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The effectiveness of anaerobic digestion process by thermal pre-treatment on food waste as a substrate

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Abstract. Thermal pre-treatment is necessary for the feedstock of Anaerobic Digester (AD) to improve the degradation process. This study aims to investigate the effect of thermal pre-treatment on Food Waste (FW) as a substrate through Biochemical Methane Potential (BMP) test followed by pilot scale using a continuous stirred tank reactor (CSTR) digester. TS, VS, COD, SCOD, and solubilization samples were measured during on CSTR reactor. FW was pre-treated by heating at 70 °C, 90 °C, and 120 °C for 45 minutes. Results showed that thermal pre-treatment at 70 °C had the most effective digestion process with TS, VS, COD reduction of 8.26%; 2.54%; and 14% and increase SCOD and solubilization of 10.2% and 24.7%, respectively compared to thermal pre-treatment at 90 °C and 120 °C (p<0.05). Therefore, thermal pre-treatment at 70 °C was chosen for AD pilot scale in this study. Meanwhile, pilot scale experiment showed the results that thermal pre-treatment at 70 °C was obtained significant differences of 31% TS; 38% VS; 42% COD; 23% SCOD; and 30% solubilization compared with without thermal pre-treatment (p<0.05). Pre-treatment with thermal on FW as a substrate may help in increasing the degradation process at the hydrolysis phase in order to increase the effectiveness of the process in AD reactor.

1. Introduction

Food Waste (FW) is a good source of substrates for Anaerobic Digestion (AD) process because it contains high organic matter, sufficient moisture content, and high biodegradability [9]. AD consists of a phase of a chemical reaction which is hydrolysis, acidogenesis, acetogenesis, and methanogenesis. The substrate in hydrolysis phase was solubilized, and polymers were converted into simple monomers while acidogenesis phase was converted into volatile fatty acid (VFA). Meanwhile, acetogenesis phase was converted VFAs into acetic acid; carbon dioxide; and hydrogen, and methanogenesis phase was converted acetates into biogas [19]. However, the optimization of AD process is necessary due to the long retention time.

Pre-treatment methods have rapidly developed in recent years because it would help to enhance soluble organic material’s accessibility and could enhance the biogas production and accelerate the digestion process especially hydrolysis phase which takes the longest time amongst another phase [12]. There were many pre-treatment methods to increase the optimization of AD processes such as mechanical treatment, thermal treatment, and thermo-chemical treatment. In this study, thermal pre-treatment was chosen because it can increase solubilization of organic materials effectively and increase total soluble chemical oxygen demand (SCOD) to enhance digestion process by anaerobic microorganisms compare to another pre-treatment [13]. SCOD represents an available source of...
energy for the microorganisms to do the activities and to show the condition of the microorganism itself in the whole process. Thus, by providing a pre-treated substrate to AD reactor, it will increase the efficiency of AD process within a shorter retention time [12].

Heating an amount of anaerobic digester’s feedstock at 120 °C for 30 minutes could increase the COD solubilization of feedstock from the initial condition of 7.9% to 43.7% while Bougrier et al. (2008) reported that thermal pre-treatment for anaerobic digester’s feedstock at 50 °C did not increase the COD solubilization, but could increase solubilization 10 – 20% at temperature of 100 °C – 120 °C.

The temperature for thermal pre-treatment can be varied from 50 °C – 250 °C which divided into two types. The temperature below 110 °C was indicated as low thermal pretreatment, and temperature above 110 °C was indicated as high thermal pre-treatment. Li et al., (2016) show that thermal pre-treatment with low temperature (55 – 90 °C) did not show a significant enhancement of COD solubilization, but thermal pre-treatment at 70 °C produced the highest solubilization. The other research from Rafique et al. (2010) reported different results using temperature from 25 °C – 150 °C and feedstock with thermal pre-treatment at 100 °C produced the highest solubilization. Meanwhile, feedstock with a heating temperature of 130 °C and 150 °C showed the lowest solubilization.

The main effect of thermal pre-treatment of food waste as AD substrates is the disintegration of the cell membrane which produces organic material solubilization so it will make easier for microorganisms to digest the feedstock within a shorter time [2]. As a result, the enhancement of COD solubilization will make a higher efficiency of the anaerobic digestion process [3].

The aims of this study were to investigate the effects of thermal pre-treatment on food waste as a substrate for AD process. Specifically, the study focused on determining the thermal temperature effects at 70 °C, 90 °C, and 120 °C on Total Solid (TS), Volatile Solid (VS), COD, SCOD, and solubilization. The study was done by laboratory scale first to choose the temperature which has the highest effect on reduction and solubilization process then pilot scale by using continuous stirred tank reactor (CSTR) digester. Highest reduction and solubilization could accelerate the digestion process in a pilot scale [16].

2. Materials and Methods

2.1. Inoculum and Substrate

The inoculum used in this study was taken from an active AD plant treating organic waste at Petamburan Market, Grogol, Jakarta. 25% of inoculum was mixed with 75% of FW, and it was acclimatized and degasified for 30 days in order to adapt the bacteria to the substrate. The inoculum was monitored every day to keep the pH not lower than 6. Sodium bicarbonate (NaHCO₃) was added as a buffer solution with 3 gr/L inoculum dosage.

FW as a substrate was used in this study from Canteen in Engineering Faculty, Universitas Indonesia, Depok, Indonesia. The sample collection used quartering method by Wellinger et al., (2013) to get only the organic waste and to avoid a few kinds of FW which have acidic content or hard texture because it would be difficult to process for bacteria. The FW shredded into 2 – 3 mm based on Basaria and Priadi (2016) to make the size of the FW was homogenous and smaller to enhance the digestion process. The FW and CM were taken once as needed and stored in the freezer to be taken at feeding.

2.2. Preparation of BMP Test

BMP process was used to analyze the most effective temperature of substrate pre-treatment based on the TS, VS, COD reduction, an increase of SCOD and solubilization. In addition, it used to determine the highest biogas production from the samples. The method was used for BMP process from Holliger et al., (2016) and carried out in triplicates.

FW as a main substrate for the BMP process was varied by thermal pre-treatment with a temperature of 70 °C, 90 °C, and 120 °C. These variations were added with 25% of inoculum which has been adapting first to the substrate, and it was analyzed to determine their initial characteristics by measure the pH, TS, VS, COD, SCOD, and solubilization by using Indonesia Nasional Standard while flowmeter was used to measure biogas production. The pH of each substrate must be in pH between 7
3.0 – 8.5 for the optimum pH range of AD process. Each substrate that had pH lower than 7 was added 3 gr/L sodium bicarbonate (NaHCO₃) as a buffer. During the experiment, there were also positive control samples, control sample, blank samples, and experiment control samples as a comparison to the variation samples.

The BMP substrates were mixed from 75% of FW which has been treated by thermal pre-treatment and 25% of inoculum which has been adapting first. 10 gr VS/L of BMP substrates were stored in vial bottles with 100 mL volume which 50 mL was filled with the substrate, and 50 mL was filled for headspace. Headspace provided to prevent high pressure from the biogas produced that will affect the BMP process (Koch, Fernandez, & Drewes, 2015). Each bottle was sealed with rubber and aluminum cap to avoid the gas leak. Then, each bottle was purged using nitrogen gas for 3 minutes to make sure that the condition of each sample was anaerobic to provide a suitable condition for anaerobic microorganisms to grow. All BMP substrate was stored in the incubator with a temperature of 37 °C for 50 days in this research or until the daily production of gas was lowered.

During the incubation period, BMP bottles were shaken once a day to prevent scum forming which could inhibit biogas production. Biogas volume was measured weekly in the first 3 weeks and days until the end of BMP process using flowmeter. BMP process was terminated when the biogas production in three days in a row was lower than 1% of the biogas accumulation volume [10].

2.3. Analytical Methods
Parameters of this study were biogas, Total Solid (TS), Volatile Solid (VS), Chemical Oxygen Demand (COD), Soluble Chemical Oxygen Demand (SCOD), and solubilization which dividing COD to SCOD. Each parameter was measured periodically with the Indonesia National Standard. However, measurement of SCOD in this study using COD method, but beforehand there was a filtration of the sample using 0.40 𝜇m filter.

Comparison of characteristics of BMP substrate before and after was analyzed to determine which temperature has the highest removal of TS, VS, COD, increasing of SCOD and solubilization, and the highest biogas volume. This information was used to determine the most effective thermal pre-treatment temperature to produce biogas and treated the FW in AD reactor. All measurements were carried out in triplicates. One way ANOVA tests and Tukey’s multiple comparison tests were performed by using SPSS software version 16.0. The level of significance was 0.05 for all the tests to determine the most effective thermal pre-treatment for AD process and investigate the effect of thermal pre-treatment on AD process.

2.4. Pilot-scale anaerobic digester reactor
The type of AD reactor was a continuous stirred tank reactor (CSTR) which has operational of 60 rpm for 4 hours per day based on our previous study. Dry AD system was conducted in this study as explained in Wijayanti et al., (2018). The most effective thermal pre-treatment temperature was applied to the pilot scale AD reactor which obtained during BMP experiment.

The amount of 90 Kg of the substrate was prepared for 2 weeks worth of feeding. The sample collection used the quartering method by Wellinger et al., (2013) to get only the organic waste and to avoid a few kinds of FW which have acidic content or hard texture. The substrate was heated in a stainless steel container and stirred manually for 45 – 60 minutes. The pre-treated substrate was cooled down to room temperature and stored in the freezer with –20 °C to prevent any change of substrate characteristics before the feeding process. The substrate was taken out from the freezer one day before feeding day.

3. Results and Discussion
3.1. Initial Characteristic
The aims of the experiment investigated the effect of thermal pre-treatment on Food Waste (FW) as a substrate through Biochemical Methane Potential (BMP) test. Initial characteristic of FW was used as a substrate and inoculum has been determining and show in Table 1.
Based on Table 1, FW had an optimum TS concentration with a range of 20 – 50% [15]. In addition, the test was also carried out to determine the initial characteristics of the inoculum. The results showed that TS was still in the optimum range of around 2 – 10% [21].

| Sample     | TS (%)    | VS (%)    |
|------------|-----------|-----------|
| Food Waste | 23.7 ± 0.15 | 22.8 ± 0.10 |
| Inoculum   | 6.06 ± 0.07 | 3.35 ± 0.05 |

On the other hand, Table 1 also shows VS concentration. Based on Table 1, VS concentration of FW was 96 ± 0.20% and VS concentration of Inoculum was 55 ± 0.08%. Biogas production can be estimated based on VS content [21]. Tsunatu et al., (2014) showed that the percentage of VS content from TS content must be in a range of 80 – 90% to produce the optimum biogas. The TS and VS content of substrates affects the performances of the anaerobic digestion process. Change in total solids content will lead to change in microbial morphology of AD systems. Thus, the FW in this study was suitable to be used as the substrates for AD process.

### 3.2. BMP Process Results

The effect of the thermal pre-treatment on the substrate was carried out using BMP testing adapted to the previous study conducted by Wellinger et al., (2013) to obtain the most optimum temperature for the pre-treatment. The results are shown in Table 2.

| Sample | Efficiency of TS Reduction | Efficiency of VS Reduction | Efficiency of COD Reduction | Efficiency of Increasing SCOD | Efficiency of Increasing Solubilization | Biogas Production |
|--------|-----------------------------|----------------------------|-----------------------------|-------------------------------|----------------------------------------|-------------------|
| 70°C   | 8.26%                       | 2.54%                      | 14.0%                       | 10.32%                        | 24.7%                                  | 16.7 ± 1.68 L     |
| 90°C   | 4.04%                       | 2.32%                      | 15.0%                       | 9.09%                         | 23.2%                                  | 15.0 ± 0.74 L     |
| 120°C  | 7.97%                       | 15.32%                     | 14.2%                       | 12.30%                        | 24.4%                                  | 14.5 ± 0.47 L     |
| Control| 4.89%                       | 0.42%                      | 10.5%                       | 5.89%                         | 14.4%                                  | 14.5 ± 0.38 L     |

As shown in Table 2, the highest efficiency of TS reduction was obtained at a temperature of 70 °C and followed by 120 °C. The results of the statistical estimation showed that there was a great significant between the control sample and pre-treatment, as well as the thermal pre-treatment resulted in thermal pre-treatment at the temperature of 70 °C had the capacity to enhance TS reduction compared to the temperature at 90 °C and 120 °C (p<0.05).

VS concentration was also evaluated to identify biogas potential produced. The statistics result showed that there was no significant change in VS content before and after BMP process (p>0.05). The thermal pre-treatment also showed a weak correlation between the change of VS content (p>0.05). However, the correlation test between VS destruction efficiency and thermal pre-treatment showed a strong correlation based on VS destruction efficiency by up to 15.32% at thermal pre-treatment of 120 °C (P<0.05). This was also supported by the previous study showed that the increase of temperature on thermal pre-treatment could help the solid organic decomposition. Thus, thermal pre-treatment at 120 °C showed the highest decreasing solid mass ratio [11].

COD parameter was also evaluated in this study. The result showed that there was a reduction in COD concentration as shown in Table 2. The reduction occurred because of the degradation of organic matters in samples. On the other hand, the thermal pre-treatment and the COD reduction showed a strong correlation up to 0.88. Therefore, the higher temperature on thermal pre-treatment will enhance COD reduction. However, thermal pre-treatment with the temperature above 90 °C will affect the stability of digestion, so organic acids will increase, and pH will decrease. This will affect the metabolism of the microorganisms on AD process.
In this study, SCOD was also evaluated to further study about the level of hydrolysis on substrates. The result showed that there was a significant difference in SCOD efficiency on all samples (p<0.05). The temperature of 120 °C will enhance the highest efficiency of SCOD by up to 12.30%. In another hand, the correlation test between the thermal pre-treatment and the efficiency of SCOD conducted a strong correlation with a value of 0.94.

COD and SCOD obtained will be used to understand the percentage of solubilization of organic matters. The results showed that there were a significant enhancement on solubilization level on all samples and a significant difference between thermal pre-treatment (p<0.05). The increasing of solubilization value was affected by the increasing of SCOD due to mass transfer of COD or substrate solids in microorganisms. Thus, the higher solubility will help the microorganism on degrade COD [4].

Biogas production statistically proved to give a significant result on biogas production on all samples as shown in Table 2 (p<0.05). The temperature of 70 °C was the most optimum condition in producing the highest biogas, as well as a high solubility [18]. Therefore, the temperature of 70 °C will be used on the pilot-scale experiment as the thermal pre-treatment.

3.3. Pilot Scale Results
After a laboratory test conducted by BMP test, the pilot-scale experiment was performed to investigate the effect of thermal pre-treatment on AD process. The initial characteristics of pre-treated and un-pre-treated were shown in Table 3.

| Parameter | Unit | Un-pre-treated | Pre-treated |
|-----------|------|----------------|-------------|
| TS        | %    | 25.7 ± 0.15    | 25.4 ± 0.74 |
| VS        | %    | 22.8 ± 0.10    | 19.0 ± 0.82 |

As shown in table 3, TS and VS concentration could be reduced by up to 1.16 ± 0.02% and 16.7 ± 0.21%. Additionally, thermal pre-treatment statistically proven will not give a significant difference in TS concentration (p>0.05). In contrast, there was a significant difference in VS concentration (p<0.05). VS concentration was reduced after heated by thermal pre-treatment. Thermal pretreatment was more efficient to break long chained or otherwise complex compound into smaller chained molecules for easier digestion. Thus, VS concentration after pre-treatment was lower than before [18].

These results were contradicting with the previous study conducted by [17] which showed that there was an increase in TS and VS concentration by up to 5.4% and 4.5% using thermal pre-treatment. Another study conducted by Dlabaja & Malat’ak [6] was also contradicting with this study by enhancing TS concentration up to 4.9%. The differences might occur due to the different chemical composition of the materials used and the pre-treatment condition (temperature and holding time) [25]. The possible reason for the low concentration of TS and VS on thermal pre-treatment substrates occurred due to the high temperatures entailing the loss of volatile substances and thus the decline in final VS content [8].

| Parameter | Unit | Feeding Using Non-Pretreated Substrate | Feeding Using Pretreated Substrate | Increase |
|-----------|------|----------------------------------------|-----------------------------------|----------|
| TS        | %    | 15.5 ± 0.18                            | 10.7 ± 0.04                       | 30.9%    |
| VS        | %    | 12.2 ± 0.27                            | 7.60 ± 0.05                       | 37.7%    |
| COD       | gr/L | 264 ± 26.20                            | 154 ± 11.50                       | 41.6%    |
| SCOD      | gr/L | 114 ± 3.60                             | 87.7 ± 13.00                      | 23.0%    |
| Solubilization | % | 43.3 ± 2.97                      | 56.5 ± 4.49                      | 30.2%    |

From the 15 days of the pilot-scale experiment, this research analyzed the effects of thermal pre-treatment on substrate and digestate of AD pilot reactor. VS of pre-treated substrate reduced
significantly to 19%. It happened because there was an organic material’s cell membrane disintegration [7].

VS concentration of digestate after 15 days on the substrate pre-treatment operational also showed a significant reduction to 7.6% besides due to the pre-treated substrate and was already sufficient amount of microorganisms that decomposed substrate inside the reactor.

On the other hand, COD also showed a reduction of 41%. This result was higher compared to Naran et al., [20] with the efficiency of removal COD was 5% by using thermal pretreatment. The possible reason for the higher reduction of COD in this study due to the breakage of chemical bonds, including VFA’s, polysaccharides, and proteins, by providing external energy in the form of heat [8].

SCOD has been analyzed because it can be used as a performance indicator of the digestion process in AD. It reflects the amount of soluble organic matter present in the substrates in the form of dissolved organic matter [8]. SCOD in the pilot scale experiment showed an enhancement of SCOD up to 43.3% compared to un-pre-treated. The solubilization also increased up to 56.5%. The increasing solubilization could enhance the capability of microorganisms to treat the substrate effectively in order to reduce COD. Furthermore, it might be increased in the production of digestate due to the transformation of solid to liquid in FW.

![Figure 1](image_url)

**Figure 1.** Results Comparison between BMP Process and AD Process

Lastly, the comparison between BMP Process and AD Process in Figure 1. Overall, AD reactor had more effective substrate treatment than BMP. It is shown by the higher value of all parameters between BMP and AD reactor. The differences were also significant as shown in the white box in Figure 1. The greatest difference was the VS reduction that indicated the degradation in AD reactor was more effective than in the BMP process. The efficiency of substrate treatment in the AD reactor improved which could be mainly due to the presence of the continuous stirrer in the pilot-scale reactor which helped maximum contact between microorganisms to treat the substrate.

**4. Conclusion**

The BMP process showed that a significant result on thermal pre-treatment on the substrates to be treated in AD reactor based on examination of TS, VS, COD, SCOD, solubilization, and biogas production (p<0.05). The most effective temperature in biogas production was 70 °C, as well as resulting in the highest solubilization. It will break long chained or otherwise complex compound into smaller chained molecules for easier digestion. This will help the microorganism to degrade COD compare to thermal pre-treatment with a temperature of 90 °C and 120 °C. In addition, thermal pre-treatment at the temperature of 70 °C will also enhance biogas production in BMP process. Thus, thermal pre-treatment at 70 °C can be used to obtain a higher solubilization rate and biogas production.
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