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# Improved Anaerobic Treatment of Palm Oil Mill Effluent in a Semi-Commercial Closed Digester Tank with Sludge Recycling and Appropriate Feeding Strategy

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## ABSTRACT

Anaerobic treatment of palm oil mill effluent (POME) in a semi-commercial closed digester tank with sludge recycling was studied using different feeding strategies; one fixed at every three hour and another at every six hour. The organic loading rate (OLR) was increased step-wise and stopped once inhibition on methane production occurred. The chemical oxygen demand (COD), feeding rate, hydraulic retention time (HRT), OLR, and sludge recycling ratio were measured. Performance was based on the COD removal efficiency and methane yield, while stability was assessed in terms of total volatile fatty acids (VFA) accumulation, total VFA-to-alkalinity ratio (VFA:Alk) and food-to-microorganisms ratio (F/M ratio). The feeding strategies, at every three hour and six hour, gave satisfactory COD removal efficiency of higher than 90%, but the latter feeding strategy gave a more stable process with total VFA concentration recorded below 500 mg $L^{-1}$ and VFA:Alk ratio of less than 0.3 at maximum OLR of 6.0 kgCOD $m^{-3}$ $d^{-1}$. The treatment period could also be extended up to 100 days without any obvious problems.

**Keywords: Anaerobic treatment, biogas, feeding interval, methane, palm oil mill effluent, sludge recycling**

## INTRODUCTION

Malaysia is blessed with suitable climatic and geographical factors for the cultivation of oil palm scientifically known as *Elaeis guineensis Jacq*. The palm oil industry is very important to Malaysia and it has contributed significantly to the country's gross domestic product (GDP). The export earnings from palm oil, palm kernel oil, and its products in 1998 amounted to almost US$5.6 billion, equivalent to 5.6% of the country's GDP. Today, Malaysia is the world's largest producer and exporter of palm oil (Yusoff, 2006). However, despite the high economic returns, the generation of liquid waste or palm oil mill effluent (POME) is also huge. It was estimated that for every tonne of fresh fruit bunch processed, between 0.5 and

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0.75 tonne of POME is produced (Yacob *et al*., 2006). POME is generated from the combination of sterilization, clarification and hydrocyclone washing processes during palm oil processing (Hassan *et al*., 2004). More than 85% of the palm oil mills in Malaysia use the conventional pond systems for the treatment of POME due to its lower operating costs (Najafpour *et al*., 2006). In the future, anaerobic treatment of POME coupled with methane gas recovery will be the preferred choice for sustainable development of the palm oil industry. In addition to the production of methane gas which can be used for electricity generation, the Certified Emission Reduction (CER) mechanism could also be triggered once the project is registered as a Clean Development Mechanism (CDM) project under the patronage of the United Nations Framework Convention on Climate Change (UNFCCC). Anaerobic treatment of POME by a closed anaerobic digestion system offers several advantages in comparison with other treatment technologies such as lower energy requirements with no aeration, producing methane gas as a valuable end product and generating sludge from the process which can be used as fertilizer or for land application (Poh and Chong, 2009).

Table 1 shows a comparison of various technologies for treating POME. The technologies (except for 500 $m^3$ digester tank reactor studied by Yacob *et al*., 2006) exhibited high COD removal efficiency, but the experiments were performed using only laboratory scale digesters and the POME used might not represent the actually characteristics of POME produced directly from the mill. Meanwhile, the large pilot scale technology available in Malaysia is the 500 $m^3$ semi-commercial closed digester tank which was installed in 2005 for FELDA Palm Industries Sdn. Bhd. in Serting Hilir Palm Oil Mill. The digester was commissioned in 2005 and a series of publications have been produced since then (Yacob *et al*., 2006; Sulaiman *et al*., 2009). This study is part of the continuation of those studies on improving the anaerobic treatment of POME for higher methane gas production.

TABLE 1
Different types and scales of anaerobic digester for the POME treatment and performance

| Type of reactor | HRT (d) | Inlet COD concentration ($kgm^{-3}$) | OLR ($kgCODm^{-3}d^{-1}$) | COD removal (%) | References |
|---|---|---|---|---|---|
| Up-flow anaerobic sludge fixed film | 1.5 | 26.21 | 17.47 | 90.2 | Najafpour *et al*. (2006) |
| Digester tank | 10 | 56.45 | 5.55 | >90 | Yacob *et al*. (2006) |
| Modified anaerobic baffled | 3 | 16 | 5.33 | 77.3 | Faisal and Unno (2001) |
| Anaerobic hybrid | 3.5 | 56.6 | 16.20 | 92.3 | Borja *et al*. (1996) |
| Anaerobic filter | 1.0 | 10.0 | 10.0 | >90 | Borja and Banks (1994a) |
| Anaerobic fluidized bed | 0.25 | 2.5 | 10.0 | >90 | Borja and Banks (1994a) |
| Up-flow anaerobic sludge blanket | 4 | 42.5 | 10.6 | 96 | Borja and Banks (1994b) |
| Immobilized cell | 6.2 | 69 | 10.6 | 96.2 | Borja and Banks (1994c) |
| Stirred tank | 5.6 | 70 | 12.60 | 97 | Cail and Barford (1985) |

The digester is of a simple and straight-forward design and hence suitable for operators with low level technical competency. The digester has recently been equipped with a sludge settling tank so that the sludge could be recycled in order to maintain a higher cell mass inside the digester and that the production of methane gas could also be improved. Many studies have reported that the operational stability of the digester could be improved in the treatment of POME and other organic wastes by recycling the sludge (Najafpour *et al*., 2006; Setiadi *et al*., 1996; Faisal and Unno, 2001). Najafpour *et al*. (2006) utilized a high recycle ratio of 11.25 to eliminate high organic over loading and supply alkalinity by blending the fresh feed with low COD and high alkalinity recycled stream. Meanwhile, the study by Setiadi *et al*. (1996) showed that a recycle ratio of more than 15 was required to maintain the pH of the anaerobic process higher than 6.8 without alkalinity supplementation. A higher recycle ratio of 30 was adopted by Faisal and Unno (2001) in stabilizing the modified anaerobic baffled reactor operation. Thus, the main objective of this study was to evaluate the performance of a large semi-commercial closed digester with sludge recycling and different feeding strategies for the anaerobic treatment of POME. In this study, the focus is on anaerobic treatment of POME, using a large 500 $m^3$ semi-commercial closed digester tank with sludge recycling from a newly installed settling tank and the feeding was performed at different time intervals so as to evaluate the digester performance and stability. The digester's performance parameters were measured in terms of chemical oxygen demand (COD) removal efficiency, methane yield, and digester stability in terms of the total accumulation of volatile fatty acid (VFA).

## MATERIALS AND METHODS

*The Set-up of the Closed Digester Tank*

*Fig. 1* illustrates the schematic diagram of the 500 $m^3$ semi-commercial closed digester tank. Raw POME was directly pumped from the mill and stored in a 50 $m^3$ holding tank to ensure continuous supply and consistent characteristics. A centrifugal pump was used to feed the digester. The POME feeding volume was measured online by a mass flow meter and recorded by Endress+Hauser Ecograph. During the feeding process, an equal volume of the treated effluent inside the digester was displaced and let to flow out into the settling tank, where the solids were trapped and recycled into the digester at 6 $m^3$ $d^{-1}$. After feeding, the content of the digester was mixed for 30 min using a mixing pump to ensure a good contact between substrates and micro-organisms. The generated biogas in the collection chamber was measured online by a mass flow meter before it was sent for storage.

*Source of Raw POME, Seed Sludge and Recycled Sludge*

Raw POME was directly obtained from the palm oil mill located beside the plant by direct pumping. The digester was seeded using the sludge from the existing 3600 $m^3$ open digester tank and diluted to give an initial total solid of 5% inside the digester (Yacob *et al*., 2006). The sludge from the settling tank was allowed to settle for 2-3 hours before it was recycled in the digester. After that, the recycled sludge was analyzed and characterized by a lower total COD content, high solids content (4-6% of TS), high alkalinity (2,000-4,000 mg $L^{-1}$ $CaCO_3$), neutral pH (7.0±0.2), high nitrogen content (286-300 mg $L^{-1}$ of TKN), low oil and grease (150-183 mg $L^{-1}$), high lignin content (2,280-2,350 mg $L^{-1}$), and high ash content (28,098-30,350 mg $L^{-1}$).

*Experimental Procedures*

In this study, two set of experiments were conducted; one with sludge recycling and the feeding interval was fixed at every three hours and another set was also with sludge recycling but the feeding interval was fixed at every six hour. The sludge recycling rate was fixed at 6 $m^3$ $d^{-1}$. The OLR was increased step-wise and the digester performance and stability were also evaluated. Table 2 shows the summary of the feeding profiles in terms of COD concentration of the raw POME, feeding rate, HRT, OLR, and

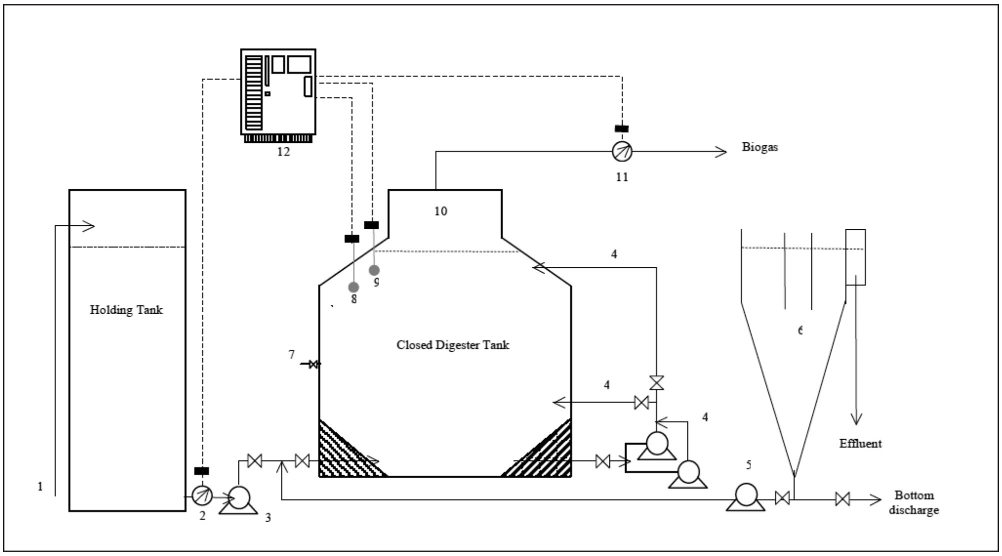


*Fig. 1: Process flow diagram of the 500 m$^3$ semi-commercial closed digester tank; 1-Raw POME inlet; 2-Mass flow meter; 3-Centrifugal pump; 4-Mixing pump system; 5-Sludge recycling pump; 6-Settling tank; 7-Sampling port; 8-pH probe; 9-Temperature probe; 10-Biogas collection chamber; 11-Biogas mass flow meter; 12-Endress+Hauser Ecograph system*

sludge recycling ratio for both the experiments. For the three hours' feeding interval experiment, the operational days only lasted for 46 days, whereas for the six hours' feeding interval experiment, the operational days improved and went on up to 100 days. The COD concentrations of the raw POME utilized for both experiments were almost consistent; however, the maximum feeding rate applied was different because different maximum OLR were achieved in both the experiments. Due to different feeding rates applied, the HRT was also different. The sludge recycling rate was fixed at 6 m$^3$ d$^{-1}$ and the recycling ratio was varied daily.

*Chemical Analyses and On-line Data*

Analysis for Chemical Oxygen Demand (COD), total volatile fatty acids (VFA), total solid (TS), volatile suspended solid (VSS), pH, alkalinity, total kjeldah nitrogen (TKN), lignin, and ash were performed according to the APHA standard methods (APHA, 1985). The raw POME volume feeding rate was measured online using an electromagnetic flow measuring system (PROline promag 50, Endress+Hauser, Germany) and the biogas produced was also measured online using a thermal mass flow meter (T-Mass AT70, Endress+Hauser, Germany). The online data were temporarily stored in

TABLE 2
Chemical Oxygen Demand inlet, feeding rate, HRT, OLR and sludge recycling ratio

| Experiment | Days of operation | COD inlet (mg L$^{-1}$) | Feeding rate (m$^3$ d$^{-1}$) | HRT (d) | OLR (kgCOD m$^{-3}$d$^{-1}$) | Recycling ratio |
|---|---|---|---|---|---|---|
| Experiment 1[a] | 46 | 17,100 - 82100 | 3.3-50.0 | 10.0 - 152.2 | 0.5 - 4.0 | 0.13 - 1.35 |
| Experiment 2 [b] | 100 | 28,900 - 79600 | 18.2 - 67.2 | 7.4 - 27.4 | 2.0-6.0 | 0.11 - 0.30 |

[a] Feeding of every three hours with sludge recycling, [b] Feeding of every six hours with sludge recycling

the Endress+Hauser Ecograph (Germany) and retrieved weekly for analysis. The concentration of methane was determined using a calibrated portable methane gas analyzer (XP-314A, Shin-Cosmos Electric Co. Ltd., Japan).

*Scanning Electron Microscope (SEM)*

Samples for the scanning electron microscope were fixed with 4% glutaraldehyde in 0.1 M of sodium cacodylate buffer for three changes of 10 min each. After post-fixation with 1% osmium tetroxide for 2 h, the samples were washed again with 0.1 M of sodium cacodylate buffer for three changes of 10 min each. The samples were then dehydrated in a graded acetone series of 35% for 10 min, 50% for 10 min, 75% for 10 min, 95% for 10 min, and finally three changes of 100% for 15 min each. After that, the samples were dehydrated using the critical point drying method (CPD) for 30 min. Finally, the CP-dried samples were sputter-coated with gold and examined using a Philips XL30 ESEM (Holland), operating at an accelerating voltage of 15 kV. The samples were viewed and the most prominent was selected for the analysis.

## RESULTS AND DISCUSSION

*Effects of the Three Hours' Feeding Interval on Digester Performance and Stability*

The digester performance and its stability, over 46 days of operation when the digester was subjected to a feeding interval of every three hours, are shown in Tables 3 and 4, respectively. The performance was evaluated in terms of the COD removal efficiency and methane yield. The methane yield is expressed in mass of methane, produced at a standard temperature and pressure over the mass of COD removed in a day (kg$CH_4$ kgCODremoved$^{-1}$). This experiment only lasted for 46 days and was stopped due to the lower methane production. Over this period, the OLR was gradually increased from 0.5 to 4.0 kgCOD m$^{-3}$ d$^{-1}$ in order to eliminate shock loading to the system. The strategy was proven successful, based on the satisfactory COD removal efficiency. The results showed a good treatment performance by the digester with > 90% removal. This is consistent with some previous studies on POME treatment, as reported by several researchers listed in Table 1 (Cail and Barford, 1985; Borja and Banks, 1994a; Borja and Banks, 1994b; Borja and Banks, 1994c; Borja *et al*., 1996; Faisal and Unno, 2001; Najafpour *et al*., 2006). In general, high COD removals (higher than 90% removals) were recorded using various technologies tested at various HRT (0.25 - 10 days), inlet COD concentrations (2.5 – 70 kgm$^{-3}$) and OLR (5.33-17.47 kgCODm$^{-3}$d$^{-1}$), except for the Modified Anaerobic Baffled Reactor where only 77.3% COD removal was reported. In addition, our previous studies also observed high COD removal efficiency using the same digester (Yacob *et al*., 2006; Sulaiman *et al*., 2009). This further implies the suitability of anaerobic treatment method for POME.

The methane yield showed a reducing trend towards the end of the experiment. Initially at lower OLR, a high methane yield was recorded as can be seen in the case OLR of 0.5 kgCOD m$^{-3}$ d$^{-1}$, where the methane yield was between 0.22 and 0.37 kg$CH_4$ kgCODremoved$^{-1}$. In this case, the methane yield was higher than the theoretical methane yield of 0.25 kg$CH_4$ kgCODremoved$^{-1}$. This is because when the OLR applied is low, adequate retention time is available for the micro-organisms to utilize the substrate in the digester and eventually result in higher methane production. Moreover at this stage, a higher recycling ratio was also applied to the system at 1.35 m$^3$ sludge m$^{-3}$ POME, which brought about the benefit of higher cell accumulation in the digester. The methane yield of higher than theoretical value of 0.25 kg$CH_4$ kgCODremoved$^{-1}$ or 0.35 L $CH_4$ kgCODremoved$^{-1}$ was also reported by Faisal and Unno (2001), where methane yield ranging from 0.355 – 0.420 were reported when the digester was operated at 5 – 8 days HRT, using a Modified Anaerobic Baffled Reactor and high recycling ratio. The trend of high COD removal efficiency and satisfactory methane yield continued up to OLR application of 1.5 kgCOD m$^{-3}$ d$^{-1}$. However, once the OLR was further increased to 2.0 kgCOD m$^{-3}$ d$^{-1}$, the methane yield was drastically reduced to only

TABLE 3
Digester performance parameter for the three hours feeding interval experiment and the sludge recycling ratio

| Days | Organic loading rate[a] | COD removal efficiency (%) | | Methane yield [b] | | Recycling ratio [c] |
|---|---|---|---|---|---|---|
| | | Range | Mean±SD | Range | Mean±SD | Mean±SD |
| 1-5 | 0.5 | 95-98 | 97±1 | 0.22-0.37 | 0.27±0.1 | 1.35±0.43 |
| 6-8 | 1.0 | 95-98 | 96±2 | 0.17-0.22 | 0.19±0.02 | 0.56±0.20 |
| 9-11 | 1.5 | 97 | 97 | 0.19-0.20 | 0.19±0.01 | 0.43±0.02 |
| 12-16 | 2.0 | 95-98 | 97±1 | 0.09-0.14 | 0.11±0.02 | 0.34±0.14 |
| 17-21 | 2.5 | 95-98 | 96±1 | 0.06-0.10 | 0.08±0.02 | 0.28±0.12 |
| 22-29 | 3.0 | 90-95 | 94±2 | 0.10-0.13 | 0.11±0.01 | 0.18±0.03 |
| 30-37 | 3.5 | 83-96 | 92±4 | 0.06-0.13 | 0.09±0.02 | 0.13±0.02 |
| 38-46 | 4.0 | 90-97 | 94±2 | 0.06-0.13 | 0.09±0.02 | 0.15±0.02 |

[a] unit is in kgCOD $m^{-3}$ $d^{-1}$, [b] unit in is $kgCH_4$ kgCODremoved $^{-1}$, [c] is defined as the sludge recycling rate over the volumetric feeding rate of the POME

TABLE 4
Digester stability for the three hours' feeding interval experiment

| Operation days | Organic loading rate[a] | Total VFA (mg $L^{-1}$) | | VFA:Alk |
|---|---|---|---|---|
| | | Range | Mean±SD | Average |
| 1-5 | 0.5 | 164-202 | 182±16 | 0.05 |
| 6-8 | 1.0 | 170-299 | 219±70 | 0.07 |
| 9-11 | 1.5 | 209-304 | 253±48 | 0.07 |
| 12-16 | 2.0 | 104-167 | 147±25 | 0.05 |
| 17-21 | 2.5 | 150-688 | 370±287 | 0.07 |
| 22-29 | 3.0 | 389-922 | 627±229 | 0.12 |
| 30-37 | 3.5 | 194-621 | 417±167 | 0.20 |
| 38-46 | 4.0 | 321-1291 | 742±373 | 0.28 |

[a] unit is in kgCOD $m^{-3}$ $d^{-1}$

0.11 $kgCH_4$ kgCODremoved$^{-1}$. At even higher OLR application (2.5 to 4.0 kgCOD $m^{-3}$ $d^{-1}$), the trend of low methane yield continued, i.e. at only between 0.08 and 0.11 $kgCH_4$ kgCODremoved$^{-1}$ which is only 32-44% of the theoretical yield. At this stage, the methanogenesis was inhibited and the sludge recycling rate at 6 $m^3$ $d^{-1}$ was inadequate to cater for the higher accumulation of VFA (742 mg $L^{-1}$) in the system. The recycling ratio was also lower, i.e. at only 21-25% of the initial recycling ratio. At the end of the experiment, the process was stopped to recover.

The digester's stability in terms of the total VFA accumulation and VFA:Alk ratio in the system is shown in Table 4. At a lower OLR range of 0.5 to 2.0 kgCOD $m^{-3}$ $d^{-1}$, the total VFA accumulation inside the system was low, i.e. ranging from 147 to 253 mg $L^{-1}$. This particular phenomenon is a common indication of a good VFA utilization by the methanogens for methane production. In addition, the available alkalinity is also adequate to buffer the total VFA accumulation in the system, as indicated by low VFA:Alk ratio of between 0.05 and 0.07. At this stage, the results of COD removal efficiency and methane yield were satisfactory. However, once the OLR was increased to 2.5 kgCOD $m^{-3}$ $d^{-1}$ and the maximum of 4.0 kgCOD $m^{-3}$ $d^{-1}$, the process became unstable, as indicated by the drastic increase of the total VFA inside the system. The maximum total VFA accumulation recorded in the system was 742 mg $L^{-1}$ at the end of the process before it was stopped for recovery. At this stage, the VFA:Alk ratio was increased to 0.28, which was almost the critical level of 0.3. Due to this non-conducive environment, the methane yield was lower at 0.09 kg$CH_4$ kg COD $removed^{-1}$. This phenomenon of higher total VFA accumulation, when the OLR was increased, was also observed in the study by Yacob *et al*. (2006) and Sulaiman *et al*. (2009).

*The Effects of Six Hours' Feeding Interval on Digester Performance and Stability*

In this experiment, the digester was subjected to a feeding interval of six hours which resulted in four times of feeding per day. The digester performance was evaluated in terms of the COD removal efficiency and methane yield achieved, whereas the stability was determined in terms of the total VFA accumulation and VFA:Alk ratio. These results are shown in Tables 5 and 6, respectively. The feeding interval of every six hour was found to produce a more stable anaerobic treatment process, where the total VFA accumulation was below 500 mg $L^{-1}$ even when the OLR was increased to 6.0 kgCOD $m^{-3}$ $d^{-1}$. Initially at low OLR ranging from 2.0 to 2.5 kgCOD $m^{-3}$ $d^{-1}$, the system maintained a remarkably high COD removal efficiency of 96-97% and the methane yield was also high at 0.17 kg$CH_4$ kg$CODremoved^{-1}$. The total VFA concentration in the system was also low (i.e. below 300 $mgL^{-1}$) and this phenomenon indicates a good VFA utilization by the methanogens. At this stage, it is believed that a balanced microbial population existed between acidogens and methanogens. As OLR was gradually increased to 3.0 and eventually to 4.0 kgCOD $m^{-3}$ $d^{-1}$, the methane yield was slightly reduced to 0.14 kg$CH_4$ kg$CODremoved^{-1}$ but the COD removal efficiency was maintained (i.e. higher than 90%). As discussed earlier, the reduction of the methane yield might be caused by the inhibition of methanogenesis process. At this stage, the recycling ratio was only 50% of the initial recycling ratio which might have resulted in lower cell retention in the digester to counter the VFA accumulation in the system at 224 mg $L^{-1}$. Therefore, in order to understand the effect of higher OLR on the system, the OLR was gradually increased to 4.5, 5.0, 5.5, and finally to

TABLE 5
Digester performance parameter for the six hours' feeding interval experiment and the sludge recycling ratio

| Days | Organic loading rate[a] | COD removal efficiency (%) | | Methane yield [b] | | Recycling ratio [c] |
|---|---|---|---|---|---|---|
| | | Range | Mean±SD | Range | Mean±SD | Mean±SD |
| 1-10 | 2.0 | 94-99 | 97±1.6 | 0.08-0.21 | 0.17±0.04 | 0.30±0.13 |
| 11-18 | 2.5 | 95-97 | 96±0.7 | 0.16-0.18 | 0.17±0.01 | 0.23±0.04 |
| 19-27 | 3.0 | 90-98 | 95±2.1 | 0.12-0.18 | 0.14±0.01 | 0.16±0.04 |
| 28-37 | 3.5 | 95-97 | 96±0.8 | 0.13-0.18 | 0.15±0.01 | 0.16±0.03 |
| 38-47 | 4.0 | 86-97 | 94±3.5 | 0.12-0.17 | 0.14±0.01 | 0.15±0.02 |
| 48-57 | 4.5 | 88-98 | 96±2.8 | 0.14-0.16 | 0.14±0.01 | 0.17±0.03 |
| 58-77 | 5.0 | 91-97 | 95±1.6 | 0.11-0.17 | 0.13±0.01 | 0.12±0.02 |
| 78-89 | 5.5 | 91-95 | 94±1.2 | 0.11-0.14 | 0.12±0.01 | 0.11±0.01 |
| 90-100 | 6.0 | 92-99 | 96±2.0 | 0.09-0.11 | 0.10±0.01 | 0.11±0.01 |

[a] unit is in kgCOD $m^{-3}$ $d^{-1}$, [b] unit is in kg$CH_4$ kgCODremoved $^{-1}$, [c] is defined as the sludge recycling rate over the volumetric feeding rate of the POME

TABLE 6
Digester stability for the six hours feeding interval experiment

| Operation days | Organic loading rate[a] | Total VFA ($mgL^{-1}$) | | VFA:Alk |
|---|---|---|---|---|
| | | Range | Mean±SD | Average |
| 1-10 | 2.0 | 163-506 | 284±122 | 0.08 |
| 11-18 | 2.5 | 211-308 | 268±32 | 0.08 |
| 19-27 | 3.0 | 195-375 | 293±67 | 0.08 |
| 28-37 | 3.5 | 171-414 | 255±76 | 0.10 |
| 38-47 | 4.0 | 105-386 | 224±89 | 0.10 |
| 48-57 | 4.5 | 211-517 | 343±88 | 0.15 |
| 58-77 | 5.0 | 183-515 | 336±85 | 0.15 |
| 78-89 | 5.5 | 318-583 | 432±83 | 0.20 |
| 90-100 | 6.0 | 327-726 | 500±109 | 0.28 |

[a] unit is in kgCOD $m^{-3}$ $d^{-1}$

6.0 kgCOD $m^{-3}$ $d^{-1}$. Remarkably, the system was found to maintain a satisfactory COD removal efficiency of higher than 90%. In spite of the good COD removal, the methanogensis process was inhibited due to the accumulation of VFA in the system. The methane yield was decreased to 0.09 $kgCH_4$ $kgCODremoved^{-1}$ at the end of the process, before it was stopped for recovery. This is only 36% of the theoretical yield. At this stage, the total VFA accumulation was only around 500 mg $L^{-1}$, but the VFA:Alk ratio was high, i.e. almost similar to its critical level of 0.3. At this stage, the available alkalinity was limited and inadequate to sustain a good buffering capacity to the system and therefore created non-conducive environment for methanogenesis. The sludge recycling ratio was only 0.11, which was only 37% of the initial recycling ratio. This was inadequate to provide a higher buffering capacity to the digester which suggests that a higher sludge recycling ratio is required, as adopted by Setiadi *et al*. (1996), Faisal and Unno (2001), Najafpour *et al*. (2006) and Sulaiman *et al*. (2009).

*Ratio of Food-to-Microorganisms (F/M Ratio)*

The ratio of food-to-microorganisms (F/M), expressed in mass of COD applied over the mass of mixed liquor volatile suspended solid in the digester of the entire experiments period for both the experiments, is shown in *Fig. 2*. In many studies the sludge recycling was observed to be able to provide active microorganisms to the system and maintain its pH without additional alkalinity supplementation as reported by Najafpour *et al*. (2006), Setiadi *et al*. (1996) and Faisal and Unno (2001). In addition to providing additional alkalinity and maintaining the pH of the system, the sludge also contains denser population of microorganisms responsible for biodegradation of organic substances in POME and conversion to methane gas. *Fig. 3* shows the scanning electron microscope (SEM) picture of the microorganisms in the sludge sample taken from the settling tank. It shows the existence of microorganisms in the flocs of the treated POME sludge believed to be *Methanosarcina* sp. and *Methanosaeta* sp. This is consistent with the earlier finding by Sulaiman *et al*. (2009) on a similar sample using the Fluorescent In-situ Hybridisation (FISH) technique. Both the methanogens are known to be very important for the production of methane from acetate (Robinson *et al*., 1984; Sekiguchi *et al*., 1999; Saiki *et al*., 2002; Yang *et al*., 2007). These researchers also confirmed the presence of both *Methanosarcina* sp. and *Methanosaeta* sp. in the samples of the anaerobic wastewater treatment plants.

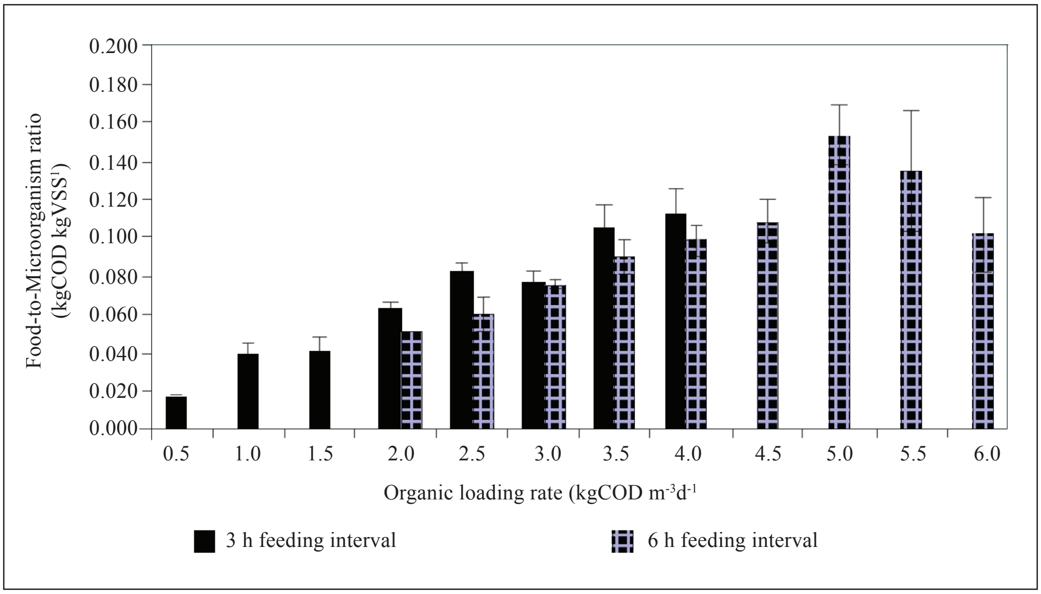


*Fig. 2: The ratio of food-to-microorganisms (F/M ratio) at different organic loading rates applied for the three hours feeding interval and six hours feeding interval experiments*

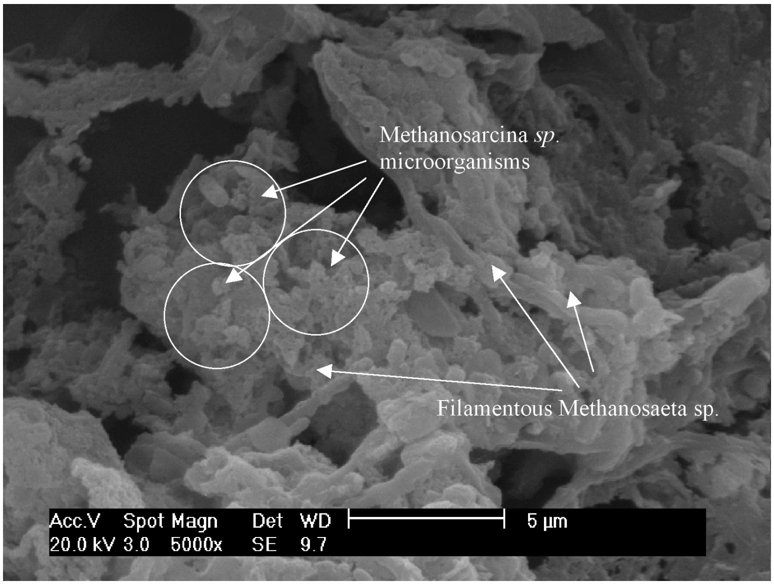


*Fig. 3: Photograph of scanning electron microscope (SEM) of the recycled sludge showing the existence of microorganisms in the flocs of POME sludge -* Methanosarcina *sp. and* Methanosaeta *sp. (magnification of 5000X)*

In both the experiments, the sludge was recycled at 6 $m^3$ in a day. The sludge recycling ratio was gradually reduced due to the higher OLR applied to the system. This resulted in higher F/M ratio at higher OLR applied, as indicated in *Fig. 2*. Generally, the F/M ratio for experiment 1 was relatively higher than experiment 2, indicating a lower concentration of microorganisms available in the system. In the three hours' feeding interval experiment, the maximum F/M ratio recorded was approximately 0.12 kgCOD $kgVSS^{-1}$ before the process was stopped for recovery due to VFA inhibition. In the six hours' feeding interval experiment, however, at the same OLR, a slightly lower F/M ratio of 0.1 kgCOD kg $VSS^{-1}$ was recorded and the methanogenesis was not yet inhibited. Moreover, it is believed that at this stage, the continuous supply of active microorganisms from the settling tank and less disturbance, due to longer feeding interval, helped to maintain the methanogenesis rate in the digester and consequently maintaining higher VFA uptake rate by the methanogens.

In the six hours' feeding interval experiment, when the OLR was increased to 5.0 kgCOD $m^{-3}$ $d^{-1}$, the F/M ratio also increased to 0.148 kgCOD $kgVSS^{-1}$. At this stage, the methane yield was further reduced, indicating a lower microorganism activity in the system. Upon further increment of the OLR (i.e. 6.0 kgCOD $m^{-3}$ $d^{-1}$), the adverse effect on methanogenesis was clearly observed where the recorded methane yield was only 0.11 $kgCH_4$ kg $CODremoved^{-1}$. At this stage, although the F/M ratio was lower, it was unable to recover due to shock loading and microorganisms' washout. Furthermore the sample of the mixed liquor volatile suspended solid (MLVSS) inside the digester might have been contaminated by the VSS of the raw POME itself, and thus resulted in a lower ratio of food-to-microorganisms. At this stage, higher sludge recycling ratio should be applied in order to maintain a higher level of microorganisms in the system which are responsible for methane production, as suggested by some researchers (e.g. Najafpour *et al*., 2006; Setiadi *et al*., 1996; Faisal and Unno, 2001; Sulaiman *et al*., 2009).

## CONCLUSIONS

This study has demonstrated the feasibility of anaerobic treatment of POME at the sludge recycling rate of 6 $m^3$ $d^{-1}$ and feeding interval of three hours or six hours. In both the experiments, the COD removal efficiency was higher than 90%. The feeding interval of six hours showed a more stable anaerobic treatment process with the total VFA concentration recorded below 500 mg $L^{-1}$ and the VFA:Alk of less than 0.3 at OLR of 6.0 kgCOD $m^{-3}$ $d^{-1}$. At the end of the treatment period, the methane yield recorded the lowest values (0.09 and 0.10 $kgCH_4$ $kgCODremoved^{-1}$) for the three hours' feeding interval and six hours' feeding interval, respectively. Future research will focus on the methane yield improvement for renewable energy capture.

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