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Mechanical pretreatment processes for enhancement of biogas production from palm oil mill effluent (POME)

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ABSTRACT

Various pretreatment methods including ultrasonication, coalescer filtration, and dissolved air flotation (DAF) have been investigated for palm oil mill effluent (POME) treatment prior to the biogas system. These methods showed significant improvement in the biodegradability characteristics of POME in comparison with non-pretreatment. Their efficiencies were determined by measuring the biochemical oxygen demand (BOD)/ total chemical oxygen demand (TCOD) ratio, soluble chemical oxygen demand (SCOD)/TCOD ratio, and reducing sugar content, as well as TCOD removal, fat, oil and grease removal, and biomethane production. Ultrasonication, coalescer, and DAF increases oil removal efficiencies by 50.30%, 53.13%, and 68.21%, respectively. The results found that the BOD/ TCOD and SCOD/TCOD values for POME were increased slightly by pretreatment methods, but remained within the range for biodegradable values. While the coalescer and DAF increased the reducing sugar content in POME from 4.36 to 4.80 g/L and 4.36 to 4.40 g/L, respectively, sonication can increase it by nearly double, from 4.36 to 8.12 g/L. Maximum methane production was 3.05 LCH_4/L POME and a methane production rate of 88.52 LCH_4/day was also found with ultrasonication.

Keywords: Palm oil mill effluent; Ultrasonication; Coalescer; Dissolved air flotation; Biogas

1. Introduction

Palm oil mill effluent (POME) is a by-product generated from the production of crude palm oil (CPO). Palm oil mills typically produce an average of 2.5 tonnes of raw POME for every ton of fresh fruit bunches they process [1]. POME is thick, brownish, viscous liquid waste with an unpleasant odour. It has high levels of colloidal suspended matter [2], oil and grease, biochemical oxygen demand (BOD_5), and chemical oxygen demand (COD) [3], which may cause water pollution in surrounding environments. In addition, the large volume of POME that is generated during CPO processing and the properties that contain high levels of pollution force the palm oil industry to shoulder an enormous responsibility in facing the problem in terms of environmental protection, economic viability, and sustainable development [4]. The wastewater treatment system is among the most important facilities for palm oil mills to reduce pollution and prevent environmental damage. Due to its chemical and physical properties, an anaerobic pond system is typically used as the initial stage of POME treatment. The most important by-products obtained from this treatment process are biogas and methane. At present, palm oil mills operate an anaerobic digestion system for both pollution reduction and the production of biogas, which can be utilised as fuel for an internal combustion engine producing electricity for offices, labour houses, and biogas plants [3,5]. Therefore, POME has

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become a promising source for the biogas production and can increase the potential of the renewable energy sector [6].

The anaerobic digestion process is comprised of four major microbiological degradation steps including hydrolysis, acidogenesis, acetogenesis, and methanogenesis [7]. A disadvantage of the anaerobic digestion technique is slow degradation or the hydrolysis of microorganisms, which is the primary degradation step in the anaerobic digestion process that limits the rate of the entire anaerobic digestion process [8]. In order to intensify biogas production, various pretreatment techniques of the substrate were found to enhance hydrolysis and anaerobic digestion performance. Several have been investigated widely such as thermal, chemical, mechanical, thermochemical, and biological pretreatment, resulting in an enhanced rate of hydrolysis and biodegradation [9]. Pretreatment prior to the anaerobic process requires techniques that accelerate the hydrolysis step and shorten the period for operation to the limit of biological pretreatment techniques [10,11].

This research investigates physicochemical pretreatment processes including ultrasonication, coalescer (Co), and dissolved air flotation (DAF) in order to enhance biogas production from POME. Although these processes consume a high amount of energy for operation, there is an exception for palm oil mills, which can generate power from biogas itself. The increased production of biogas could support internal use as well as be sold to the Provincial Electricity Authority. In addition, this alternative approach would also recover oil residue before biogas production. The recoverable oil could be returned to the CPO process for the production of grade B oil palm. This would be beneficial to palm oil mills while simultaneously treating the wastewater, which could mitigate environmental concerns in nearby communities [12]. Ultrasonic disintegration is an emerging and very useful mechanical method that contributes to property enhancement of the biomass for anaerobic digestion [13]. Ultrasonication (US) has been shown to disintegrate sludge particles, destroy biomasses, and create favourable conditions for biodegradation. As a consequence, it may also enhance biogas production. The process efficiency of ultrasonic disintegration is mainly affected by three factors: ultrasonic frequencies, ultrasound energy levels, and sludge characteristics [14]. It has been reported that methane production by anaerobic digestion of waste activated sludge increases by 64% over 30 min of sonicated pretreatment. Meanwhile, the Co and DAF can also enhance the separation phase of oil from water. The Co has been reported to have high efficiency in treating oily wastewater [15]. It also improves the efficiency of the skim oil treatment process by providing condensation of small oil drops on the surface of the water. Hence, these processes could be an alternative approach for oil recovery in POME.

The main objective of this research was to investigate the effect of different pretreatment processes on the biomethane potential (BMP) of pretreated POME. The BMP tests were performed on a laboratory scale. Among the available experimental methods, BMP testing has been the most successful for describing and evaluating digestion performance [16,17]. They are operated as a batch condition, measuring the maximum amount of biogas or biomethane produced per gram of volatile solids (VS) contained in the organics that are used as substrates [18]. As there is no standard protocol for BMP testing, the most common conditions used in the literature were applied [19]. The results were analysed for POME characteristic change after pretreatment and trend in order to improve biogas production efficiency of POME using the various pretreatment techniques described previously.

2. Materials and methods

2.1. POME and sludge samples

2.1.1. Sample collection and characterisation

POME was collected from an acid pond (before anaerobic pond) of an industrial palm oil wastewater treatment plant belonging to Pitak Palm Oil Co., Ltd. in Trang Province, Thailand and brought back to the laboratory for analysis. It was kept in a refrigerator at 4°C until use. COD or total chemical oxygen demand (TCOD), BOD_5, suspended solid (SS), total solids (TS), VS, fat, oil and grease (FOG), and pH were determined. The analysis was done according to the procedures outlined in standard methods [20]. The amount of total reducing sugar extracted from the wastewater was determined using the 3,5-dinitrosalicyclic acid method [21]. The American Dye Manufacturers Institute (ADMI) unit colour was determined according to the availability on the Model DR 6000 UV–Vis spectrophotometer (Method 10048).

The inoculum was anaerobic sludge from anaerobic digesters (at sludge retention time [SRT] of 15 d) of the same manufacturer. It was used as seed sludge for methane fermentation and kept in a refrigerator at 4°C until use.

2.1.2. Sample preparation

POME sampling underwent natural sedimentation (SE) for the removal of FOG above the layer of the sample, which is similar to a conventional palm oil mill operation. After that, three types of pretreatment processes were applied to pretreat the POME.

2.2. Pretreatment of POME

The conditions for pretreatment processes used in this research were selected from optimum conditions obtained from preliminary testing, which are described in the following sections.

2.2.1. Ultrasonication

The wastewater sample was pretreated with an ultrasonic processer (Cole Parmer ultrasonic processor VC750 model, USA). It was operated with a power supply of 750 W and operating frequency of 20 kHz with a 1-inch probe submerged 2 cm below the surface of the water. One litre of the sample was sonicated at a power input of 0.04 W/mL for 30 min.

2.2.2. Coalescer pretreatment

The Co reactor used was a vertical column with 15 cm of diameter, 50 cm of height, and 8 L of volume operated in a continuous mode at 266 mL/min for 30 min. The filter media packed in the column was polyethylene terephthalate in fibre forms.

2.2.3. Dissolved air flotation pretreatment

DAF was operated with a pressure vessel recycled by 1 L of stream pressurised under a 4-bar pressure gauge. The retention time used was 5 min and recycle rate by 20% of influent. The efficiency of oil separation was evaluated by determining the amount of oil remaining in the under-liquid layer. The TCOD and FOG removal efficiencies were calculated with Eqs. (1) and (2), respectively [22]:

$$\text{TCOD removal \%} = \frac{\text{TCOD}_i - \text{TCOD}_f}{\text{TCOD}_i} \times 100 \quad (1)$$

$$\text{FOG removal \%} = \frac{\text{FOG}_i - \text{FOG}_f}{\text{FOG}_i} \times 100 \quad (2)$$

where TCOD_i was initial total TCOD, TCOD_f was TCOD after pretreatment, FOG_i was initial oil and grease, and FOG_f was oil and grease after pretreatment.

2.3. Biochemical methane potential (BMP) assay

Biogas production from POME was investigated in a 250 mL digester with a working volume of 200 mL operated at a temperature of 35°C, which was operated by a thermostatically controlled electronic heated water bath. Each digester was operated for a period of 45 days. Due to their low pH, sodium hydroxide was added to the feed stocks before feeding the digester in order to initiate pH adjustment to 7.0 ± 0.5. The mixing was always done using a magnetic stirrer. The digesters were fed by organic loading rate up to 2 g $\text{VS}_{\text{substrate}}$ g^{-1} $\text{VS}_{\text{inoculum}}$ [19]. The digestion course of inoculums was done in parallel as a control experiment. Biogas was collected and measured by water displacement using a gas counter once a day at zero gauge pressure and ambient temperature.

The modified Gompertz equation was used to study cumulative methane production, as in previous researches [16,18]. The Gompertz equation can be expressed as:

$$M_p = P_m \exp\left[-\exp\left\{\frac{R_m}{P_m}e(x_0 - x) + 1\right\}\right] \quad (3)$$

where M_p is cumulative methane production (mL); P_m is ultimate methane production (mL); R_m is methane production rate (mL/day); x_0 is lag-phase time (days); and e is exponential 1 [23].

2.4. Analytical methods

2.4.1. Pretreated POME and biogas characteristics

The TS, VS, total suspended solids, volatile suspended solids, TCOD, SCOD (soluble chemical oxygen demand), BOD_5, pH, and grease and oil content were analysed according to the standard methods [20]. Methane and carbon dioxide concentration in the biogas were measured using a gas chromatograph equipped with a thermal conductivity detector and helium serving as the carrier gas. The injector, detector, and oven temperatures were 150°C, 200°C, and 120°C, respectively.

2.4.2. Fourier transforms infrared spectroscopy analysis

A spectrum one Fourier transforms infrared spectroscopy (FTIR) spectrometer (EQUINOX 55, BRUKER) equipped with a KBr beam splitter and a deuterated triglycine sulphate detector was used to quantify the chemical bonds between the functional group and POME structure. Spectra were recorded from 500 to 4,000 cm^{-1} at a resolution of 4 cm^{-1}. A zero-filling factor was applied to give a final resolution of one point per wave number.

3. Results and discussion

3.1. POME, pre-treated POME characteristics and structure

POME used for the experiment contained high concentrations of TCOD (98.5 g/L) and TS (70.14 g/L). Other characteristics of the raw POME before pretreatment and pretreated POME is presented in Table 1. The data presented in Table 1 indicate that the amount of SS was substantially reduced after pretreatment, especially SE + DAF pretreatment. Fig. 1 shows the SE + US, SE + DAF, and SE + Co increase in oil removal efficiency and slight decrease for TCOD value in the wastewater. The percentage of oil removal was 50.30, 62.21, and 53.13, respectively, while percentage of TCOD removal was 15.1, 22.39, and 18.56, respectively. The oil removed could be recovered and returned to the process for lower-grade oil production. In this study, all experiments showed the pretreatment methods could qualify TCOD for oil and grease to have good value for biogas production. The recommended initial value of wastewater characteristics for biogas production is not over 8.0 g/L for oil and grease and 50–80 g/L for TCOD [24]. Although the oil content in any substrate can be digested to produce methane, the intermediate products of oil decomposition can inhibit methane production. For example, the degradation of fat will increase the concentration of fatty acids, which limits further biodegradation by methanogens [25]. Meanwhile, these processes can partially remove SS from POME. The percentage of SS removal efficiencies was 20.30, 88.91, and 38.89 for SE + US, SE + DAF, and SE + Co, respectively.

Fig. 2 illustrates the FTIR spectra of pretreated POME. Peaks are shown at wave numbers of 3,410, 2,924, 2,853, 1,615, 1,419, 1,114, and 1,048 cm^{-1}. For organic substances, the band at 3,600–3,200 cm^{-1} normally corresponds to O–H group [26] in accordance with the band appearing at peak 3,410 cm^{-1}. The bands located at 2,924 and 2,853 cm^{-1} are referred to as hydrogen vibrations of the C–H group for aliphatic methylene group [27] and were expected to be generated from the degradation of hemicellulose. The hemicellulose is usually more easily degraded because the structures are made of weak matrix polysaccharides and its structure contains mainly monomer sugar [28]. The absorption band at 1,615 cm^{-1} might come from the double bond C=C. For any absorption bands that are detected in the range of 1,000–1,200 cm^{-1}, they represent the hydroxyl and phosphate group. There were bands appearing at peaks 1,114 and 1,048 cm^{-1} in the POME sample. These bands are regarded as characteristics of lipids and phospholipids [29]. The FTIR spectra of POME showed a strong similarity to the main absorption bands in the regions of 1,000–4,000 cm^{-1}. Similar IR spectra were obtained from the analysis of oil palm decanter cake and oil palm empty fruit bunches as well [30,31].

Table 1
Characteristics of raw and pretreated POME

Parameter	Raw POME	Pretreated POME			
		SE	SE + US	SE + DAF	SE + Co
pH	4.85–5.65	5.43 ± 0.50	5.03 ± 0.50	4.25 ± 0.50	5.45 ± 0.50
TS (g/L)	70.14 ± 0.30	42.01 ± 0.10	41.07 ± 0.10	11.98 ± 0.10	32.71 ± 0.10
VS (g/L)	49.10 ± 0.20	29.31 ± 0.10	28.75 ± 0.10	8.42 ± 0.10	23.10 ± 0.10
SS (g/L)	21.74 ± 0.20	12.78 ± 0.10	10.18 ± 0.10	1.41 ± 0.10	7.81 ± 0.10
VSS (g/L)	15.22 ± 0.10	8.97 ± 0.10	7.20 ± 0.10	0.93 ± 0.10	5.67 ± 0.10
Oil and grease (g/L)	10.16 ± 0.10	8.27 ± 0.05	5.05 ± 0.05	0.18 ± 0.05	4.76 ± 0.05
TCOD (g/L)	98.52 ± 0.10	88.08 ± 0.20	83.64 ± 0.20	29.88 ± 0.20	80.23 ± 0.20
SCOD (g/L)	19.53 ± 0.20	21.42 ± 0.20	21.44 ± 0.20	19.57 ± 0.20	20.60 ± 0.20
BOD_5 (g/L)	52.48 ± 0.30	49.02 ± 0.20	50.19 ± 0.20	49.02 ± 0.20	45.00 ± 0.20
Reducing sugar (g/L)	4.36 ± 0.02	4.30 ± 0.02	8.12 ± 0.02	4.40 ± 0.20	4.80 ± 0.20
Colour (ADMI)	3,250 ± 10.00	2,989 ± 9.00	2,506 ± 7.00	2,520 ± 8.00	2,515 ± 7.00

Remark: SE – natural sedimentation POME, SE + US – natural sedimentation and ultrasonication, SE + DAF – natural sedimentation and dissolved air flotation, SE + Co – natural sedimentation and coalescer.

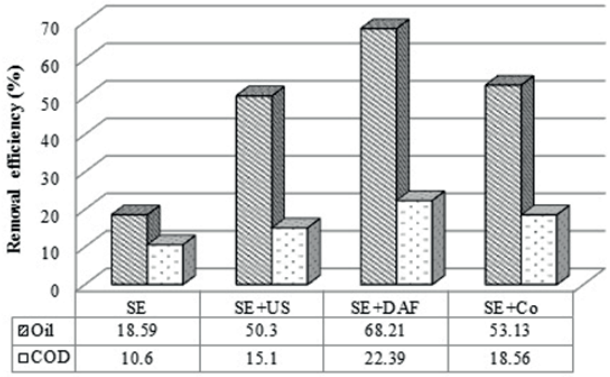

Fig. 1. Oil and COD removal efficiencies by different pretreatment POME.

After pretreatment and thorough anaerobic digestion processing, the change of the peaks position could not be observed. The reason is that the SE + DAF, SE + CO, and SE + US did not relate with any chemical reaction of the functional groups. However, SE + US showed breaking bonds between the sugar monomers in the polymeric chains formed by the hemicelluloses and the cellulose in POME. However, they are in the same functional groups. Therefore, the peak positions did not change.

3.2. Biodegradability after pretreatment

3.2.1. Reducing sugar release

The increase of reducing sugar content can also increase substrate digestibility. Ultrasonication can double the time for reducing sugar concentration (from 4.36 to 8.12 g/L), while Co and DAF slightly increases the reducing sugar concentration from 4.36 to 4.80 g/L and 4.36 to 4.40 g/L, respectively (Fig. 3). This is consistent with the experimental results of FTIR analysis and confirms that

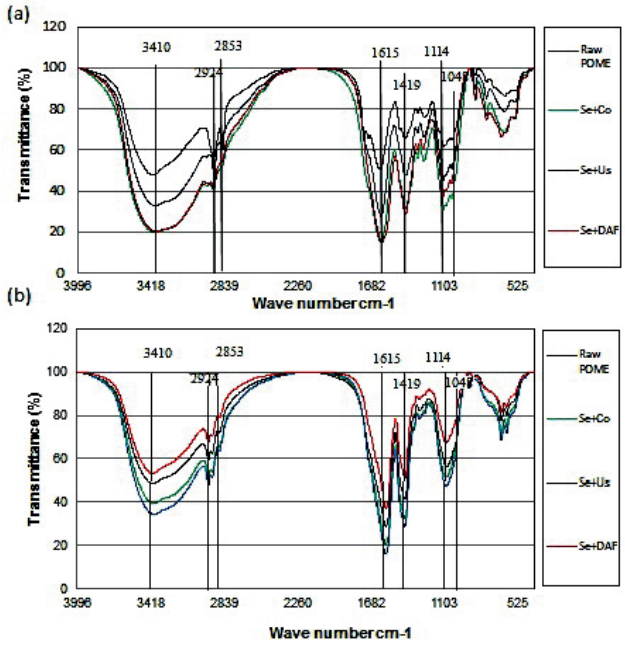

Fig. 2. FTIR spectra of POME with different pretreatment: (a) before anaerobic digestion and (b) after anaerobic digestion.

the ultrasonication could break the heterocyclic ether bonds between the sugar monomers in polymeric chains formed by the hemicelluloses and the cellulose [30]. The breaking of these bonds releases several compounds, mainly sugars such as xylose, glucose, and arabinose. As coalesce and DAF focus on separation mechanisms, the effect is less reducing sugar released.

3.2.2. BOD_5 and TCOD ratio

The analysis data showed that the BOD_5/TCOD values for POME pretreated by SE, SE + US, SE + DAF, and SE + Co were 0.53, 0.60, 0.50, and 0.56, respectively (Fig. 3). These values were

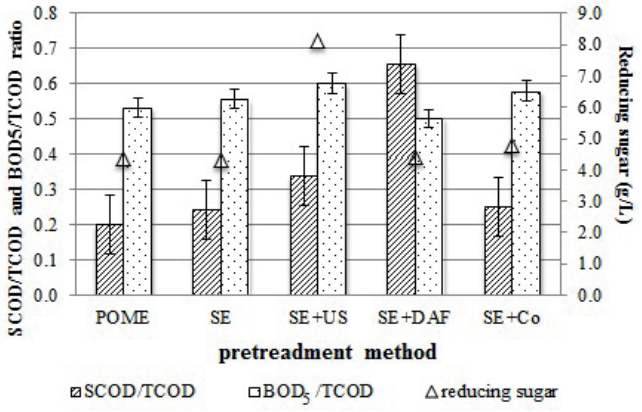

Fig. 3. BOD_5/TCOD, SCOD/TCOD and reducing sugar of pretreated POME.

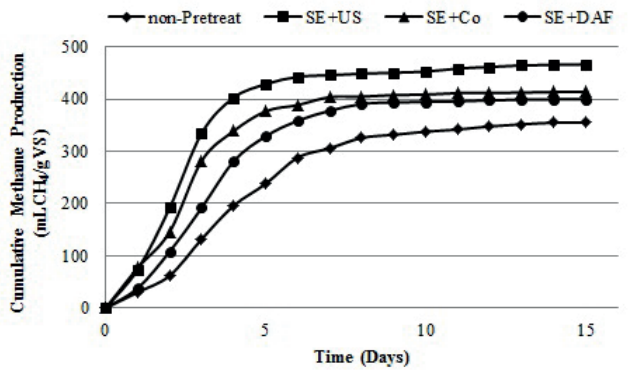

Fig. 4. Accumulative methane production in bath experiments with different pretreatment.

not different or slightly increased from the initial value of raw POME at 0.53. The ratio of BOD_5 and TCOD (BOD_5/TCOD) reflects the ability of an organic substrate to be degraded by microorganisms, also called biodegradability. The BOD_5/TCOD can vary from 0.1 to 0.8 [26]. Larson et al. [32] found that a BOD_5/TCOD ratio equal to 0 is non-biodegradable. Moreover, one that is less than 0.1 indicates a low biodegradability and needs to be improved to achieve biodegradation. BOD_5/TCOD values in the range of 0.3–0.6 show that the addition of microorganisms is required to aid in biodegradation. If it is greater than 0.6, it indicates that there is an optimum condition for biodegradation as well.

3.2.3. SCOD and TCOD ratio

SCOD/TCOD ratio represents the release of the organic matter from a solid state to a liquid state after pretreatment. The SCOD increases due to solubilisation of the solid-phase matter and increase in the concentration of organic matter in an aqueous phase. Therefore, SCOD/TCOD can be used as a parameter to evaluate the sludge degradation of anaerobic digestion [26]. In this experiment, SCOD/TCOD was increased from 0.20 (SE) to 0.34, 0.65, and 0.26 for SE + US, SE + DAF, and SE + Co pretreatment, respectively. However, the increase of SCOD/TCOD value in the DAF process was a product of the significant reduction of initial TCOD (75%), not due to the increase of SCOD. In addition, the DAF resulted in a reduction of SS and oil and grease, which affected the slight decrease of BOD_5/TCOD, while the reducing sugar remained unchanged (4.36 g/L). Therefore, the increasing of SCOD/TCOD value for this pretreatment method does not contribute to biogas production, as shown in Fig. 4.

3.3. Biogas production and methane yield

BMP tests were carried out to determine the anaerobic biodegradability of pretreated POME. The results of methane yield are given in Fig. 4. At the end of the experimental period, the maximum methane yield at 467 mL/gVS (3.05 LCH_4/L POME) and methane production rate at 147 LCH_4/day was found in POME pretreated by SE + US. Meanwhile, the methane yield of SE + DAF and SE + Co pretreatment obtained was 400 mL/gVS (2.65 LCH_4/L POME) and 414 mL/gVS (2.74 LCH_4/L POME), while methane production rates obtained were 93 and 114 LCH_4/day, respectively. In accordance with the experimental results, SE + DAF showed the lowest methane yield. In a study by Choorit and Wisarnwan [33], a completely stirred tank reactor (CSTR) was used in the treatment of raw POME. The CSTR was operated at 55°C and investigated for maximum methane yield at 2.65 LCH_4/L POME.

The modified Gompertz equation was used to fit the cumulative daily methane production. The individual cumulative methane production data were used to estimate the three constants for the modified Gompertz equation (methane production potential, maximum methane production rate, and duration of lag phase). The parameters were estimated using a SOLVER tool of Excel™, as listed in Table 2. The modified Gompertz equation gives a good fit for experimental data with high R^2 values of 0.9997, 0.9991, and 0.9998 for SE + US, SE + Co, and SE + DAF, respectively. The calculated ultimate methane production and lag time were close to the observed data. All of the BMP tests had short lag times (0.53–0.85 day), demonstrating that the bacteria could adapt to the environment quickly. It also had a tendency to reduce the lag time period in comparison with non-pretreatment (Fig. 4). The difference between observed and calculated data for methane production values was about 1%–2%. It was confirmed that the pretreatment method studied in this research could improve the biodegradability of POME for biogas production. However, SE + US yielded the highest biogas yield potential and production rate, which increased the biogas yield potential by 29.7% (Table 2).

4. Conclusion

Partial oil removal by sweeping off the surface and pretreatment by ultrasonication, DAF, and Co pretreatment did not impact POME structural change because they are physical methods. However, the breakdown of some substances into monomer sugar increased the biodegradability of POME for biogas production. These processes could also be used as an alternative approach to oil recovery from POME and returning recovered oil back into the CPO process to produce grade B oil palm, which would benefit the palm oil mill. The pretreatment process, especially ultrasonication, showed a significant

Table 2
Model parameters identified from regression of the methane production profiles based on the Gompertz equation

Pretreatment	Methane production experiment (mL CH_4/gVS)	Methane potential, P_m (mL CH_4/gVS)	Methane production rate, R_m (mL/day)	Lag time, x_0 (day)	R^2	Methane potential increase (%)
SE	355	353	64	0.98	0.9998	
SE + US	467	458	147	0.61	0.9997	29.74
SE + Co	414	411	114	0.53	0.9991	16.43
SE + DAF	400	399	93	0.85	0.9998	13.03

increase in reducing sugar and improvement of substrate digestibility as well as biogas production when compared with non-pretreated POME. If biogas production was increased by 29%, an estimated gain of 594,000 kWh would result. With this expected capacity, it could provide support electrical use for an additional 87 households. Therefore, it is an interesting method for further research and additional conclusions.

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