Chemical pre-treatments on oil palm empty fruit bunches: Impacts on characteristics and methane potential

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Abstract. Production of biogas from lignocellulosic biomass by anaerobic digestion (AD) has attracted much interest. Oil palm empty fruit bunches (OPEFB), one of lignocellulosic biomass, is highly abundant in Indonesia and has potential as feedstock for bioenergy production such as biogas or methane. Yet, pre-treatments are needed to improve biogas production due to its complex crystalline structures. Chemical pre-treatments with acid or alkaline solution were reported to increase cellulose or highly reduce the lignin content of OPEFB. This study aimed to evaluate the effect of acid and alkaline pre-treatments on the characteristics of OPEFB and methane potential. The acid pre-treatment experimental design was used factor of H2SO4 concentration (1, 1.3, and 1.6 (%v/v)) and NaOH concentration (1.8, 2.8, and 3.8 (%w/v)). Methane potential evaluation was carried out using the biochemical methane potential (BMP) test with the Automatic Methane Potential Test System (AMPTS) II under mesophiliic condition (37°C), operated for 28 days. The results showed that both dilute acid and alkaline pre-treatment positively impact altering the characteristics of OPEFB, hence the specific methane potential. Alkaline pre-treatment with NaOH 3.8 (%w/v) gave the highest average SMP value of 0.161 ± 0.005 m3 CH4/kgVS added.

1. Introduction

Palm oil (Elaeis guineensis) production in Indonesia continues to increase from 42.88 million tons (in 2018) to 49.12 million tons (in 2020) [1]. A previous by Hayashi [2] reported that 1 ton of fresh fruit bunches (FFB) produces 21.8% of Crude Palm Oil (CPO), 22.5% of OPEFB; 14.3% of fibre; 6.7% of shell; and 5.4% of kernels. In Indonesia, the generation of OPEFB, as the most solid waste produced, is continuously increased. Based on the data of oil palm production from the Directorate General of Plantations in 2020, it can be estimated that the OPEFB production was 11 million tons [3].

Law et al. [4] stated that OPEFB, categorized as lignocellulosic biomass, contains 44.2% cellulose, 33.5% hemicellulose, and 20.4% lignin. The dominance of cellulose in OPEFB indicates its potential as energy-producing feedstocks, such as biogas production [5]. Biogas, produced from the anaerobic digestion cess, is a gas mixture containing up to 60% [6]. However, the complex structure of lignocellulose inhibits the conversion of OPEFB to biogas. Hence improving the AD efficiency is, therefore, essential [7,8].

According to Tsabitah et al. [9], dilute acid, and alkaline solutions are the most commonly used for OPEFB pre-treatment. Several studies have shown that chemical pre-treatments on lignocellulosic biomass using dilute sulfuric acid (H2SO4) and sodium hydroxide (NaOH) have significantly increased the delignification and methane production [10–12]. In chemical pre-treatments, with acid or alkaline
solution, two fractions are generated include solid (also known as residual solids) or liquid (also known as hydrolysate) fraction [13]. As a result of cellulose and hemicellulose degradation, some sugars are present in both fractions, which may affect biogas and methane yield during the AD process [14]. A previous study highlighted that hydrolysate, resulted from chemical pre-treatment, contains considerable amounts of sugar from cellulose and hemicellulose degradation. Thus, disposing of the hydrolysate may probably reduce the source of organic matter for methanogenic bacteria [15]. Furthermore, if using sulfuric acid pre-treatment excessively, the presence of sulphate ions in the hydrolysate may act as an inhibitor in the AD process due to competition between methanogens and sulphur reducing bacteria [16]. Such conditions may contribute to lowering the biogas and methane yield.

Several studies have emphasized the potential of using H$_2$SO$_4$ and NaOH in the chemical pre-treatment of OPEFB. For instance, using 8%, H$_2$SO$_4$ was found to alter lignin content from 28.78% to 19.6%, then pre-treatment was continued by 10N NaOH and achieved 70% of delignification in total [17]. Tye et al. [18] studied OPEFB pre-treatment using 2% of H$_2$SO$_4$ at 120°C for 45 min obtained total glucose yield (TGY) by 35% and 34.8% after using 3% of NaOH at 110°C for 45 min. Other studies have reported an enhanced methane yield from chemically-treated solid fractions following washing pre-treatment prior AD process [10–12,19,20]. For example, pre-treated corn straw with 2% of H$_2$SO$_4$, following with washing using clean water, produced a higher methane yield by 74.5% (or 175.6 mL CH$_4$/g VS) than untreated corn straw at 100.6 mL CH$_4$/g VS [10]. Another study reported that pre-treated OPEFB with 8% of NaOH and washed by distilled water generated methane yield by 100% (or 404 mL CH$_4$/g VS) higher than that of untreated OPEFB (202 mL CH$_4$/g VS) [19]. Wadchasit et al. [20] also showed a quite good increase in methane yield by 29.88% (from 266.0 to 345.5 mL CH$_4$/g VS) with treated-OPEFB using 7% of NaOH. However, there is a lack of information about biogas or methane potential from OPEFB acid-treated as substrate, significantly dilute acid (H$_2$SO$_4$), in chemical pre-treatment of OPEFB. This study aimed to evaluate the effect of dilute acid (H$_2$SO$_4$) and alkaline (NaOH) pre-treatments on the physical and chemical characteristics of OPEFB and methane potential.

2. Material and methods

2.1. Feedstocks and inoculums
Fresh OPEFB was obtained from PT Sawit Arum Madani, Blitar Regency (East Java, Indonesia). The OPEFBs were then dried using sunlight for five days and chopped with a chopper to particle size of 0.5 - 1 cm. The characteristics of both untreated and treated OPEFBs were analyzed using proximate analysis include moisture content (MC), total solids (TS), volatile solids (VS) and ash content.

For the biochemical methane potential (BMP) test, the inoculum was prepared from the digestate taken from a full-scale mesophilic digester treating cattle slurry at Balai Besar Pelatihan Peternakan (BBPP), Batu City (East Java, Indonesia). The digestate taken was filtered using a 1 mm sieve to remove large particles, and then pre-incubated at 37°C for 48 hours prior to use. Inoculum analysis was carried out for MC, TS, VS, ash content and pH.

2.2. Pre-treatment process

2.2.1. Dilute sulfuric acid pre-treatment. Dilute H$_2$SO$_4$ pre-treatment was conducted according to method described previously by Bouza et al. [21], with some modification. First, the dried OPEFBs (70 g) were mixed into 560 mL of an acid solution at concentrations of 1, 1.3, and 1.6 (%v/v) in beaker glass then covered with aluminium foil. These mixture samples were heated to 150°C for 90 minutes, then filtered using filter paper. The remaining OPEFB residues were cooled using running clean water for 1 minute and drained the excess water prior to BMP test.

2.2.2. Dilute sodium hydroxide pre-treatment. Dilute NaOH pre-treatment was performed based on Han et al. previously [22], with some modifications. The dried OPEFBs (70 g) were mixed into 420 mL of an alkaline solution at 1.8, 2.8 and 3.8 (%w/v) in beaker glass then covered with aluminium foil. The
mixture samples were heated to 127.7°C for 22 minutes and filtered using a filter paper. The remaining OPEFB residues were then cooled using running clean water for 1 minute and drained the excess water prior to the BMP test.

2.3. BMP test set-up

The BMP test was carried out based on our previous study reported by Suhartini et al. [23,24], with some modifications. The BMP test was performed using an automated methane potential test system (AMPTS II, Bioprocess Control Sweden AB) at 37°C for 28 days. The inoculum-to-substrate (I/S) ratio used was 4:1 on a VS basis. This work was performed in 500 mL reactors with a working volume of 400 mL, all prepared in triplicate composed of blank samples, positive control samples (α-cellulose powder from Sigma-Aldrich, Dorset-UK), and substrate samples. Blank samples contained inoculum only, intended to measure methane production from indigenous bacteria present in inoculum. The positive control sample consisted of inoculum and α-cellulose, which were intended to measure the activity of the inoculum. The substrates samples reactors contained inoculum and OPEFB samples (i.e. untreated, acid-treated and alkaline-treated OPEFBs).

The reactors were closed with rubber stopper and then connected to the mechanical agitator, which set up for 60s on and 60s off at 30 rpm. The resulted biogas passed through one-way valve and entered into 3 M NaOH solution unit for CO₂ scrubbing. The 3 M NaOH solution was prepared using NaOH solution with addition of thymolphthalein solution (0.4%) as pH indicator. Then, the methane gas passes to the sensor chamber is detected and recorded online by AMPTS software.

2.4. Analysis

The MC, TS, VS, and ash content of materials were measured according to the standard methods explained in APHA [25]. The pH was measured before and after BMP tests using a digital pH meter, calibrated in buffers at pH 7 and 9.2. The scanning electron microscopy (SEM) was carried out using Scanning Electron Microscopy (SEM) to identify the structure and morphology of OPEFB before and after chemical pre-treatments. The methane volume was measured online by AMPTS II, while the specific methane potential was calculated according to Strömberg et al. [26], with the assumption of methane concentration of 60% [24,27]. In contrast, the estimation of the electrical energy potential was calculated based on Suhartini et al. [28,29]. Mean values and standard deviation were calculated by using Microsoft Excel.

3. Results and discussion

3.1. The effect of chemical pre-treatments on the characteristics of OPEFB

Table 1 shows the characteristics of OPEFB before and after chemical pre-treatments. It can be seen that untreated OPEFB contains high organic content as indicated by a high value of VS (92.68%TS). After pre-treatment with dilute acid and alkaline solution, an increase in VS content was found in all samples. However, dilute acid pre-treatment resulted much higher VS improvement (in the range of 6.12 – 6.64 %) compared to that of dilute alkaline pre-treatment (in the range of 2.98 – 4.42 %). An increase in VS content, after pre-treatment, may indicate a potential improvement in biogas and methane production. This study is in agreement with other reported findings. For example, a previous study by Petersson et al. [30] has reported that a high VS content in biomass feedstock may positively affect the biogas production. Another study also demonstrated that a high organic matter (as indicated by the VS content) in the OPEFB indicates its potential as a sustainable feedstock in AD [27].

Table 1 also indicates that both after acid and alkaline pre-treatments, all samples have a significant increase in MC values, following a decrease in TS and VS content (on a wet weight basis). Nieves et al. [31] also reported a significant decrease in both TS and VS, as well as an increase in MC values, following acid and alkaline pre-treatments. They found that, the soaking of OPEFB in acid and alkaline solutions was greatly lowering the TS and VS content compared to that of the untreated dried OPEFB. Furthermore, an increase in MC was found to be proportional with an increase in VS (as %TS), which
is possibly due to additional polar liquid in each pre-treatment. According to Jawaid et al. [32], cellulose is one of the components in OPEFB fibre which has hydrophilic properties, causing hydrogen bonds to other polar components. Due to the association of polar functional groups, the capillary action causes the intake of water, thus increasing its water content. Previous study by Fang et al. [33] also showed that the surface structure of OPEFB fibre becomes larger or rounder in size, typically as a result of an accumulation of liquid due to the water absorption. While, Orhorhoro et al. [34] claimed that a high water content could probably increase the microbial activity to transform organic content presents in the substrate.

### Table 1. The characteristics of OPEFB before and after chemical pre-treatments.

| Parameters | Untreated OPEFB | After pre-treatment |
|------------|-----------------|---------------------|
|            | Dilute acid (%v/v) | Dilute alkaline (%w/v) |
| TS (%WW)   | 88.59           | 29.94 - 30.51       |
| VS (%WW)   | 82.05           | 29.48 - 29.14       |
| VS (%TS)   | 92.68           | 98.48 - 95.44       |
| MC (%WW)   | 9.42            | 70.06 - 54.57       |
| Ash (%WW)  | 5.98            | 0.46 - 1.39         |

Note: WW = wet weight. Data are expressed as means of triplicate samples.

As shown in Table 1, acid and alkaline pre-treatment has a significant impact of reducing the amount of ash content in treated OPEFB in the range of 91.97-93.98% and 76.76-83.61%, respectively. Ash contains mineral and inorganic materials that may affect the rate of combustion in biomass samples [35]. According to He et al. [36], higher ash content in biomass substrates may cause to lowering the degradation of organic content into methane by microorganisms.

Figure 1 shows the SEM images of OPEFB before and after acid or alkaline pre-treatments. It can be seen that untreated OPEFB (Figure 1a) has an intact and solid covered by a ball-like structure. However, after being subjected to acid pre-treatments, as shown in Figure 1b-d, disruption on the OPEFB’s cell wall was observed at all concentrations tested. After alkaline pre-treatment, a slight removal of silica was evident. These findings are similar to a previous study by Sannigrahi et al. [37] who found the presence of ridge-like features in acid-treated lignocellulosic materials, which could attribute to the preferential degradation of the more labile components such as hemicellulose and cellulose. Barlian et al. [38] also found that the fibre surface of alkaline-pre-treated OPEFB was rougher and had more pores possibly due to the release of silica from the circular craters. The SEMs images further confirmed that acid and alkaline pre-treatments have greater efficacy in damaging the cell wall of OPEFB, which may have a positive impact on enhancing the biogas and methane potential. Pre-treatment improved the structure of OPEFB by the appearance of ridge-like features, and more pores were expected to increase the surface area of the fibres. This, makes it easier for anaerobic microorganisms to access organic content inside OPEFB.
Figure 1. Image of OPEFB: (a) before pre-treatment; (b-d) after acid pre-treatment; and (e-g) after alkaline pre-treatment. SEM at magnification of 500x (200 μm).
3.2. The effect of chemical pre-treatments on methane potential

Table 2 shows an average of specific biogas potential (SBP) and specific methane potential (SMP) based on a VS basis in each sample. In this study, untreated OPEFB had SMP value of $\sim 0.070 \text{ m}^3 \text{CH}_4/\text{kgVS}_{\text{added}}$ and SBP value of $\sim 0.117 \text{ m}^3 \text{CH}_4/\text{kgVS}_{\text{added}}$. The results showed that both acid and alkaline pre-treatment on OPEFB caused an increase in SMP and SBP value. This was possibly due to an increase in VS concentration of treated OPEFB. Furthermore, as shown in the SEM images, a disruption on the cell’s wall OPEFB after acid and alkaline pre-treatment, making it more accessible by the microorganisms to attack easily degradable organic material. This study also shows that increasing the acid concentration leads to reduction in the SMP and SBP values. Acid-treated OPEFB at a concentration of 1 (%v/v) generated the highest SMP and SBP values compared to the other counterparts, giving the average values of $\sim 0.091 \text{ m}^3 \text{CH}_4/\text{kgVS}_{\text{added}}$ and $\sim 0.152 \text{ m}^3 \text{biogas/kgVS}_{\text{added}}$, respectively. This phenomenon may possibly be due to the loss of hemicellulose and cellulose fractions after acid pre-treatment, as described by Guo et al. [39]. Considerable amounts of sugar resulted from degradation of cellulose and hemicellulose are still remained in the hydrolysate (or the liquid fractions) [12]. Furthermore, a decrease of SMP values can also be caused by formic acid and furfural remained in the residual solid fractions after acid pre-treatment [40]. Thus, such conditions resulted in decreasing the total sugar yield, as well as reduced organic content in treated OPEFB to be further converted into methane.

On the other hand, the SMP values of alkaline-treated OPEFB at a concentration of 1.8, 2.8, and 3.8 (%w/v) were about 0.132, 0.134, and 0.161 CH$_4$/kgVS$_{\text{added}}$, respectively. The alkaline pre-treatment with concentration of 3.8 (%w/v) produced the highest SMP value compared to that of other treatments. This indicates that increasing the concentration of NaOH was parallel to an increase in the methane potential from alkaline-treated OPEFB. Bartos et al. [41] explained that hemicellulose and lignin content continuously decrease with increasing NaOH concentration. Their study also reported that the optimum concentration of NaOH solution was around 5% after 1h treatment time.

From Table 2, it can be seen that both acid and alkaline pre-treatment have positive effect in the biogas and methane production than untreated OPEFB. However, the study also revealed that alkaline pre-treatment on OPEFB highly improved the SMP and SBP by more than 88%. While acid pre-treatment was able to enhance SMP and SBP of OPEFB in the range of $\sim 6$ to $30\%$. This is clear that alkaline pre-treatment offers superior performance in increasing the biogas and methane production from OPEFB. This finding is in agreement with Hernández-Beltrán et al. [6], who also found that higher methane yield was produced from alkaline-treated OPEFB than that of acid-treated OPEFB.

Digestate characteristics before and after BMP test trials were also measured and the results are shown in Table 2. The mono-digestion of alkaline-treated OPEFB with NaOH concentration of 3.8 (%w/v) produced digestate with the lowest VS value (1.15 %WW) and the highest SBP and SMP. A lower VS value in the remained digestate may have been associated with higher biogas or methane production, as previously explained by Suhartini et al. [42]. Furthermore, the remained digestate still contains a high organic fraction, which can be potential to be further utilized as bio-fertilizer, compost, or for direct application to the soil as growing media. Yet, future work in the identification of macro-nutrient (i.e. N, P, and K) in the digestate from AD of OPEFB is essential for achieving zero-waste and integrated biorefinery in bioenergy production. As highlighted by Nkoa [43], digestate application to soil has been associated with accumulation and presence of nutrients, further investigation is still needed.
Table 2. Performance and digestate characteristics from the BMP test trials of untreated and treated OPEFB.

| Sample type                  | Ave. SMP (m³ CH₄/kg VSₐdded) | Ave. SBP (m³ CH₄/kg VSₐdded) | MC (%WW) | TS (%WW) | VS (%WW) | VS (%TS) | Ash (%WW) | pH Start | pH End |
|------------------------------|-------------------------------|-------------------------------|----------|----------|----------|----------|-----------|---------|-------|
| Without pre-treatment trials | Untreated OPEFB               | 0.070 ± 0.012                 | 0.117 ± 0.019                   | 96.66    | 3.34     | 3.00     | 89.88    | 0.34    | 6.84   | 6.74   |
| Acid pre-treatment trials    | 1 (%v/v)                      | 0.091 ± 0.002                 | 0.152 ± 0.004                   | 97.70    | 2.30     | 1.89     | 82.11    | 0.41    | 6.26   | 6.06   |
|                              | 1.3 (%v/v)                    | 0.080 ± 0.015                 | 0.134 ± 0.024                   | 97.98    | 2.02     | 1.63     | 81.01    | 0.38    | 6.25   | 6.10   |
|                              | 1.6 (%v/v)                    | 0.074 ± 0.010                 | 0.124 ± 0.016                   | 97.84    | 2.16     | 1.77     | 81.89    | 0.39    | 6.05   | 6.04   |
| Alkaline pre-treatment trials| 1.8 (%w/v)                    | 0.132 ± 0.015                 | 0.220 ± 0.024                   | 98.35    | 1.65     | 1.21     | 73.36    | 0.44    | 6.89   | 6.63   |
|                              | 2.8 (%w/v)                    | 0.134 ± 0.024                 | 0.223 ± 0.039                   | 98.39    | 1.61     | 1.18     | 72.93    | 0.44    | 6.91   | 6.58   |
|                              | 3.8 (%w/v)                    | 0.161 ± 0.005                 | 0.268 ± 0.008                   | 98.43    | 1.57     | 1.15     | 71.16    | 0.42    | 6.96   | 6.58   |

Note: Data are expressed as means of triplicate samples. The symbols of ± are expressed as the standard deviation of three measurements.
As shown in Table 2, the initial pH of untreated and alkaline-treated OPEFB before BMP test was in the range of 6.84–6.96, well within than the ideal pH for AD (ranged from 6.3 to 7.2) as recommended by Zou et al. [44]. However, the initial pH of acid-treated OPEFB was slightly lower than that of the ideal pH value, which ranged from 6.05-6.26. This study shows that addition of acid-treated OPEFB tend to reduce the pH, possibly due to the remained sulphuric acid in the solid fractions. It is clear that increasing the concentration of sulphuric acid from 1 to 1.6 (%v/v) during acid pre-treatment, contribute to reducing the initial pH of the mixture samples in the BMP test. However, all tested samples were found to produce biogas and methane at different values and rates. This indicates that AD process is stable and the presence of anaerobic consortia in the inoculum is still able to transform organic matters present in the OPEFB into biogas or methane. Espino-escalante [45] reported that AD process is unstable at pH values below 6.0 and above 8.0.

4. Conclusions
OPEFB has a high amount of organic content, making it a potential feedstock for AD. Dilute acid (H$_2$SO$_4$) and alkaline (NaOH) pre-treatments were found to enhance the characteristics of OPEFB, thus allowing better biodegradation than that of untreated OPEFB. Alkaline-treated OPEFBs using NaOH concentration of 3.8 (%w/v) generated the highest SMP value of 0.161 m$^3$ CH$_4$/kgVS$_{added}$ than that of untreated OPEFB (0.070 m$^3$ CH$_4$/kgVS$_{added}$). Therefore, alkaline pre-treatment with NaOH is recommended to be combined with AD of OPEFB system, aiming for better methane yield and safe process.

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