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ANAEROBIC CO-DIGESTION OF OIL PALM FROND WASTE WITH COW MANURE FOR BIOGAS PRODUCTION: INFLUENCE OF A STEPWISE ORGANIC LOADING ON THE METHANE PRODUCTIVITY

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Abstract: *Anaerobic co-digestion of oil palm frond waste with cow-manure was evaluated. The study aimed to evaluate a stepwise organic load with an increase solid concentration to the on-going anaerobic digestion of cow manure. The anaerobic digestion process was operated in continuous mode under the mesophilic condition. Results showed that the maximum methane productivity of 1700 ml* $CH_4 \cdot day^{-1}$ *was obtained when the anaerobic co-digestion of OPFW and cow manure was loaded with the substrate concentration between 4 and 8% TS. The pH culture dropped dramatically from 6.9 to 6.3 when substrate concentration was increased from 10 to 12% TS. The acidic pH had restricted the conversion of organic materials in which the COD removal was less than 25% removal. This study is exceedingly notable for the industrial development of waste management processes, which handle and treat tons of organic wastes daily.*

Key words: *methane yield, substrate overloading, organic matter conversion.*

1. Introduction

Currently, some countries in the Southeast Asia, such as Indonesia and Malaysia have been the leading palm oil-producing countries in the world even though the palm oil production is growing rapidly in Africa at which the oil palm tree

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is originated [36]. Indonesia and Malaysia are the largest producers of palm oil in the world, and accounted for about 85% of the worlds palm production [31], [38]. The United States Department of Agriculture noted that Indonesia has been the world's largest producer and exporter of palm oil. This is because it has expanded million hectares of palm area spread along the islands of Sumatera, Kalimantan, Sulawesi and Papua [4], [20]. An increase in palm oil production followed with the expansion of oil palm cultivation has generated some issues towards the sustainability and environmental impacts of oil palm plantations, notably concerning in deforestation, biodiversity and air pollution [3], [5].

Oil palm frond waste (OPFW) is one of the primary waste products in the oil palm plantation and normally it is burned during the harvesting and maintenance periods. This practice obviously may cause environmental problems such as air pollution in the areas of oil palm plantation and factory, and sometimes burning the waste could lead to forest fires [7], [25]. OPFW is wood lignocellulosic biomass, which can be used as renewable energy feedstock for biogas production since it could be biologically processed through anaerobic digestion [30].

Biogas is produced from different types of biomass including energy crops, wastewaters (i.e. manure, municipal, industrial and domestic wastewater), forestry residues and agricultural crop residues. However, the conversion rates and/or biodegradation efficiency of the various waste streams would be different, which highly depends on their composition [11]. Some feedstocks containing soluble and insoluble carbohydrates (i.e. sugars and starch), lipids and proteins are biodegradable while lignocellulosic biomass feedstocks containing cellulose are not easily degraded during the process of anaerobic digestion. This is because lignocellulosic biomass feedstock has lignin contain that may hinder hydrolysis, and thereby could not be easily converted to methane as the main product of anaerobic digestion. To enhance biodegradation efficiency of lignocellulosic biomass, the feedstock should be pre-treated and/or reduced its size prior to loading into the anaerobic digester [2], [12].

Since the oil palm plantations continually generate tons of oil palm frond waste for producing palm oil, utilizing the waste as bio-energy feedstock would be highly significant to cut the greenhouse gas emissions caused by burning the waste and/or improper decomposition of the organic waste [24], [32]. Anaerobic digestion is an established technology that could be effectively used for treating the waste and converting it into useful end-products (i.e. biogas and bio-fertilizer). Some studies revealed that the anaerobic digester performances in the process of conversion the feedstock into methane are greatly affected by operational parameters [37], such as hydraulic retention time [14], temperature, agitation and organic loading rate [13], [15], [22]. Choosing the types of the operational parameters of the reactor should consider some aspects, such as the current condition of the reactor and the type of feedstock used and its availability [16], [19]. This is highly important in order to ensure that the anaerobic digester could operate sustainably, and could reduce the operational costs.

The current study deals with the production of methane from anaerobic co-digestion of oil palm frond waste with cow manure. A stepwise organic solid loading was applied to investigate the maximum feasible organic solid concentration added to the on-going process of anaerobic digester. The effects of the organic solid concentration on the total methane production, the yield of methane and the organic solid and nitrogen removals were also evaluated.

2. Materials and Methods

2.1. Material Preparation

Oil palm frond waste (OPFW) used as a co-substrate for this research was taken from a palm oil mill in PPKS Bukit Sentang, Babalan-District of Langkat, North Sumatera. The collected dried feedstock was milled by using a laboratory grinding mill with the average particle size of 2.5 ± 0.5 mm prior to feeding into the reactor. Cow manure used as the main substrate for the experiment was collected from the cow farming at Limpok, Darussalam-Banda Aceh, Indonesia. The manure was screened to get rid of any contaminants (e.g. stone, sands, gravels, plastics, woods and metals), and kept it into the refrigerator with the temperature of 4.5 ± 0.5°C until using it.

2.2. Experimental Design and Procedures

The experiments were carried out at the Laboratory of Post Harvest Technology and Bioprocess, Department of Agricultural Engineering, Syiah Kuala University. Some sample analysis measurements were conducted at the Institute for Research and Standardization of Industry, Banda Aceh. Two identical transparent acrylic bioreactors were utilized for running the process of anaerobic digestion. The two reactors were steadily agitated at 100 rpm. The first reactor was used for the control reactor performing anaerobic digestion of cow manure alone while the second reactor was utilized for operating the process of anaerobic co-digestion of oil palm frond waste with cow manure. Both reactors were operated under the steady state condition in which the operational temperature was kept at the mesophilic condition with the temperature of 35 ± 1°C by using thermostatic water bath.

Each reactor was set up with the working volume of 4 litres. As the process of anaerobic digestion was operated under the semi-continuous mode, the organic loading rate applied to the reactors would be based on the hydraulic retention time (HRT) as well as the working volume applied. To accomplish the semi-continuous process, the HRT set for the experiment was 25 days. Hence, the organic loading rate introduced into the anaerobic reactors was 160 $ml.day^{-1}$. The digesters used were equipped with the sample ports of influent as well as effluent on the top of each reactor.

To evaluate the reactor performance, samples of the effluent were taken daily for further analysis. To ensure the anaerobic digesters performed stably and/or avoid the digester failure, pH of the influent as well as effluent was monitored during the feeding and wasting periods. During the digestion process, pH was not controlled. Hence, no bases and/or acids were added to the anaerobic digestion culture. Methane produced from the anaerobic digesters was measured every day by utilizing the gas meter according to the method of water displacement.

Biogas generated from the anaerobic reactors was purified with 500 ml of a 4 M NaOH solution. The solution was filled into the filter flask. The flasks containing alkaline solution were placed and/or connected between the anaerobic reactors and the gas meters. Hence, the measured gas appeared in the gas meter was methane. Before running the experiments, the culture was acclimatized to the anaerobic as well as mesophilic environments until it reached at the steady state condition. During the period of acclimatization, no substrates and solutions (acids and/or bases) were added to the reactors, and also no effluents or culture were withdrawn.

2.3. Investigation of Loading Characters

To investigate and evaluate the effects of a stepwise addition of organic solids to the on-going anaerobic digestion of cow manure co-digested with oil palm frond waste, chemostat tests were set up by using continuous operating reactors. The process of anaerobic digestion lasted for two cycle of HRT, which was about 50 days of incubation. Before starting the experiments, each culture was acclimatized for 10 days under the anoxic and mesophilic conditions. During this period, there were neither substrates added to the reactors nor effluent withdrawn from the reactors [14].

Evaluating the effects of various organic solid concentrations loaded to the on-going process of anaerobic co-digestion of the OPFW and cow manure was conducted after 10 days of incubation. First, started at day 11 the co-digestion reactor was loaded with 0.5% TS of the milled OPFW for 5 days. At day 16, the reactor was loaded with 1% TS of the ground OPFW for 5 days of incubation. Started from day 21 to day 25, the digester was fed with 2% TS of the ground OPFW. From day 26 to 30, the co-digestion reactor was loaded with 4% TS of the milled OPFW. Then, from day 31 to 35, the digester was loaded with 8% TS of the milled OPFW. From day 36 to 40, the digester was added with 10% TS of the milled OPEFB. The last stage of incubation, started from day 41 to the rest of experiment period (day 5), the digester was loaded with 12% TS of the ground OPFW. During the tests, pH of the culture was not maintained and/or adjusted, suggesting that no alkaline and/or acids were added to the anaerobic reactors.

2.4. Analytical Methods

Samples of influent and effluent were taken for analyzing parameters, including pH, alkalinity, chemical oxygen demand (COD), total dissolved solids (TDS), total solids (TS), total kjedahl nitrogen (TKN) volatile solids (VS), moisture content (MC). All parameters were analyzed based on the Standard Method [34]. To assess the availability of organic materials in the culture and their conversion, the measurement of organic composition and removal analysis was carried out. To evaluate the performance of anaerobic digesters, the analysis of biodegradation efficiency were carried out the assessment of COD removal as well as VS reduction. Daily methane production rates were measured according to the volumetric methane produced per unit of time. Evaluation of substrates uptake as well as methane formation during the process of anaerobic digestion were carried out via methane yield measurement in which the yield was based on the cumulative methane produced per unit of volatile solids added [12], [21].

3. Results and Discussions

To evaluate the methane potential and biodegradability of the OPFW and cow manure, two completely mixed laboratory-scale reactors were set up. The two identical reactors were operated continuously under the mesophilic condition. Since the performance of anaerobic co-digestion highly depends on the characteristics of the substrates utilized, analysis of the physical and chemical properties was carried out in order to assess the potential and limitation of the organic matter content of the substrates loaded. The physicochemical characteristics of the substrates utilized were presented in Table 1.

Physicochemical properties of substrates Table 1

Parameters	Unit	Cow manure	Oil palm frond waste
Total solids	%	1.88	18.44
Volatile solids	%	23.73	82.96
Moisture content	%	98.12	81.56
Total kjedahl nitrogen	$mg \cdot l^{-1}$	400	-
Chemical oxygen demand	$mg \cdot l^{-1}$	9100	-
Alkalinity	$mg \cdot l^{-1}$	2600	-
Total dissolved solids	$mg \cdot l^{-1}$	2720	-
pH	-	6.81	-

Results revealed that the cow manure used for anaerobic digestion (AD) had pH of 6.81, which was somewhat optimal for AD. The use of OPFW as a co-substrate in the process of anaerobic digestion of cow manure could potentially increase more methane production in comparison to the digestion of cow manure only. This is because the OPFW had a significant amount of organic matters represented in the high percentage of volatile solids, which was about 83% VS in comparison to the volatile solids of cow manure that was only about 24% (Table 1). This suggested that the use of OPFW as co-substrate would potentially increase methane production during the process of anaerobic digestion.

The current study showed that the use of OPFW as a co-substrate could potentially stabilize the process of anaerobic digestion. This is because the influent of anaerobic co-digestion of OPFW with cow manure possessed higher alkalinity (3950 $mg \cdot l^{-1}$) than the alkalinity of solely cow manure (2600 mg/L). As presented in the Table 2, the reactor processing anaerobic co-digestion of OPFW and cow manure had higher organic materials represented in high COD (10000 $mg \cdot l^{-1}$) and VS (82%) content in comparison to the reactor digesting cow manure alone, which only had COD of 9127 $mg \cdot l^{-1}$ and VS of 44%. Besides, the results revealed that anaerobic co-digestion of OPFW and cow manure had about four times higher organic loading rates (1.6 g·VS·L^{-1}·day^{-1}) in comparison to the anaerobic digester processing merely cow manure, which had a lower amount of organic matters added to the reactor (0.4 g·VS·L^{-1}·day^{-1}). This suggested that the addition of OPFW to the anaerobic digestion of cow manure would likely enhance methane productivity and optimize the performance of anaerobic digestion process.

Influent of anaerobic reactors Table 2

Parameters	Unit	Control	Anaerobic co-digestion
Total solids	%	2.39	4.68
Volatile solids	%	44	82.92
Moisture content	%	97.61	95.3
Total kjedahl nitrogen	mg·l^{-1}	413.4	434
Chemical oxygen demand	mg·l^{-1}	9127	9900
Total dissolved solids	mg·l^{-1}	3050	3240
pH	-	6.83	6.82
Alkalinity	mg·l^{-1}	2600	3980
Organic loading rate	g·VS·L^{-1}·day^{-1}	0.421	1.552

Results of the study showed that both control reactor and anaerobic co-digestion reactor had low methane productivity at the early ten days of the anaerobic digestion process. This is because the microbial culture of the two reactors still ran into the acclimatization process. During this period, both culture went through the lag phase in which the microbe involved adapted themselves to the anaerobic conditions for their growth. In this stage, both control and test reactors produced small amount of methane, which was about 30-50 ml per day. A significant change occurred at the eleventh day of the digestion process when the anaerobic co-digestion reactor was loaded 0.5% TS of the OPFW in which the methane production increased gradually from 50 to 280 ml.day^{-1}. When the substrate concentration was increased from 1 to 2 %TS in the anaerobic co-digestion of OPFW and cow manure, the methane production increased significantly from 475 to 1310 ml.day^{-1} (Figure 1). This suggested that the production of methane in the process of anaerobic digestion of cow manure could be significantly increased by loading an ample amount of organic materials of the OPFW as a co-substrate.

Methane generated from the reactor of anaerobic co-digestion of OPFW and cow manure still gradually increased from 1500 to 4000 ml.day^{-1} when the substrate concentration was increased from 4 to 8%. Methane production decreased from 1700 ml.day^{-1} to 900 ml.day^{-1} when a 10% of the substrate concentration was loaded into the co-digestion reactor from day 36 to day 40 of the digestion process (Figure 1). At this stage, pH of the anaerobic co-digestion culture dropped dramatically from 7.00 to 6.67 (Figure 2), indicating that organic acids built-up and accumulated in the digester. The addition of 12% of the substrate concentration from day 41 to day 50 to the on-going reactor had aggravated anaerobic co-digestion process in which pH of the culture dropped sharply from 6.7 to 6.3 (Figure 2). Low pH in the reactor was caused by organic acids build-up. This condition could inhibit methane production, and lead to the digester failure. This suggested that acidic condition in the anaerobic reactor cannot be recovered when fresh manure added with high amount organic materials, and the addition of high amount of degradable materials should be avoided when pH of the reactor dropped lower than the neutral level.

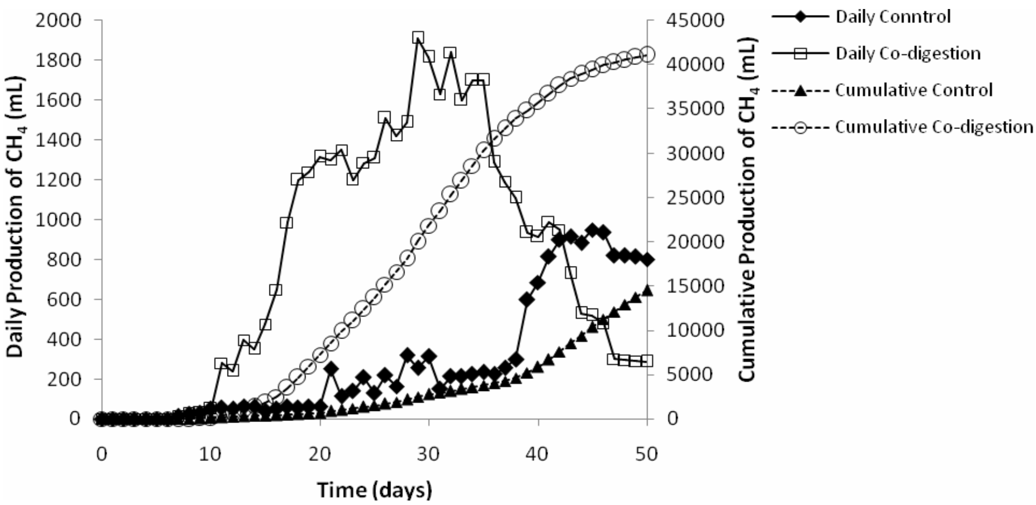

Fig. 1. *Daily and cumulative methane gas production last for two cycles of HRT*

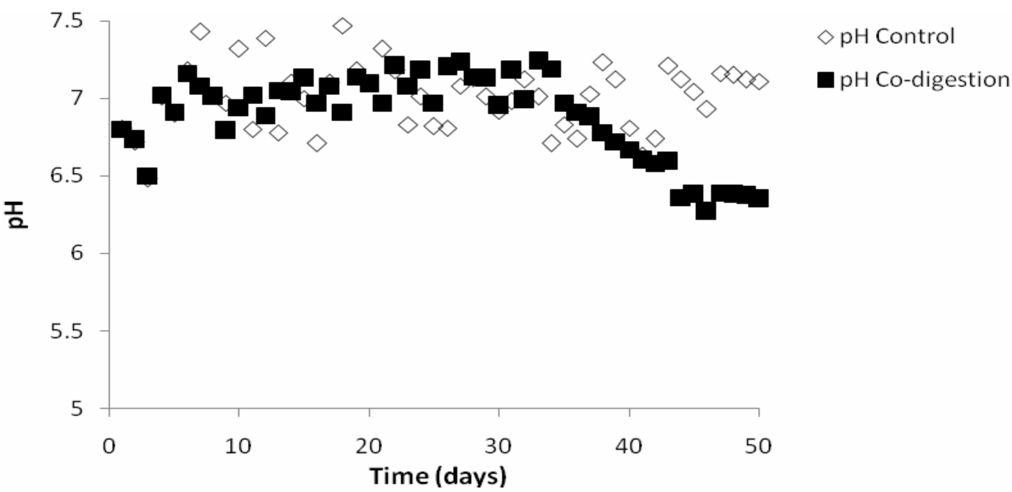

Fig. 2. *Changes in pH of control and co-digestion reactors*

Results revealed that the effluent pH of the anaerobic co-digestion reactor was more acidic (6.35) than the effluent pH of the control reactor (7.10) that was quite neutral (Table 3). This is because a significant amount of organic matters was added to the digester. The results of the experiment also showed that organic loading rate applied for the co-digestion reactor was about four times higher (1.6 $g \cdot VS \cdot l^{-1} \cdot day^{-1}$) the organic loading rate used in the control reactor that was only about 0.4 $g \cdot VS \cdot l^{-1} \cdot day^{-1}$.

Effluent data of anaerobic digestion processes Table 3

Parameters	Unit	Control	Anaerobic co-digestion
Total solids	%	1.87	8.73
Volatile solids	%	23.73	41
Moisture content	%	98.13	91.27
Total kjedahl nitrogen	$mg \cdot l^{-1}$	380	411
Chemical oxygen demand	$mg \cdot l^{-1}$	7237	7600
Total dissolved solids	$mg \cdot l^{-1}$	2590	3221
Alkalinity	$mg \cdot l^{-1}$	2220	3210
pH	-	7.10	6.35

As shown in Table 4, results of biodegradation efficiency analysis showed that methane productivity of the anaerobic co-digestion was almost three times higher (823 ml $CH_4 \cdot day^{-1}$) than the methane productivity of the control (290 mL $CH_4 \cdot day^{-1}$). Even though the concentration of organic solid matters was increased stepwise, the conversion rate of organic solid materials in the anaerobic co-digestion reactor was almost two times higher (2% VS reduced per day) than the conversion rate of the control reactor (1.2% VS reduced per day). Besides, within 50 days of the digestion process, the reduction of organic matters represented in volatile solids (VS) reduction of the anaerobic co-digestion was at about 86%. This suggested the OPFW is still considered as a feasible and biodegradable feedstock used for co-substrate in the process of anaerobic digestion of cow manure in order to enhance methane production.

Biodegradation efficiency of anaerobic digesters Table 4

Parameters	Unit	Control	Oil palm frond waste co-digested with cow manure
COD removal	%	20.71	23.23
Nitrogen removal	%	8.08	5.30
Volatile solids reduction	%	60.27	85.7
Cumulative methane production	ml CH_4	14552	41124
Methane productivity	ml $CH_4 \cdot day^{-1}$	291.04	822.48
Methane yield	ml $CH_4 \cdot g^{-1} \cdot VS^{-1}$	345.95	264.93

Some studies mentioned that pH is an important parameter that significantly affects the process of anaerobic digestion [17], [35], [40]. The present study showed that cow manure used as the main substrate had pH close to the neutral level (pH 6.81) in which the pH was somewhat optimal for anaerobic digestion [9]. The authors reported that the optimum pH in anaerobic digester should be in the range from 6.8 to 7.2. This suggested that the use of cow manure in the current study should be feasible for running anaerobic digestion.

Some studies revealed that the use of manure as a substrate could enhance the digestion process as it could help accelerate the decomposition and degradation process of the organic compounds [18], [44]. This is due to the fact that manure typically contains various types of microorganisms that could speed up the digestion processes [18]. However, the use of cow manure as a single substrate would be highly risky, and lead to unstable and/or imbalanced process of the anaerobic digestion [29]. This is because the digestion of manure normally could generate ammonia build-up, and increase pH culture more than optimal level. Yenigun and Demirel [42] mentioned that ammonia is considered as a potential inhibitor during anaerobic digestion, especially when digesting complex type of substrates such as manure and organic fraction of municipal solid waste. Some studies added that ammonia accumulated in anaerobic digester could toxify methanogens and hinder the formation of methane during the methanogenesis [27], [29]. This is because high amount of ammonia tended to generate basic and/or alkaline conditions in the anaerobic digester. Hence, the use of OPFW as co-substrate would balance the pH level and potentially stabilize the digestion process and could optimize methane production.

The influent characteristics of the anaerobic co-digestion culture presented in Table 2 revealed that the culture would be highly potential for enhancing methane productivity. This is because that the pH culture was very close to the neutral level (6.82), which was considered as a suitable level for anaerobic digestion. The current result was in agreement with the study by Cioabla et al. [9] revealing that pH culture ranged from 6.8 to 7.2 was highly feasible for processing anaerobic digester. Ali Shah et al. [1] added that the optimal pH range for methanogens lied between 6.8 and 7.5, those range could optimize methane production during anaerobic digestion.

The OPFW co-digested with cow manure increased organic matter content in the anaerobic digester denoted with high percentage of volatile solids (83% VS), while the control reactor processing cow manure alone had low content of organic matters, which was only 44% VS. Some studies mentioned that the reliability of organic matters in the feedstock used may relatively affect the availability of the organic compounds in the anaerobic digester [28]. This is highly important since the concentration of the organic compounds would remarkably effect on the performance of anaerobic digester [15].

The gas production reached a peak at 29 days of the incubation period, which was about 1910 ml CH_4. The results showed that the logarithmic phase occurred from day 11 to day 30, in which methane production increased gradually from 280 to 1900 ml CH_4. A stationary phase occurred between 25 and 35 days of digestion in which the methane production was stable between 1500 and 1700 ml CH_4 (Figure 1). Some studies suggested that the microorganisms living during the exponential or logarithmic phase are more likely to be affected by the environmental changes in comparison to the microbes living under the stationary phase [23], [33]. The stationary phase occurs not only due to the depletion of nutrients and/or substrates but also due to the accumulation of organic acids in the digester. Some studies added that organic acids build up in the digester could significantly inhibit microbial growth and restrict the formation of the end-product [10], [39].

When the organic materials loaded to the digester were increased from 8 to 10% TS, the sign of inhibition was observed in which the gas production decreased from 1700 to 900 ml CH_4. During this period, pH culture dropped from 6.97 to 6.67. Cioabla et al. [9] revealed that anaerobic digestion process could tolerate a range pH of 6.5 up to 8.0. Liu et al. [26] found that an upset anaerobic digester may occur when the pH dropped lower than 6.1 and increased more than 8.3. In this current study, low pH may cause imbalance reactions between organic acid formation and organic acid utilization in the digester.

An increase of proton concentration in the digester due to organic acid accumulation had lowered buffer capacity of the anaerobic co-digestion culture in which the alkalinity decreased from 4000 to 3200 $mg \cdot l^{-1}$. This condition inhibited anaerobic digestion process and restricted methanogens to grow and convert volatile fatty acids (VFA) into methane gas [41]. The authors mentioned that acetic acid was the major VFA that inhibit methanogenesis. Zhang et al. [43] found that neither acetate nor acidic pH, but free acetic acid formed in the digester would be the main factor for the inhibition of methanogenesis in mesophilic mixed culture fermentation.

The current study revealed that high concentration of organic materials (> 8% TS) loaded to the digester would potentially generate digester upset due to an accumulation of organic acids. The acidic pH caused by organic acids build up may slow down the conversion of organic matters of OPFW into methane. In this study, the conversion efficiency of organic matters represented in COD removal was less than 25% removal. This suggested that substrate overloading applied to the digester of anaerobic co-digestion of cow manure and OPFW may completely inhibit the decomposition of organic matters into methane. This finding is in agreement with the study by Bujoczek et al. [6] revealing that the conversion efficiency of organic matters would decline once the feeding of organic loads to the reactors was lowered. Zuo et al. [45] reported that with increasing organic loads, total VFA concentration would increase, followed by the decrease of pH culture from 6.4 to 5.2. Chen et al. [8] added that substrate overloading applied to the digesters would result in the process imbalance of anaerobic digestion, generate VFA accumulation, and ceased methane production.

4. Conclusions

This study revealed that the addition of OPFW to the on-going anaerobic digestion of cow manure produced higher methane productivity (823 ml $CH_4 \cdot day^{-1}$) compared to the anaerobic digestion of cow manure alone (291 ml $CH_4 \cdot day^{-1}$). A drop of pH from 7.0 to 6.6 in anaerobic co-digestion culture occurred when the solid concentration of the substrate was increased from 8 to 10% TS. The low pH and acidic anaerobic co-digestion culture resulted in the restriction of organic matter conversion, and inhibit methanogenesis leading to the decrease of methane production.

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