

# ANAEROBIC TREATMENT OF POLYETHYLENE GLYCOL (PEG) -CONTAINING WASTEWATER IN A FIXED-BED REACTOR

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## ***ABSTRACT***

This paper presents the suitability of anaerobic treatment of PEG-containing wastewater as a pre-treatment in order to recover the potential energy in the form of biogas. Polyethylene glycols (PEGs) are polymers composed of repeating sub-units of identical structure, called monomers, and are the most commercially important polyethers. PEGs are liquid or low-melting solids, depending on their molecular weights. It is prepared by polymerization of ethylene oxide. PEGs are non-toxic and are widely used in the manufacture of cosmetics, water-soluble lubricants, antifreeze agents for automobile radiators, and pharmaceutical, in the conservation treatment of ancient wood and in the production of non-ionic surfactants. Therefore, millions of tons of PEGs are manufactured or involved in industrial processes worldwide every year. Most of them reach conventional wastewater treatment systems after industrial utilization.

A batch-assay test was performed in order to examine the degradability and the methane production potential of PEG-containing wastewater. A laboratory-scale fixed bed reactor made from glass column was also used in this study in order to determine the maximum loading rate during continuous feeding mode. Mesophilic condition (37 °C) and synthetic wastewaters were applied in both experiments. In the batch-assay tests, most of the organic matters (mainly PEG) were degraded and converted to biogas after 70 hours of incubation. A methane production potential of approx.  $0.36 \text{ m}^3 \cdot \text{kg}^{-1} \text{COD}_{\text{added}}$  or  $0.60 \text{ m}^3 \cdot \text{kg}^{-1} \text{PEG}_{\text{added}}$  was observed in this batch-assay test. During the continuous-feeding mode in the fixed-bed reactor, an OLR of  $8 \text{ kg-COD} \cdot \text{m}^{-3} \cdot \text{d}^{-1}$  was considered as the maximum OLR with more than 90% of the COD in the wastewater was eliminated.

Considering the performance of the laboratory-scale reactor (*i.e.* COD elimination and biogas production), it can be concluded that anaerobic treatment is a suitable method for the treatment of PEG-containing wastewater.

**Keywords:** anaerobic digestion, polyethylene glycols, wastewater, energy recovery, biogas production

## 1. Introduction

Polyethylene glycols (PEGs) with typical molecular formula of HO-CH<sub>2</sub>-(CH<sub>2</sub>-O-CH<sub>2</sub>)<sub>n</sub>-CH<sub>2</sub>-OH are considered as an important group of non-ionic synthetic water-soluble polymers of ethylene oxide. PEGs have various molecular weights ranging from 106 to 20,000 g·mol<sup>-1</sup>. These compounds are non-toxic compounds and widely used in industrial processes such as the production of pharmaceuticals, cosmetics, lubricants and antifreeze for automobile radiators, as one component in copper electroplating solution which acts as a brightener and stabilizer to enhance the copper deposition rate or for direct use such as de-icing agents at airports during winter time (Chang *et al.*, 2003, Huang *et al.*, 2005 and Nitschke, 1996). Millions of tons of PEGs are involved in industrial processes worldwide every year which most of them have to be discharged as wastewater after industrial utilization.

Due to its huge production, the fate of PEG-containing wastewater has been a main concern in the last three decades. Basically there are two treatment options to reduce PEG content in wastewater (Chang *et al.*, 2003). The first option is physico-chemical removal of PEGs using destructive processes such as ozonation, ozonation/UV, or H<sub>2</sub>O<sub>2</sub>/UV oxidation, and recuperative processes such as adsorption into activated carbon or montmorillonite clay. The second option is biological processes using aerobic or anaerobic treatment. Several reports on biodegradation of PEGs under aerobic or anaerobic conditions show that the capability of growth of bacterial strains depends on the molecular weight and the structural conformation of PEG molecules. Whereas short-chain PEGs with molecular weights up to 600 g·mol<sup>-1</sup> are aerobically degraded, PEGs with molecular weights higher than 1,000 g·mol<sup>-1</sup> are considered "bioresistant". Complete degradation of PEGs with molecular weights up to 20,000 g·mol<sup>-1</sup> have been reported when these compounds were oxidized by pure and mixed cultures of aerobic bacteria (Schink and Stieb, 1983, Otal and Lebrato, 2003 ).

Anaerobic reactors for wastewater treatment have been used since more than 2000 years ago in the form of Indian and Chinese animal manure digesters (Veenstra, 2000). However, the anaerobic process had been ignored for relatively long time as an option for wastewater treatment since it was considered to be inefficient and too slow to serve the needs of quickly expanding wastewater volumes especially in industrialized and dense populated area (Polprasert *et al.*, 2001). Pushed by the world energy crisis, anaerobic digestion for wastewater treatment has been "rediscovered" in the late of 1960s. Since the work of Young and McCarty (1969, cited in Hickey *et al.*, 1991), the application of anaerobic process for industrial and municipal wastewater treatment has been considered as a practical and an economical alternative to aerobic wastewater treatment. Although several studies reported that anaerobic degradation of PEG-containing wastewater has limited success (Schink and Stieb, 1983), this method has potential advantages such as potential energy recovery in the form of biogas, low biomass production thus less cost for excess sludge treatment and no requirement of oxygen.

This paper presents the results of an initial investigation of suitability of anaerobic treatment to reduce PEG content as the main pollutant constituent and to recover the potential energy from PEG-containing wastewater in the form of biogas. Anaerobic fixed-bed reactor was chosen because of its suitability in treating soluble waste since it can prevent the loss of biomass and prolong the biomass residence time.

## 2. Materials and methods

### 2.1 Wastewater and inoculum

PEG-containing wastewater in this study was artificial wastewater simulating the real wastewater from a solar-cell factory. The factory produces 70 – 200 m<sup>3</sup> PEG-containing wastewater per day with COD content (mainly from PEG) ranging from 2 to 30 g · L<sup>-1</sup>. In this study, the artificial wastewater was made using pure PEG and diluted with tap water in order to have COD content of approximately 15 g · L<sup>-1</sup> (see Table 1). The anaerobic sludge inoculum was obtained from the effluent of a full-scale anaerobic digestion plant treating industrial wastewater. Before using the anaerobic sludge as inoculum for batch assays and continuous fed reactor, the sludge was acclimatized with PEG for about 2 weeks.

**Table 1.** Composition of 10 L artificial PEG-containing wastewater

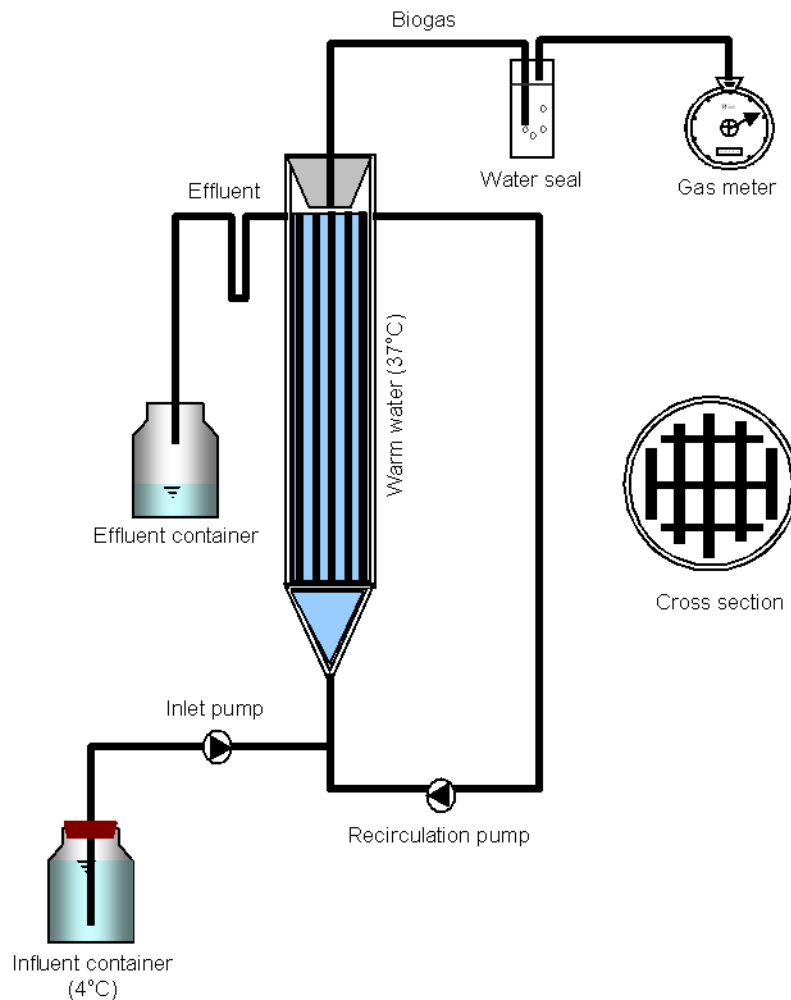
Components	Unit	Amount
Tap water	L	10
PEG	g	90
Urea+phosphate solution	mL	20
Trace metals:		
Iron	mg	10.0
Nickel	mg	1.00
Cobalt	mg	0.10
Molybdenum	mg	0.10
Wolfram	mg	1.00

### 2.2 Experimental set-up

Methane production potential of PEG was investigated in triplicate assays in Schott-bottles of one liter volume. The test was performed by adding 0.372 g of PEG to 200 mL of inoculums. Methane production potential from the inoculum alone was also determined as a control assay. The assays were tightly closed with rubber stopper. After displacing the head space air with N<sub>2</sub>, the assays were incubated at 37 °C on an orbital shaker. Cumulative methane production of the assays was measured 2-3 times a day. To assess the suitability of PEG-containing wastewater as a substrate for anaerobic digestion, a laboratory-scale anaerobic fixed bed reactor consisted of a glass column with a liquid working volume of 7.0 L was operated in continuous feeding mode. Supporting material for the growth of biomass made from liaporclay sinterlamellas was arranged inside the reactor. The temperature of the reactor was maintained at 37 °C by a warm water jacket. To obtain an adequate mixing, liquid and/or biogas from the top of the reactor was withdrawn by a pump and recirculated through the bottom of the reactor together with the feeding (see Figure 1).

In order to examine the performance of the reactor, pH-value, chemical oxygen demand (COD) and residual volatile fatty acids (VFA) in the effluent and biogas production were daily measured. COD was determined according to Wolf and Nordmann (1977) using potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>) in a mixture of sulphuric acid and phosphoric acid (H<sub>2</sub>SO<sub>4</sub> + H<sub>3</sub>PO<sub>4</sub>) as oxidiser and silver sulphate (Ag<sub>2</sub>SO<sub>4</sub>) as a

catalyst. The VFA concentrations were determined using a method described by Gallert and Winter (1997). This method employed a PACKARD model 437 A gas chromatograph, equipped with a flame ionisation detector (FID). Hydrogen and synthetic air were used as burning gases whereas nitrogen was used as the carrier gas. Separation of fatty acids was obtained in a Chromosorb C101 (Sigma, München) Teflon column (2 mm inner diameter x 2 m length). The temperature was set isothermally at 180 °C for the column and 210 °C for injector and detector. One  $\mu\text{L}$  of acidified sample (1:1 volumetric addition with 4 %  $\text{H}_3\text{PO}_4$ ) was injected into the liner in front of the column. The calculation of volatile fatty acids was based on peak area comparison between samples and a mixed volatile fatty acid standard. Biogas composition (methane and carbon dioxide) was analysed with a gas chromatograph CP 9001 (Chrompack, Frankfurt, Germany), equipped with a thermo conductivity detector (nitrogen as carrier and reference gas). Gases were separated isothermally at 110 °C on a Carboplot 007 capillary glass column (27.5m x 0.53 mm, Chrompack, Frankfurt).

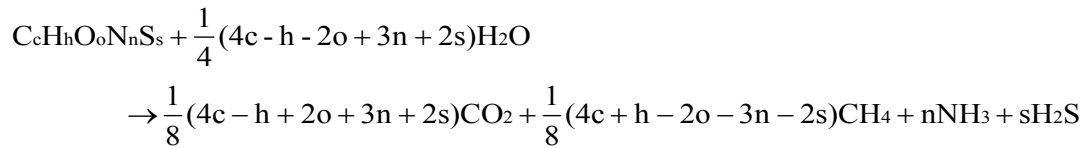


**Figure 1.** Schematic diagram of fixed bed reactor used in this study

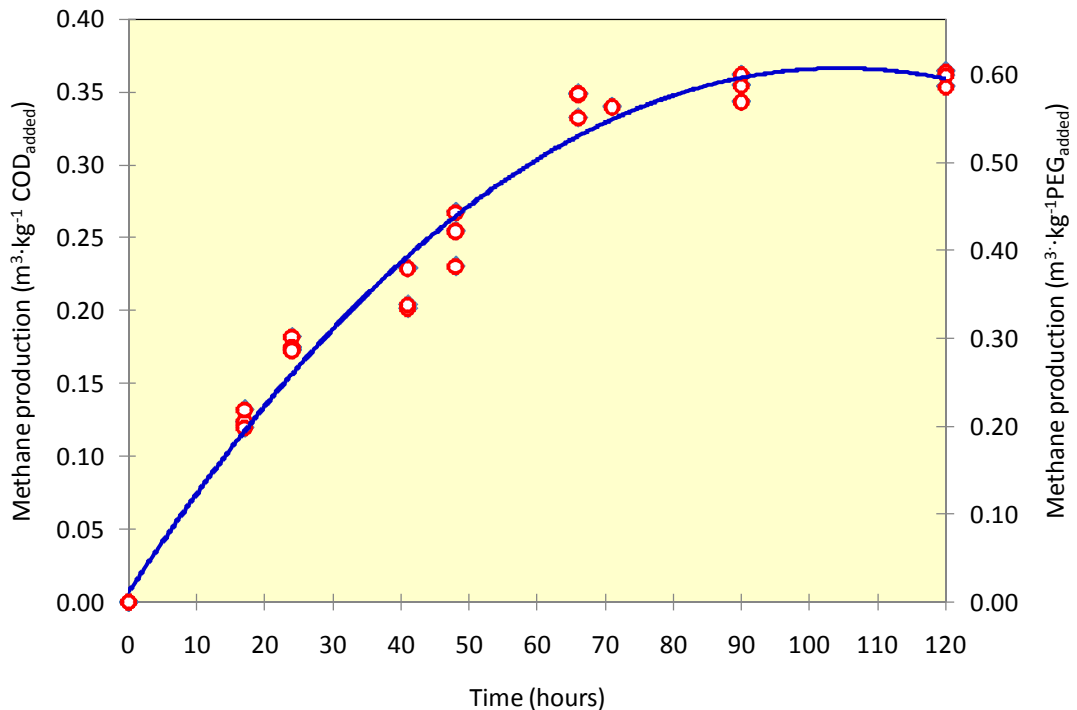
### 3. Results and discussion

#### 3.1 Methane potential production of PEG-containing wastewater

Anaerobic digestion is described as a series of processes involving microorganisms to break down biodegradable material in the absence of oxygen. The overall result of anaerobic digestion is a nearly complete conversion of the biodegradable organic material into methane, carbon dioxide, hydrogen sulfide, ammonia and new bacterial biomass (Veeken *et al.*, 2000; Kelleher *et al.*, 2002). Buswell (1952 as cited in Gallert and Winter, 2005) proposed a generic formula describing the overall chemical reaction of the anaerobic fermentation process of organic compounds which can be used for the prediction of biogas production:



The biogas potential of organic waste including wastewater depends on the content of digestible carbohydrates, lipids and proteins, as well as on the content of more resistant cellulose, hemicellulose and lignin (Hartmann and Ahring, 2006). If a certain type of wastewater has higher readily digestible compounds, the methane production potential is also higher. Glucose for instance, is considered as the most readily degradable carbohydrate. Theoretically, if one mol glucose is completely degraded, the process will yield three moles of methane (1 g glucose yields ca. 0.37 L methane at standard conditions for temperature and pressure, STP)



**Figure 2.** Methane production potential of PEG-containing wastewater. Curves represent methane production from press water only and were obtained by subtracting methane production in assays with and without press water addition.

Figure 2 depicts the biogas production potential with time from the PEG-containing wastewater the batch assays. More than 80 % of the theoretical methane production potential was released during the first 2-3 days. After 4 days of incubation, the methane production ceased and even upon prolonged incubation no biogas was evolved any more. Therefore, it was decided that after 5 days of digestion, the methane production potential of PEG-containing wastewater reached its maximum value. The maximum methane production potential was approximately  $0.36 \text{ m}^3 \cdot \text{kg}^{-1} \text{ COD}_{\text{added}}$  corresponded to  $0.60 \text{ m}^3 \cdot \text{kg}^{-1} \text{ PEG}_{\text{added}}$ . The maximum methane production rate was obtained within the first two days with a value of  $0.13 \text{ m}^3 \cdot \text{CH}_4 \cdot \text{kg}^{-1} \cdot \text{COD} \cdot \text{d}^{-1}$ . The value of methane potential production of PEG is close to that of glucose, indicating that PEG-containing wastewater is a readily biodegradable substrate.

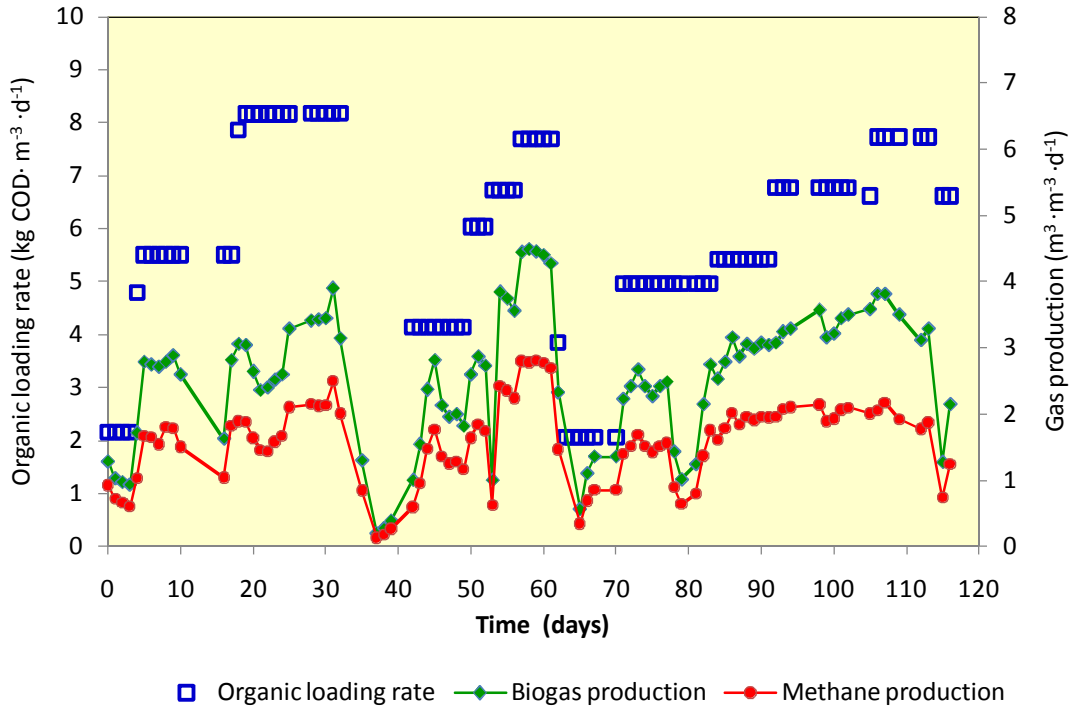
### 3.2 Performance of anaerobic fixed-bed reactor

The laboratory fixed-bed reactor was operated for about four months. The reactor was filled with ca. 5.5 L of inoculums then immediately fed with artificial PEG wastewater. Initially the reactor was fed with an organic loading rate (OLR) of  $2.2 \text{ kg COD} \cdot \text{m}^{-3} \cdot \text{d}^{-1}$ . The OLR was step-wisely increased in order to investigate the maximum OLR of the reactor. The increase of the OLR was done by increasing PEG-containing wastewater feeding (shortened the hydraulic retention time, HRT) or increases the COD concentration of the artificial wastewater (the HRT remained the same with higher OLR). Each OLR increment was performed when the reactor was considered at steady-state conditions. The steady-state condition was judged by the COD elimination efficiency, methane content of the biogas, pH of the effluent and concentration of residual VFA in the effluent.

When the OLR was operated at an OLR of  $8.2 \text{ kg COD} \cdot \text{m}^{-3} \cdot \text{d}^{-1}$  for about two weeks, the reactor showed a symptom of overload. Although the COD elimination efficiency and the biogas production were not really influenced, pH value of the effluent dropped to below 6.8 (data not showed). The pH value of the digester content is an important indicator of the performance and the stability of an anaerobic digester. Although acceptable enzymatic activity of acid-forming bacteria can occur at pH 5.0, methanogenesis proceeds only at a high rate when the pH is maintained in the neutral range. Most anaerobic bacteria including methane-forming bacteria function in a pH range of 6.5 to 7.5, but optimally at a pH of 6.8 to 7.6, and the rate of methane production may decrease if the pH is lower than 6.3 or higher than 7.8 (Stronach *et al.*, 1986; Lay *et al.*, 1998). Therefore, the feeding was stopped for about one weeks in order to give adaptation time for the reactor to obtain its previous capacity. The similar symptom of failure was also observed when the reactor was operated for two months at an OLR of  $8.0 \text{ kg COD} \cdot \text{m}^{-3} \cdot \text{d}^{-1}$ . In this case, the feeding was not stopped but reduced to an OLR of  $2.1 \text{ kg COD} \cdot \text{m}^{-3} \cdot \text{d}^{-1}$  and the reactor was working again.

Biogas and methane production and their relationship with OLR during the reactor experiment are shown in Figure 3. From the figure, it can be seen that the volumetric biogas and methane production of the reactor increased linearly with the increment of the OLR except for the first week when the reactor was fed with an OLR of  $8.2 \text{ kg COD} \cdot \text{m}^{-3} \cdot \text{d}^{-1}$ . When the reactor was continuously fed with an OLR up to  $5.5 \text{ kg COD} \cdot \text{m}^{-3} \cdot \text{d}^{-1}$ , the methane yield reached an average value of  $0.34 \text{ m}^3 \cdot \text{kg}^{-1} \text{ COD}_{\text{added}}$  (94% of the maximum potential methane production of PEG,  $0.37 \text{ m}^3 \cdot \text{CH}_4 \cdot \text{kg}^{-1} \text{ COD}_{\text{added}}$ ). This indicated that the initially inoculated population contained all those

organisms in sufficient amounts that were required for efficient PEG-containing wastewater biodegradation. However, when the OLR was increased to  $8.2 \text{ kg COD} \cdot \text{m}^{-3} \cdot \text{d}^{-1}$ , the methane yield dropped to only  $0.21 \text{ m}^3 \cdot \text{kg}^{-1} \text{ COD}_{\text{added}}$  (57% of the maximum methane production potential). This decrease together with the drop of pH value indicated that there was a disturbance in methanogenesis process, most probably due to the low pH value within the reactor.

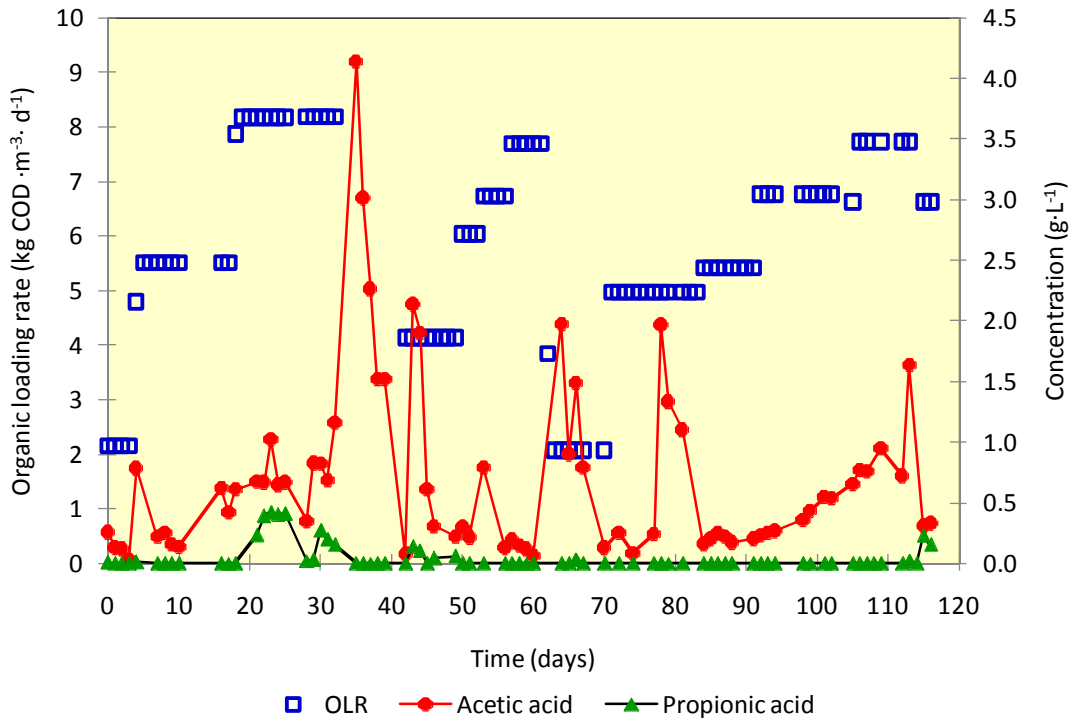


**Figure 3** OLR and daily volumetric biogas and methane production

In a well-balanced anaerobic digestion process, almost all products of a metabolic stage are continuously converted into the next breaking down product without any significant accumulation of intermediary products such as different fatty acids. If the production rate of VFA by polymer hydrolysis and acidogenesis exceeds their consumption rate, an increased level of VFA (causing a low pH) may inhibit the whole process. Therefore, the concentration of residual VFA in the effluent is an important parameter to examine the performance of an anaerobic reactor. Figure 4 depicts the concentrations of VFA in the effluent of the reactor against its OLR. Although the analysis of residual VFA was done for four different type of VFA (acetic, propionic, butyric and valeric acids), only acetic and propionic acids were detected in significant amounts.

The initially present acetic acid was rapidly degraded, whereas the propionic acid concentration was not detectable. Acetic acid increased drastically and reached a maximum concentration of  $4.2 \text{ g} \cdot \text{L}^{-1}$  when the OLR was increased to  $8.2 \text{ kg COD} \cdot \text{m}^{-3} \cdot \text{d}^{-1}$ . In the same time, the concentration of propionic acid increased to about  $0.5 \text{ g} \cdot \text{L}^{-1}$ . As has been reported by several authors (*e.g.* Inanc *et al.* 1999; Gallert *et al.*, 2003 and Nayono *et al.*, 2009a), the accumulation of fatty acids is normally occurred during start-up periods or process instability following shock loading. Among VFAs, propionic acid

plays an important role during both start-up period and process instabilities following shock loading or reparation. As an intermediate product during the breakdown of organic matter to fatty acids, propionic acid is believed to be the most toxic VFA. McCarty and Brosseau (1963 in Vavilin *et al.*, 2003) reported that methanogenic bacteria population were inhibited at propionic acid concentration of 1,000 mg·L<sup>-1</sup> while the population could tolerate acetic and butyric acids up to 10,000 mg·L<sup>-1</sup>.



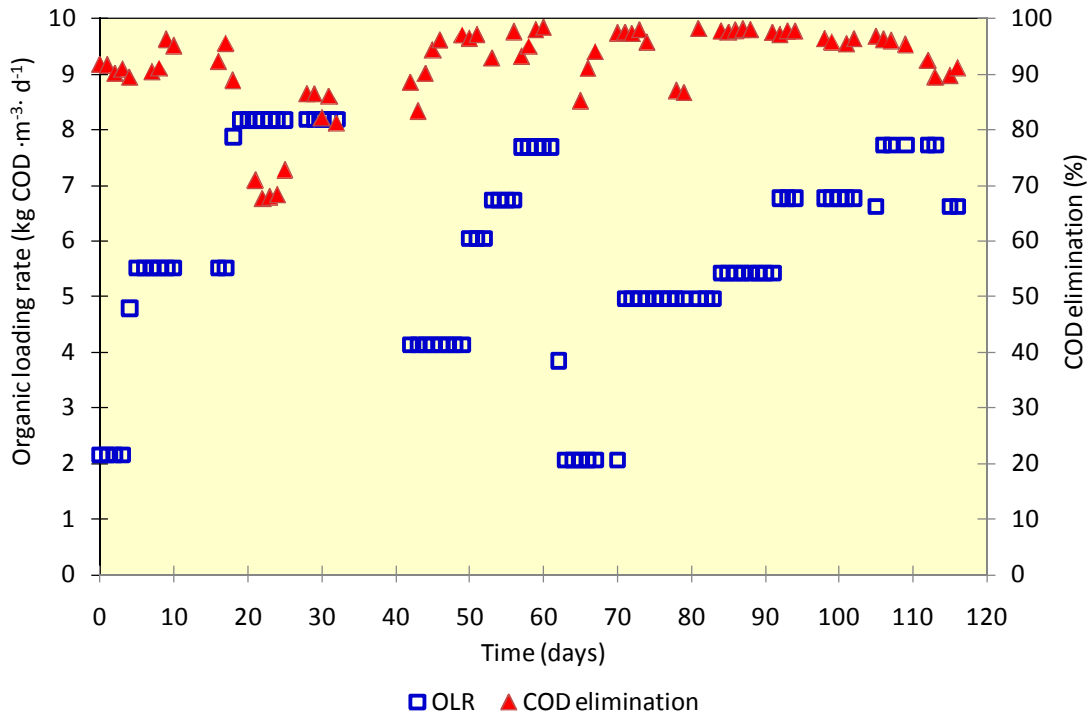
**Figure 4** OLR and residual volatile fatty acids in the effluent

In this study, however, the increase of both acetic and propionic acid caused a decrease of pH value to lower than 6.8. This problem led to a decrease of biogas/methane production and lower COD removal efficiency (see also Figure 5). As have been previously mentioned, the feeding was stopped for about one week to repair the deteriorated performance of the reactor. Within a few days both VFA concentrations decreased to a non-measurable concentration, indicating that the propionate-degraders within the group of acetogenic bacteria and acetate-degraders methanogenic bacteria had adapted their activity to the new situation. However, when the feeding was introduced again (with lower OLR), acetic acid concentration increased to 2.0 g·L<sup>-1</sup> but then decreased to lower than 0.5 g·L<sup>-1</sup>. Butyric and valeric acid was not measurable at any time. It is either not produced as an intermediate product or its acetogenic conversion to acetate and hydrogen proceeds much faster at any time than its generation (Gallert and Winter, 2005; Nayono *et al.*, 2009b; Palmisano and Barlaz, 1996).

The efficiency of the reactor to reduce organic compounds was measured daily by determining the elimination of total COD. Many governments impose strict regulations regarding the maximum chemical oxygen demand allowed in wastewater before they can be returned to the environment. In European countries for example, a maximum oxygen demand between 200 and 1000 mg/L must be reached before wastewater or



industrial water can be returned to the environment. Therefore, a good performance of a reactor to reduce COD as representation of organic compounds is expected. Figure 5 presents the daily COD elimination efficiency at different OLR levels. From the figure, it can be concluded that during the steady-state condition, anaerobic fixed-bed reactor reached a relatively high COD elimination efficiency. When the reactor was operated at OLR ranging from 2.1 to 6.7 kg COD · m<sup>-3</sup> · d<sup>-1</sup> the elimination efficiency reached up to 95%. However, when the OLR was increased to more than 8.0 kg COD · m<sup>-3</sup> · d<sup>-1</sup>, the efficiency decreased to below 70%.



**Figure 5** OLR and COD elimination efficiency

Several studies of anaerobic treatment of wastewater using anaerobic fixed-bed reactor also reported satisfactory results in term of COD elimination efficiency. Anaerobic digestion of silage effluent using anaerobic fixed-bed reactor, for instance, achieved a good result with a average percentage decrease in pollution potential obtained was 75.7% with respect to COD (Barry and Colleran, 1982). Alves *et al.* (2001) reported that more than 90% of COD was removed from a synthetic wastewater containing 12 g COD · L<sup>-1</sup> of sodium oleate as a sole carbon source. Another study reported that a combined up-flow anaerobic fixed-bed, using pumice as a biofilter material and a suspended aerobic activated sludge bioreactor equipped with a microfiltration unit exhibited high performance on the COD removal efficiencies ranging from 94 to 98.7% (Kocadagistan *et al.*, 2005). Handajani (2004) concluded that anaerobic fixed bed reactor is superior compared to completely stirred tank reactor (CSTR) and upflow anaerobic sludge blanket (UASB) in treating wastewater with high soluble organic matter and less settleable solids. The author also reported that about 85% COD of whey-containing wastewater from dairy industry can be removed using upflow anaerobic fixed bed reactor.

#### 4. Conclusions

From the results of the experiment, it can be concluded that anaerobic treatment using fixed-bed reactor is a suitable method to reduce pollutant content of PEG-containing wastewater. The COD elimination efficiency reached up to 95% when the reactor was operated at an OLR up to  $6.7 \text{ kg COD} \cdot \text{m}^{-3} \cdot \text{d}^{-1}$ . Moreover, the application of anaerobic treatment offers an advantage of energy recovery in the form of biogas. The methane yield of the continuously-fed reactor reached an average value of  $0.34 \text{ m}^3 \cdot \text{kg}^{-1} \text{ COD}_{\text{added}}$  or corresponded to 94% of the maximum potential methane production of PEG.

However, three attempts to increase the OLR over  $\text{kg COD} \cdot \text{m}^{-3} \cdot \text{d}^{-1}$  resulted a poor performance of the reactor and even indicated a failure. The symptom of failure was indicated by the decrease of biogas production, high concentration of residual VFA in the effluent, low COD elimination efficiency and low pH value. This poor performance is probably resulted from “over capacity” of the fixed-bed media especially acetate-degrader methanogens (indicated by high residual acetic acid concentration in the effluent). One suggestion to solve the problem will be addition of fixed-bed media to increase the surface area in order to increase the “capacity” of the reactor. Miyahara *et al.* (1995) confirmed that the number of methanogenic bacteria closely packed anaerobic fixed bed reactor was higher than that of sparsely packed reactor.

Another possibility is to provide a pH-control mechanism within the reactor system. If the pH value of the reactor decrease to below optimum range, it can be adjusted thus the performance of the reactor can be maintained. Alkalinity and pH in anaerobic digestion can be adjusted using several chemicals such as sodium (bi-) carbonate, potassium (bi-) carbonate, calcium carbonate (lime), calcium hydroxide (quick lime) and sodium nitrate. Addition of any selected chemical for pH adjustment should be done slowly to prevent any adverse impact on the bacteria. Because methanogenic bacteria require bicarbonate alkalinity, chemicals that directly release bicarbonate alkalinity are preferred (*e.g.* sodium bicarbonate and potassium bicarbonate are more preferred due to their desirable solubility, handling, and minimal adverse impacts). Lime may be used to increase digester pH to 6.4, and then either bicarbonate or carbonate salts (sodium or potassium) should be used to increase the pH to the optimum range (Gerardi, 2003).

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