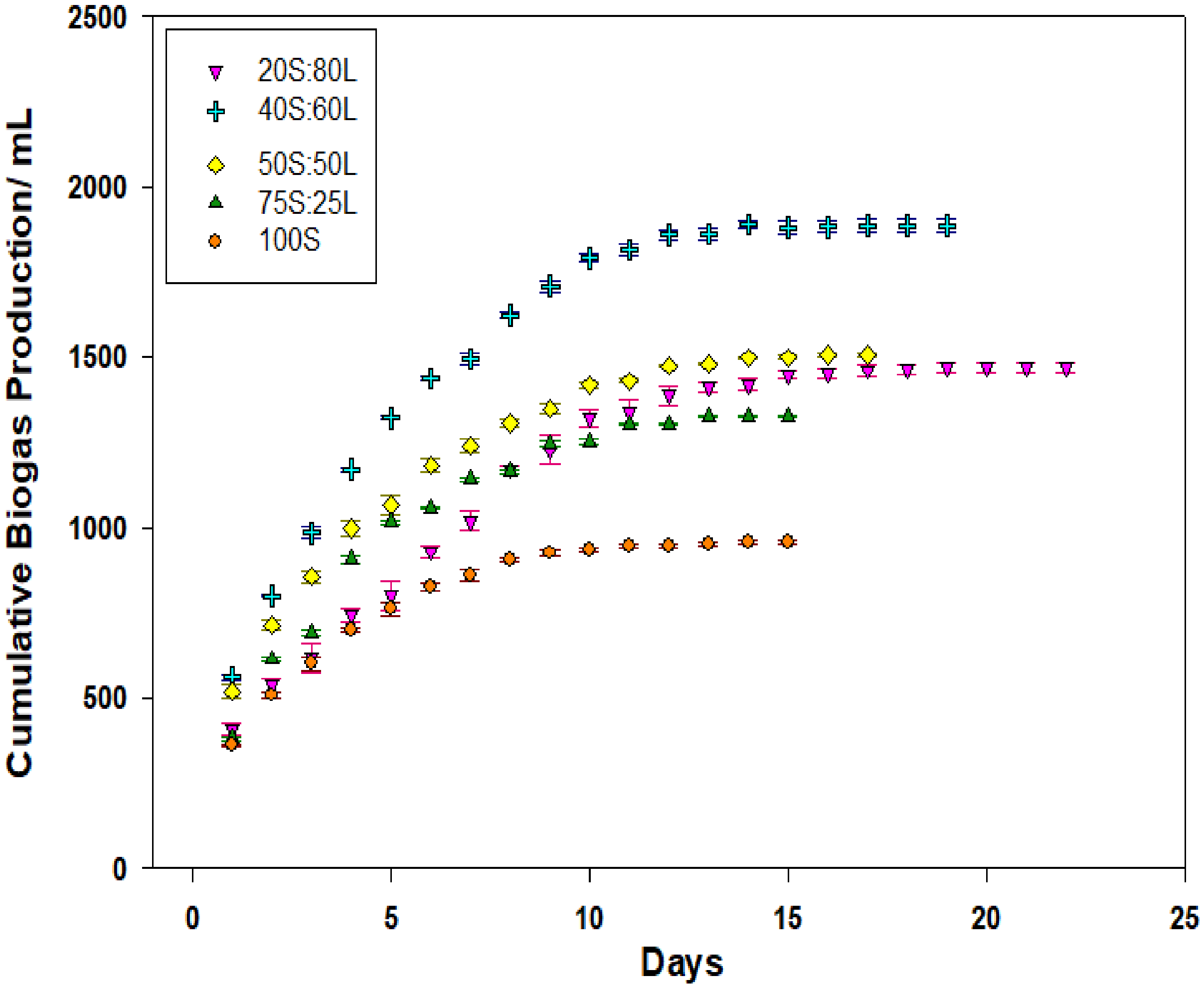

Figure 2: Combined cumulative biogas production graphs for thermally treated POME at different solid: liquid ratios

Table 1: Results obtained for the thermophilic anaerobic digestion of untreated POME at different solid loadings

Ratios	Dry Solids Content/ %TS	Initial pH	Final pH	Cumulative Biogas Production/ mL	Maximum Methane Composition /%	Minimum Methane Composition /%	H_2S composition/ mg/L	Total Anaerobes/ 100mL	Duration of Experiment /days	COD removal /%	BOD removal /%	TSS removal /%	O&G removal /%
20S:80L	3.29	7.23 ±0.05	7.52 ±0.03	539.44 ±10.29	73.83 ±2.42	50.36 ±2.07	204±4	$1.2x10^{6}$	≈25	62.53 ±1.14	58.07 ±1.76	55.44 ±3.46	26.20 ±0.46
40S:60L	4.02	7.21 ± 0.02	7.74 ±0.09	1431.67 ±17.56	77.33 ± 1.20	57.80 ±2.67	341±16	$2.1x10^{6}$	≈20	48.89 ±1.12	32.64 ±1.66	39.57 ±2.26	25.05 ±0.62
50S:50L	5.25	7.22 ±0.12	7.56 ±0.04	1322.78 ±13.62	64.26 ±2.71	54.13 ±0.87	560±21	$7.5x10^{5}$	≈18	33.26 ±0.71	23.97 ±1.30	29.40 ±1.55	19.24 ±0.45
75S:25L	6.4	7.27 ±0.10	7.44 ±0.04	1122.70 ±9.94	57.73 ±1.62	40.67 ±0.58	1823±23	$1.5x10^{5}$	≈17	23.11 ±0.41	24.10 ±0.52	20.76 ±0.57	26.57 ±0.18
100S	7.86	7.23 ±0.07	7.75 ±0.07	833.88 ±17.11	33.17 ±1.30	20.87 ±3.04	2968±52	$9.3x10^{4}$	≈17	7.67 ±1.05	21.78 ±1.84	19.06 ±2.85	31.16 ±0.62

Table 2: Results obtained for the thermophilic anaerobic digestion of thermally pretreated POME at different solid loadings

Ratios	Dry Solids Content/ %TS	Initial pH	Final pH	Cumulative Biogas Production/ mL	Maximum Methane Composition/ %	Minimum Methane Composition / %	H_2S composition/ mg/L	Total Anaerobes/ 100mL	Duration of Experiment /days	COD removal /%	BOD removal /%	TSS removal /%	O&G removal /%
20S:80L	2.88	7.23 ±0.04	7.53 ±0.02	1471.10 ±15.23	79.23 ±1.34	71.30 ±2.71	176±8	$1.5x10^{6}$	≈ 22	84.50 ±1.01	84.41 ±0.15	83.03 ±0.91	82.88 ±0.34
40S:60L	3.98	7.27 ±0.02	7.57 ±0.07	1886.11 ±21.63	83.40 ±0.31	78.83 ±1.31	256±21	$4.6x10^{6}$	≈ 19	81.63 ±0.46	81.01 ±1.16	80.72 ±0.16	80.02 ±0.11
50S:50L	5.14	7.30 ±0.05	7.48 ±0.02	1509.43 ±4.43	76.97 ±0.73	71.40 ±0.79	392±29	$1.1x10^{6}$	≈ 17	65.38 ±0.04	62.72 ±0.36	67.20 ±0.75	64.81 ±0.40
75S:25L	6.32	7.20 ±0.02	7.71 ±0.06	1326.13 ±4.74	51.97 ±2.03	43.00 ±2.35	1503±35	$2.4x10^{5}$	≈ 15	51.51 ±1.62	50.88 ±0.56	53.32 ±0.36	50.56 ±1.12
100S:0L	7.29	7.22 ±0.01	7.58 ±0.06	970.00 ±2.89	38.20 ±0.75	23.24 ± 1.25	2745±68	$1.5x10^{5}$	≈ 15	41.40 ±1.39	40.12 ±2.16	40.59 ±1.84	50.65 ±1.54

Figures 3 and 4 depict the methane composition of the biogas produced during the untreated and the thermally treated anaerobic digestion of POME at different solid loadings, respectively. In Figure 4 below compared to Figure 3, it can be noted that the methane composition in the biogas produced is at a higher percentage from day 1 in the treated sample whereas it takes longer time for methane conversion to take place in the untreated sample except for 100S solid loading which generated 15.3% of the methane from day 1. An explanation for the faster methane conversion which occurs in the treated ratios is owing to the organic matter being readily available for the methanogens to break down and convert to methane.

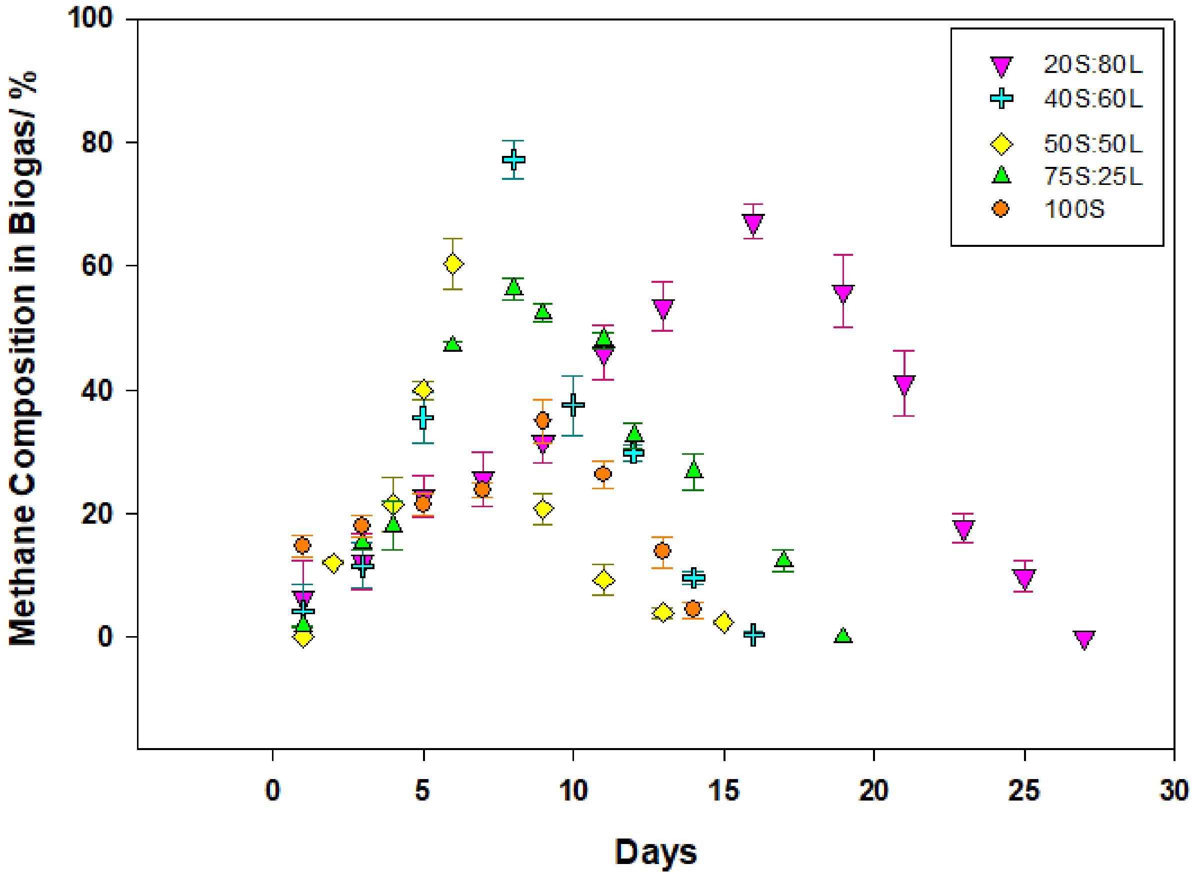

Figure 3: Combined daily methane composition graphs for Untreated POME at different solid: liquid ratios

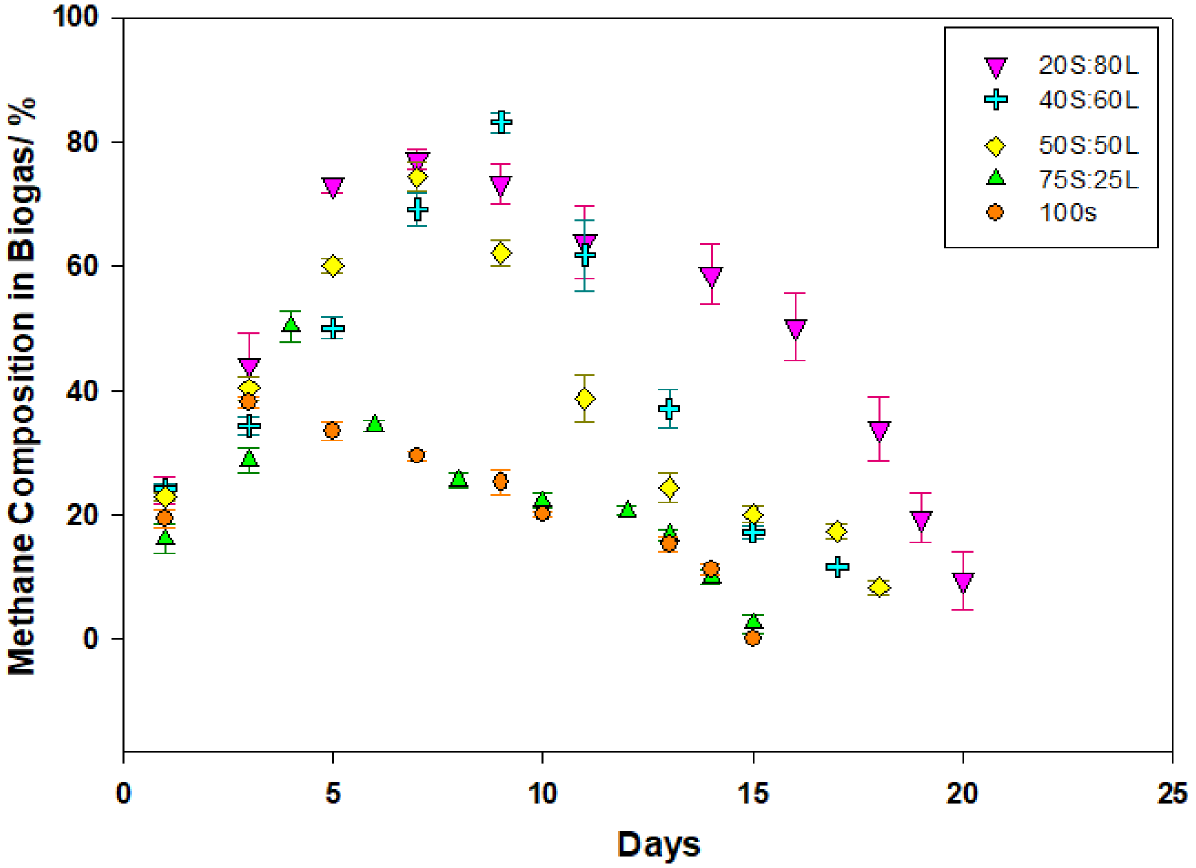

Figure 4: Combined daily methane composition graphs for thermally treated solid loadings

After thermal pretreatment, it can be observed that the initial COD, BOD, TSS, and O&G for all the conditions are less compared to the non-treated assays indicating that thermal pretreatment by itself can successfully reduce the COD, BOD, TSS and O&G content of POME. For example, the initial COD, BOD, TSS, and O&G for the untreated 20S:80L solid loading were recorded to be 37600±190, 16350±152, 9500±100, and 268±23 mg/L, respectively whereas the initial COD, BOD, TSS, and O&G for the thermally treated 20S:80L solid loading was found to be 30300±125, 12740±190, 11000±200 and 198±20 mg/L, respectively which is comparatively lower than the untreated sample (Pilli et al., 2015a, 2015b).

3.1 Chemical properties analyses in terms of COD, BOD, TSS, and O&G removal

The chemical properties experiments were undertaken, as described in section 2.3. Nine sets of data were used to evaluate the chemical characteristics discussed below. The chemical properties of the untreated 20S:80L condition prior to anaerobic digestion in terms of COD, BOD, TSS, and O&G were 37600±190, 16350±152, 9500±100, and 268±23 mg/L, respectively; while, the COD, BOD, TSS, and O&G of the untreated 20S:80L after anaerobic digestion was found to be 14088±430, 6855±287, 4233±328 and 197±1 mg/L. Alternatively, after anaerobic digestion of the thermally treated 20S:80L ratio, the COD, BOD, TSS, and O&G significantly declined to 4696±305, 1986±150, 1866±100 and 34±1 resulting in prominent percentage removal of 84.50±1.01, 84.41±0.15, 83.03±0.91 and 82.88±0.31 % of COD, BOD, TSS, and O&G respectively. The treated 20S:80L has 21.97, 26.34,27.59, and 56.68 % higher removal efficiencies of COD, BOD, TSS, and O&G than the nontreated 20S:80L ratio congruently.

The COD, BOD, TSS, and O&G evaluated prior to anaerobic digestion for the untreated 40S:60L condition were 47700±120, 20740±138, 16200±113, and 279±13 mg/L, respectively and after anaerobic digestion of the untreated 40S:60L, the COD, BOD, TSS, and O&G was reduced to 24377±537, 13696±345, 9788±366 and 209±2 mg/L. Contrarywise, the initial COD, BOD, TSS, and O&G for pretreated 40S:60L solid loading were 40800±100, 20090±130, 16000±150 and 208±12 mg/L respectively. Post anaerobic digestion the COD, BOD, TSS, and O&G drastically decreased to 8155±44, 4015±67, 3162±65 and 42±1 achieving a notable removal percentage of 80.63±0.46, 81.01±1.16, 80.72±0.16 and 80.02±0.11 % of COD, BOD, TSS, and O&G respectively. The treated 40S:60L gave rise to 32.74, 48.37, 41.15, and 54.97 higher removal efficiency of COD, BOD, TSS, and O&G compared to the untreated 40S:60L samples. Though the COD, BOD, TSS, and O&G removal efficiency is similar to the treated 20S:80L solid loading, the increased biogas production and methane yield from the treated 40S:60L solid loading is highly advantageous. It was perceived that the anaerobic digestion experiment of the treated 40S:60L solid loading was even more stable than the treated 20S:80L solid loading which is indicated by the smaller calculated percentage deviation as shown in Table 2 in addition to all 9 runs exhibiting similar results in terms of chemical characteristics on top of biogas production. Figure 5 shows the comparison of the chemical properties between the untreated and pretreated solid: liquid ratios after anaerobic digestion. It can be seen in Figure 5 that the COD, BOD, TSS, and O&G after anaerobic digestion of the untreated assays are significantly higher than that of the pretreated ratios indicating that the effluent quality of the pretreated ratios is notably ameliorated.

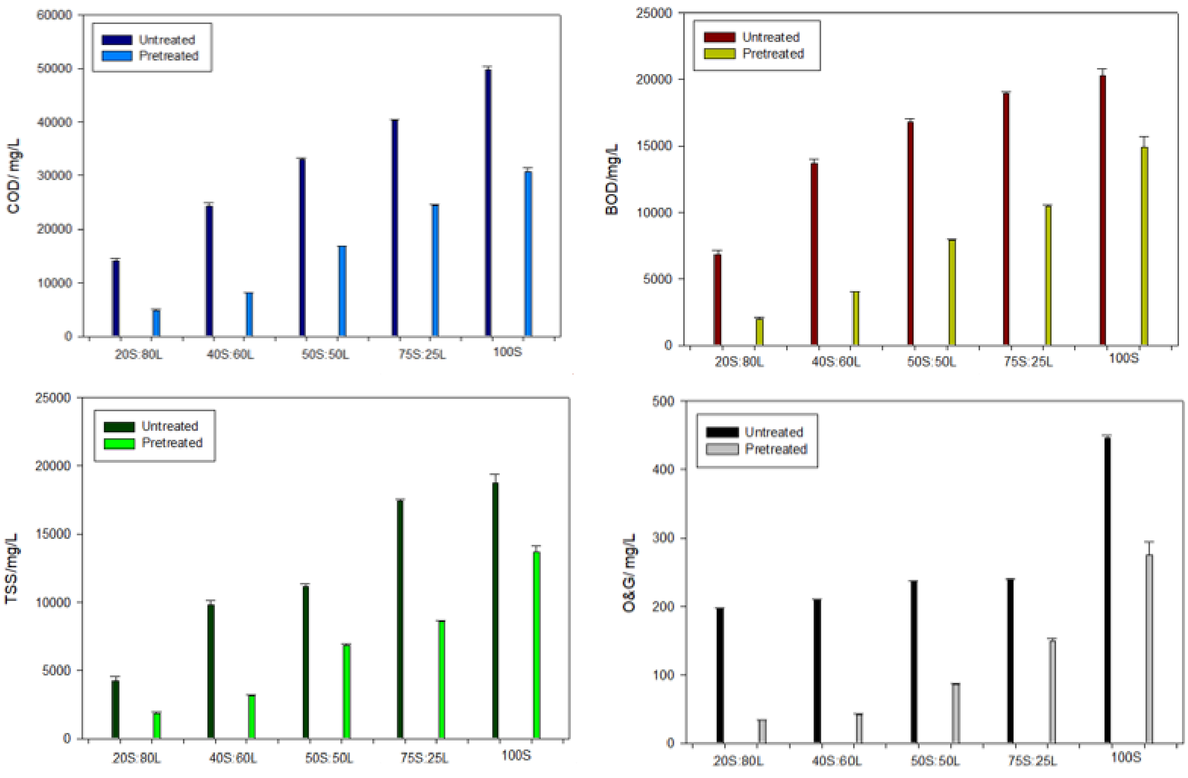

Figure 5: Comparison between the chemical properties of untreated and pretreated solid: liquid ratios after anaerobic digestion

As for the chemical properties analyses for the 50S:50L assays, before anaerobic digestion, the COD, BOD, TSS, and O&G for the nontreated 50S:50L ratio were 49500±210, 22098±195, 15800±115, and 294±25 mg/L, respectively while post anaerobic digestion, the recorded COD, BOD, TSS, and O&G were 33033±352, 16799±287, 11155±244 and 237±1 mg/L respectively. Conversely, the initial COD, BOD, TSS, and O&G for the pretreated 50S:50L solid loading were 48500±200, 21280±125, 20800±135, and 245±15 mg/L, respectively. While after anaerobic digestion the COD, BOD, TSS, and O&G was reduced to 16789±22, 7932±77, 6822±156 and 86±1 mg/L. The pretreated 50S:50L sample achieved a percentage removal of 65.38±0.04, 62.72±0.36, 67.20±0.75, and 64.81±0.40% of COD, BOD, TSS, and O&G, congruently. Compared to the untreated 50S:50L ratio, the pretreated counterpart attained 32.12, 38.75, 37.80, 45.57% better COD, BOD, TSS, and O&G removal. Although the removal efficiency of COD, BOD, TSS, and O&G decreased compared to treated 20S:80L and 40S:60L solid loadings, the removal efficiency of COD, BOD, TSS and O&G for treated 50S:50L solid loading is still superior in contrast to all the untreated tested conditions. It was established that the COD, BOD, TSS, and O&G removal for all the thermally pretreated conditions were much higher than that of the non-treated ones.

For the untreated 75S:25L chemical analyses, prior to anaerobic digestion, the COD, BOD, TSS, and O&G condition were 52400±145, 24950±230, 22000±162, and 322±35 mg/L, respectively; after anaerobic digestion, the COD, BOD, TSS, and O&G was reduced to 40289±212, 18959±129, 17433±126 and 236±1 mg/L. However, the initial COD, BOD, TSS, and O&G the pretreated 75S:25L ratio were 50600±210, 21327±125, 20500±105, and 305±10 mg/L, respectively. Whereas after anaerobic digestion the COD, BOD, TSS, and O&G declined to 24533±128, 10475±118, 8569±74 and 150±13 mg/L. The pretreated 75S:25L ratio brought about removal efficiencies of 51.51±1.62, 50.88±0.56, 53.32±0.36, and 50.56±1.12 % of COD, BOD, TSS, and O&G respectively. Consequently, the removal efficiencies of COD, BOD, TSS, and O&G for the treated 75S:25L assays were 28.40, 26.78, 32.56, 23.99% greater than the untreated corresponding ratio.

An important observation made in the nontreated samples for 75S:25L and the 100S loadings is that the O&G removal was higher than the 40S:60L and the 50S:50L. This occurrence can be explained owing to the formation of a layer of scum at the top of the 75S:25L and the 100S loadings where some of the O&G may have been lost in the scum layer (Khadaroo et al., 2020; Soares et al., 2019). The formation of the scum layer usually occurs in high fat, oil and grease content substracts whereby long-chain fatty acids cause digester instability; as a result, it is more challenging for the bacteria consortium to break down the lipids in the medium (Long et al., 2012; Martínez et al., 2011). On the other hand, for the 75S:25L and the 100S loadings pretreated samples, no scum layer was observed as the O&G was broken down by the thermal pretreatment before anaerobic digestion (Pilli et al., 2015a, 2015b). This incidence is seen in the O&G removal trend, whereby it steadily decreases from pretreated 20S:80L to 100S solid loadings.

Furthermore, the COD, BOD, TSS, and O&G recorded for the nontreated 100S solid loading before anaerobic digestion were 54000±250, 25975±125, 23100±190, and 649±32 mg/L, respectively; after anaerobic digestion, the COD, BOD, TSS, and O&G declined to 49856±568, 20315±478, 18711±658 and 446±4 mg/L. Alternatively, the initial COD, BOD, TSS, and O&G for thermally treated 100S solid loading were 48500±200, 21280±125, 20800±135, and 558±15 mg/L, respectively. While after anaerobic digestion the COD, BOD, TSS, and O&G decreased to 30766±692, 14906±788, 13722±425 and 275±20 mg/L. The pretreated 100S resulted in removal efficiencies of 41.40±1.39, 40.12±2.16, 40.59±1.84, and 50.65±1.54 % for COD, BOD, TSS, and O&G respectively. Accordingly, the removal efficiencies of COD, BOD, TSS, and O&G for the treated 100S was 33.73, 18.34, 21.53, and 19.40% enhanced in contrast to the untreated equivalent solid loading.

3.2 Biogas Production and Total number of anaerobes

The pretreated 20S:80L samples achieved a maximum biogas production of 1471.10 mL, which was 960 mL more than the untreated sample. A comparison in the performance of the thermally treated assays and the nontreated ones in terms of cumulative biogas production can be seen in Figure 6. Based on the results for the individual runs for pretreated 20S:80L solid loading, it was noted that the anaerobic digestion process was more stable than the untreated 20S:80L solid loading when comparing the percentage error as indicated in Table 1 and 2. The noticeable increase in biogas production is due to more intracellular matter available for the methanogens to convert to biogas as well as good substrate/microbes contact in the thermally treated samples. In contrast to the limited amount of solids present in the untreated 20S:20L solid loading medium, which are confined by the cell walls and making it further challenging for the bacteria to access the intracellular organic matter. An indication of the statement mentioned above whereby the microorganisms cannot thrive in such conditions is designated by the total number of anaerobes estimated in the untreated and thermally treated experiments as displayed in Tables 1 and 2 (Cappai et al., 2015; Pilli et al., 2015b). It can be clearly seen that the total number of anaerobes in the untreated sample is much less than that of the thermally pretreated assay. The longer anaerobic digestion duration can be explained by less solid and a smaller number of anaerobes present in the 20S:20L medium (Mishra et al., 2019; Rouches et al., 2019).

Comparable to the untreated 40S:60L solid loading, the pretreated 40S:60L solid loading brought forth the best results in comparison to the other pretreated conditions in regard to the total biogas production, the methane purity in the biogas produced, methane yield as well as the treated effluent quality. From Figure 2, it can be observed that the total biogas produced by the pretreated 40S:60L was 1886.11±21.63 mL, which accounts for 415.01 mL more biogas produced than pretreated 20S:80L solid loading. Contrarywise to the untreated 40S:60L ratio, the treated counterpart generated 454.44 mL more biogas. When comparing the 40S:60L of the non-treated POME to that of the treated one as shown in Figure 6, it can be noted that the amount of biogas produced in the pretreated experiments is substantially higher accounting for 456 mL more biogas generated than the untreated 40S:60L solid loading. The latter occurs owing to readily accessible organic matter since the cell membrane holding the latter is disintegrated when thermally pretreated. This occurrence in turns allows more bacteria to grow and thrive, the MPN values obtained for pretreated 40S:60L solid loading was evaluated to be $4.6x10^6$ total anaerobes/100mL while the bacteria count for the untreated 40S:60L solid loading was $2.1x10^6$ total anaerobes/100mL which is slightly less than half the number of bacteria present in the pretreated 40S:60L medium.

The anaerobic digestion duration for treated 50S:50L solid loading was determined to be around 19 days, which is even shorter than both the treated 20S:80L and 40S:60L solid loading along with all the untreated conditions. It can be observed that the maximum biogas produced by the pretreated 50S:50L solid loading was 1509.43±4.43 mL, which was approximately 186.71 mL more biogas produced than the untreated 50S:50L ratio and 377 mL less than the pretreated 40S:60L solid loading. Subsequently, the anaerobic digestion of the treated 50S:50L solid loading showed the removal efficiencies of COD, BOD, TSS, and O&G decreased compared to that of the pretreated 40S:60L solid loading. The total number of anaerobes present after anaerobic digestion in all the thermally treated conditions was higher than that of the untreated ones. The reduced time for anaerobic digestion can be due to the implementation of the thermal treatment, which coincides with numerous other studies (Carrere et al., 2016; Carrère et al., 2010). It can be noted that treated 20S:80L and 50S:50L solid loadings attained particularly close total biogas production but not regarding the percentage removal of COD, BOD, TSS, and O&G. Either condition can be made beneficial in terms of either biogas production or treated effluent quality, depending on the mills' prerequisites.

The anaerobic digestion duration for pretreated 75S:25L solid loading was observed to be shorter than the treated 50S:50L solid loading, which was around 15 days. The pH of the system ranged from 7.20±0.02 to 7.71±0.06. The cumulative biogas achieved from pretreated 75S:25L solid loading was 1326.13±4.74 mL; this is less biogas produced compared to pretreated 20S:80L, 40S: 60L and 50S:50L solid loadings but approximately 204 mL more biogas produced in contrast to the untreated 75S:25L solid loading. As for the total number of anaerobes, it was found that the untreated 75S:25L assay contained approximately $1.5\mathrm{x}10^5$ total anaerobes/100mL while the thermally pretreated sample yielded $2.4\mathrm{x}10^5$ total anaerobes/100mL. However, the recorded values of total anaerobes are significantly lower than those present in both treated and untreated 20S:80L, 40S:60L, and 50S:50L solid loadings.

The data obtained for 100S exhibited a similar trend to that of 75S:25L solid loading. The total volume of biogas produced by the treated 100S solid loading was obtained as 970.00±2.89 mL, which results in 137 mL more biogas than the untreated 100S solid loading. Another explanation for the reduction in biogas production at higher solid loadings is attributable to the decrease in water content and the associated least effective transport and mass transfer conditions whereby the microorganisms cannot thrive in an environment with limited soluble substrates, thus making the anaerobic digestion process less efficient (Le Hyaric et al., 2012; Xu et al., 2014; Zhang et al., 2018).

3.3 Methane Composition in the biogas produced

For the 20S:80L solid loading, it is worthy to note that compared to the untreated 20S:80L solid loading, which produced the maximum methane at day 15, as shown in Figure 3, the thermally treated assays produced maximum methane by day 6, which is much earlier than the former as shown in Figure 4. The incidence above is considerably more favorable as the amount of biogas produced in the earlier days is distinctly higher, thus generating more methane compared to after day 10. The result obtained from the anaerobic digestion of pretreated 20S:80L solid loading was remarkable in contrast to its non-treated counterpart. The maximum methane purity of the biogas in the pretreated samples reached up to 79.23±1.34%, which is slightly more than the untreated 20S:80L solid loading.

The purity of methane in the biogas produced by the thermally treated 40S:60L improved to 83.40±0.31% while the methane composition recorded for the untreated assay was 77.33± 1.20% when comparing the maximum methane purity. The purity of methane produced in the pretreated 40S:60L solid loading was approximately 13% higher than that of the untreated 40S:60L solid loading. It was also observed that at 24 hours after the setup of the experiment, the percentage of methane produced was twice the amount of that produced by the untreated sample. The rate of methane production was faster and more sustained for the thermally pretreated 40S:60L experiments in comparison to the untreated one.

Though the methane purity for treated 50S:50L solid loading was found to be 76.97±0.73%, the untreated counterpart methane composition was measured to be 64.26±2.70%. It was observed that the methane composition for pretreated 20S:80L and 50S:50L are especially close, which further shows that the anaerobic digestion process for POME can be customized as per the requirements of the mill.

The methane composition for the untreated 75S:25L sample was measured to be 57.73±1.62%, while the treated sample had a lower methane composition of 51.97±2.03%. A noteworthy observation distinguished in the pretreated 75S:25L solid loading was that the methane purity was moderately low, and the amount of hydrogen sulfide gas produced was excessively elevated, ranging from 1503 to 1823 mg/L for the treated and untreated samples as shown in Tables 1 and 2. This occurrence can be explained by the dissimilatory sulfate reduction, which is associated with the growth of sulfate-reducing bacteria (Choi et al., 2018). The sulfate-reducing bacteria utilizes organic matters and sulfate as electron donors and an electron acceptor, respectively. In anaerobic treatment, the methanogenesis can be monumentally influenced by the sulfide produced from sulfate reduction (Yan et al., 2018). A high concentration of free-hydrogen sulfide can lead to the inhibition of methanogenesis and, eventually, a deficient anaerobic digestion process (Mizuno et al., 1998).

The higher production of hydrogen sulfide gas and the decline in methane composition were also observed with the 100S solid loading. The H_2S composition was noted to be around 2745 and 2968 mg/L for the treated and untreated samples. The maximum methane purity obtained was further inhibited in both the treated and non-treated assays, the pretreated 100S sample was recorded to be 38.20±0.75% while that of the untreated counterpart was measured to be 33.17±1.30%.

3.4 Methane Yield

The methane yield is defined as the amount of methane generated for a specific amount of organic matter removed, and this is the consequence of the activity of the anaerobic consortium of bacteria. The methane yield is constant during steady-state conditions for a particular carbon substrate in anaerobic digestion conditions and is subjected to the fraction of the biodegradable matter and the characteristics of the compounds present in the medium. In other words, the methane yield is constant when the anaerobic ecosystem uses carbon for growth and conservation only. Theoretically, the methane yield of an anaerobic consortium is 350 mL CH_4/g $COD_{removed}$ when the anaerobic digestion is undertaken at room temperature. Reaching this value would indicate that the bacteria would have used all the carbon available for anaerobic digestion and growth (Michaud et al., 2002). However, the theoretical methane yield can be higher at thermophilic temperatures.

The methane yield calculated for the untreated and pretreated 20S:80L ratios were 36.20 and 313.18 mL CH_4/g $COD_{removed,}$ correspondingly, which brings about a substantial 9-fold increase in the methane generated. While the methane yield evaluated for the non-treated and the thermally pretreated 40S:60L ratios were 58.40 and 328.73 mL CH_4/g $COD_{removed}$, which is approximately 6-fold higher than the untreated assays. From Figure 3 and 4, it can be discerned that the maximum methane yield occurred between day 6 to day 8 for both the untreated and treated assays. It can be noted that the methane yield achieved for thermally pretreated 20S:80L and 40S:60L is relatively close to the theoretical value.

The methane yield for the treated assays was approximately 2-folds higher than the untreated 50S:50L assays. The total methane yield was evaluated to be 40.06 and 89.88 mL CH_4/g $COD_{removed}$ for the untreated and the thermally treated 50S:50L ratios, respectively. The methane yield for both the treated and the untreated 50S:50L solid loadings are, however, lower than that obtained for the pretreated 40S:60L solid loading.

The methane yield for the untreated and thermally pretreated 75S:25L ratios were 27.84 and 54.06 mL CH_4/g $COD_{removed}$, correspondingly, which is also 2-fold superior to the untreated 75S:25L solid loading. Whereas, the methane yield was computed to be 16.69 and 31.52 mL CH_4/g $COD_{removed}$ for the untreated and the treated 100S assays, correspondingly. An important observation is that the methane yield for thermally pretreated 50S:50L, 75S:25L, and 100S all produced approximately 2-folds more methane than their untreated counterparts.

The difference in the anaerobic digestion performance between the thermally treated and non-treated samples was prominent. It can be ascertained that the amount of biogas produced, and the methane yield was notably enhanced in comparison to the non-treated samples. The thermally pretreated conditions had more than twice better removal efficiencies when it comes to the COD, BOD, TSS, and O&G across all the different solids contents tested. Another consistent occurrence across all the thermally pretreated conditions was that the anaerobic digestion process of POME was more stable when pretreated compared to those of the untreated solid loadings. Based on Table 2, it can be seen that 40S:60L and 50S:50L ratios achieved a more stable anaerobic digestion process as the percentage error in the cumulative biogas is significantly lower than 20S:80L, 75S:25L, and 100S solid loadings. In other words, the biogas production for all 9 assays for the ratios mentioned above is comparatively consistent. The above-described observation is in agreement with Farhat et al. (2018) study on thermally pretreated municipal sewage sludge and Toutian et al. (2020) investigation on the effect of temperature on the biogas yield of thermally hydrolyzed waste activated sludge.

The present study allows a means of control not only on the anaerobic digesters' load but also on the results sought to be achieved. For example, if the industry aims to produce more biogas with a high methane purity for steady electricity generation, in which case, it is more favorable to use lower solid loadings. However, if the mill prefers to achieve a better-treated effluent quality such that the treated solids can be used as fertilizers in the plantations, then a higher solid loading is more suitable. Whereas for the treated liquid to conform to the environmental standards to be discharged in watercourses, lower solid loadings are more favorable. In a context where all the scenarios mentioned above need to be fulfilled, then using a solid loading close to 40S:60L will aid in attaining the desired outcomes.

With approximately 80% removal of COD, BOD, TSS, and O&G, the treated effluent will require less time in the secondary treatment, which is the aeration treatment to sufficiently remove the remaining COD, BOD, TSS, and O&G from the effluent. As the aeration treatment is known to be a high energy consumption process, the lesser the time for which the effluent needs to be aerated, the higher the savings for the electrical energy required for the continuous supply of oxygen. The substantial reduction in energy consumption is a primary advantage when it comes to utility cost and hygienisation (Ahmad and Krimly, 2014; Khadaroo et al., 2019b).

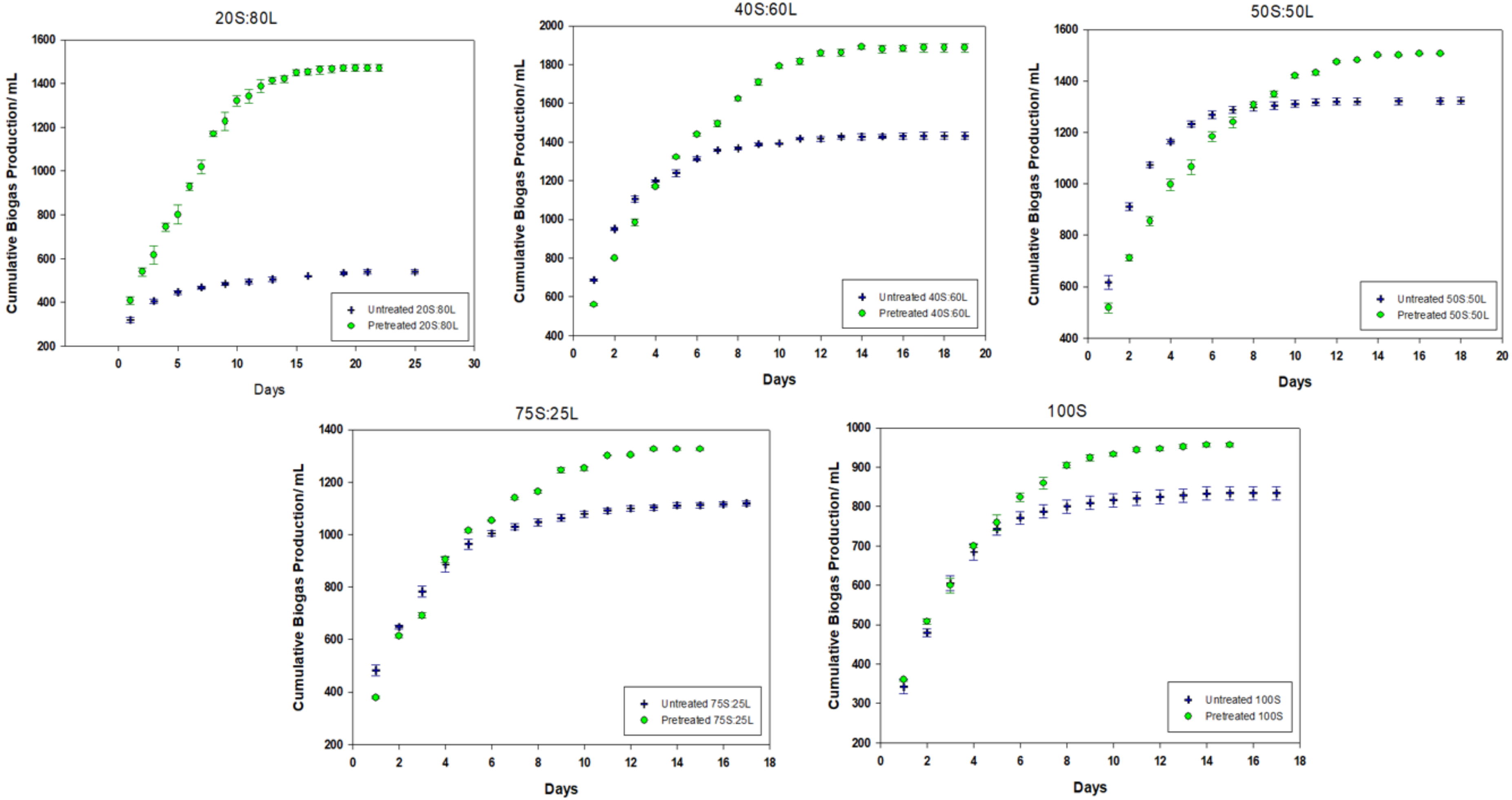

Figure 6: Comparison between thermally pretreated and untreated POME cumulative biogas production graphs

4. Conclusion

Thermally pretreated POME achieved better biogas production, a higher purity along with methane yield, and the removal efficiencies for COD, BOD, TSS, and O&G are extensively superior compared to the non-treated POME. The best performing condition was established to be the thermally treated 40S:60L solid loading, which produced a maximum biogas production of 1886.11±21.63 mL, whereby the methane composition was measured to be 83.40±0.31%. The methane yield calculated for the thermally treated 40S:60L solid loading was 328.73 mL CH_4/g $COD_{removed}$. As for the thermally treated 40S:60L, removal efficiencies in terms of COD, BOD, TSS, and O&G achieved was 80.63±0.46, 81.01±1.16, 80.72±0.16, and 80.02±0.11% respectively. The pretreated POME provides a better treated effluent quality such that when the treated effluent is sent for the secondary treatment, the latter can effectively further reduce the COD, BOD, TSS, and O&G so that the treated effluent conforms to the stipulated environmental standards. This study on pretreated solid loadings demonstrates that the complex anaerobic digestion process can be made more versatile and customizable based on the desired attainable results by allowing a means of control on the digesters' load. Implementing the thermal pretreatment and a solid-liquid separation step in the process shows remarkable potential in improving the treatment process of POME, all while producing a significantly higher amount of biogas with an enhanced methane composition and improving the treated effluent quality making the process sustainable and cleaner since it also notably reduces the environmental encumbrance by allowing the use of the treated effluent as fertilizers.

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Nomenclature

APHA	American Public Health Association
ASTM	American Society for Testing Material
BOD	Biological Oxygen Demand
COD	Chemical Oxygen Demand
CPO	Crude Palm Oil
L	Liquid
MPN	Most Probable Number
O&G	Oil and Grease
POME	Palm Oil Mill Effluent
S	Solid
TS	Total Solids
TSS	Total Suspended Solids
USEPA	United States Environmental Protection Agency
VS	Volatile Solids

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