



Long-term stability of thermophilic co-digestion submerged anaerobic membrane reactor encountering high organic loading rate, persistent propionate and detectable hydrogen in biogas

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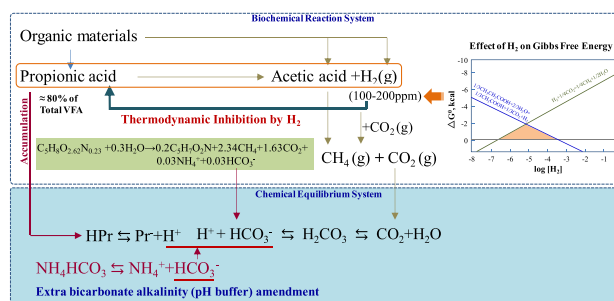
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HIGHLIGHTS

- Long-term stability of thermophilic submerged AnMBR was evaluated.
- Amendment of pH buffer was obligatory for co-digestion system.
- The effect of H₂ partial pressure on propionate accumulation was confirmed.
- Results of 16SrDNA show that propionate-oxidizing bacterium was insufficient.

GRAPHICAL ABSTRACT



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ABSTRACT

The performance of thermophilic anaerobic co-digestion of coffee grounds and sludge using membrane reactor was investigated for 148 days, out of a total research duration of 263 days. The OLR was increased from 2.2 to 33.7 kg-COD/m³ d and HRT was shortened from 70 to 7 days. A significant irreversible drop in pH confirmed the overload of reactor. Under a moderately high OLR of 23.6 kg-COD/m³ d, and with HRT and influent total solids of 10 days and 150 g/L, respectively, the COD removal efficiency was 44.5%. Hydrogen in biogas was around 100–200 ppm, which resulted in the persistent propionate of 1.0–3.2 g/L. The VFA consumed approximately 60% of the total alkalinity. NH₄HCO₃ was supplemented to maintain alkalinity. The stability of system relied on pH management under steady state. The 16SrDNA results showed that hydrogen-utilizing methanogens dominates the archaeal community. The propionate-oxidizing bacteria in bacterial community was insufficient.

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1. Introduction

An anaerobic membrane bioreactor (AnMBR), an innovative combination of membrane technology and a biological process for methane fermentation has great advantages over the conventional process (Liao et al., 2006). It can retain slow growing bacteria

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which would be normally washed out of reactors and support the growth and persistence of these bacteria under unfavorable conditions. A refractory substrate would benefit from the membranes which are designed to develop acclimated populations. An increased organic loading rate (OLR) and biogas production rate from an AnMBR would help to decrease digester volume and save capital investment. As reported by Kanai et al. (2010), an AnMBR volume can be scaled down to approximately 1/5 of the conventional one. Therefore, the application of AnMBR in refractory substrate and under a rigorous condition would be expected.

A huge amount of coffee grounds (annually, six million tons) was generated from the world most popular beverage industry (ICO (International Coffee Organization) www.ico.org/trade_statistics.asp, 2013). A high organic content of 99% (Dinsdale et al., 1997a) and a lipid content of 30% (Dinsdale et al., 1996) in total solids (TS) show that coffee grounds can be suitable for methane fermentation. In a batch experiment, a high biogas conversion efficiency of 84% was obtained (Kostenberg and Marchaim, 1993). This result stimulated the bio-energy reclamation from coffee grounds by fermentation. However, many failures results in continuous reactor studies revealed that coffee grounds were not ideal substrates, especially in thermophilic system. In these experiments, stable biogas production was not achieved despite adopting a pH control and micronutrient supplementation in the thermophilic process (Kida et al., 1994; Lane, 1983). Dinsdale et al. (1996, 1997a,b) reported that mesophilic process exhibit higher reactor stability than a thermophilic system. Whereas, Lane (1983) experienced the failure of a mesophilic reactor because of unknown inhibition factors. Many attempts have been made to manage the process stability. Neves et al. (2006) mixed sludge into coffee grounds to evaluate the feasibility of a thermophilic co-digestion system. However, subsequent long-term system experiments were not reported. The positive effects of mixing sludge with coffee grounds for thermophilic fermentation remain unclear. Therefore, although the research on thermophilic anaerobic fermentation of coffee grounds has been proposed since 1980s, a credible process has not been established. Only a few studies on thermophilic coffee waste fermentation have been reported since the above articles were published in the 1990s.

Methane fermentation systems aim to increase net energy production efficiency. Volumetric methane production increases with the higher OLR. The establishment of a long-term and stable system is the foundation for investigating the cost-effectiveness of bio-energy production from coffee grounds. The highest OLR obtained from 61 days of continuous experimentation was 8.6 g-VS/Ld (Kostenberg and Marchaim, 1993). However, Lane (1983) showed that the system failed at 80 days. Therefore, a stable OLR that can be used as a design criterion was not realized. Despite the result achieved from a short-term operation, this OLR is not sufficient compared with that from food waste (OLR, 21.8 g-VS/L) reported by Dai et al. (2013). This leads to the problem of considering methane fermentation as a method for processing the coffee grounds.

In the present study, a long-term thermophilic coffee ground co-digestion reactor was operated for more than 263 days. Based on the long-term experiment, the objectives of the present study include the investigation of the thermophilic co-digestion performance under high OLR, process amendment strategies, and micro-community structure corresponding to process performance.

2. Methods

2.1. Properties of coffee grounds and sludge

The coffee grounds used in this study were sampled from an instant coffee manufactory in Japan. The raw coffee grounds were solid particles with a TS content of $34.7 \pm 0.29\%$. The dewatered activated sludge was obtained from the coffee waste water treatment unit. The TS content of the sludge was around $15.8 \pm 0.45\%$. The coffee grounds and sludge were kept in a 4 °C refrigerator before mixing. The characteristics of the coffee grounds and the sludge are given in Table 1. The coffee grounds, sludge, and tap water were mixed and shredded in a high speed blender (LBC-15) at 18,500 rpm for 20 min to prepare the substrate mixture. The ratio of dry coffee to dry sludge was 85:15 due to the unstable

Table 1
Characteristics of coffee ground and sludge.

	Coffee ground	Sludge
TS (%), n = 10	34.7 ± 0.29	15.8 ± 0.45
VS (%), n = 10	34.4 ± 0.46	12.4 ± 0.57
VS/TS (%)	99	79.45
COD/TS (g/g)	1.60	0.98
Carbohydrate (g/g-TS)	0.59	0.31
Protein (g/g-TS)	0.24	0.69
Lipid (g/g-TS)	0.24	0.02
Tannins, (g/g-TS)	4.35	/
C (%), n = 3	55.23 ± 3.71	34.04 ± 0.22
H (%), n = 3	7.07 ± 0.39	5.47 ± 0.07
O (%), n = 3	34.44 ± 4.54	25.69 ± 0.42
N (%), n = 3	2.33 ± 0.24	5.93 ± 0.09
S (%), n = 3	0.30 ± 0.19	0.70 ± 0.02
C:N	23.7	5.8

reactor by feeding mixture containing sludge less than 15% in our previous experiment. The TS of the coffee and sludge was measured before mixing to accurately determine the composition of sludge and TS in the feedstock.

2.2. Submerged anaerobic membrane reactor

This research adopted a submerged anaerobic membrane reactor (AnMBR). The HRT and SRT were separated between day 115 and 184. From day 185 to 263, the effluent was directly discharged. Membrane performance was not provided in this paper. The reactor had a working volume of 7 L and a total volume of 15 L. The digester was kept in thermophilic condition (55–57 °C) by circulating hot water (62 °C) through the water-jacket. A wet gas meter (Shinagawa, 0.1 L/rev) was connected to the digester to record daily biogas volume. A 200 mL water seal was connected between the gas meter and the reactor to avoid allowing air back into the reactor. Seed sludge was taken from a food waste thermophilic fermentation digester with a TS of 3.1%. The digester was fed 4 times per day by an automatically controlled peristaltic pump. Feedstock was stored at 4 °C in a substrate tank connected to a cooling water bath. The system setup is represented in Fig. 1.

A flat sheet microfiltration membrane module was immersed in the lower half of the digester. The membrane was made of chlorinated polyethylene with a normal pore size of 0.2 μm and a total area of 0.116 m² (Kubota Membrane Cartridge, Japan). A coarse tube diffuser was located below the flat sheet membrane. The biogas in the headspace was recirculated by a gas pump (Iwaki Air Pump APN-085LVX-1) at a flow rate of 5 L/min to provide hydrodynamic shearing. The gas lift operation mode was also used for digester mixing. Permeate was suctioned by a peristaltic pump (Master Flex Cole-Parmer). The flux was controlled by adjusting the pump speed. The membrane in the AnMBR was oversized. The excess permeate was recirculated back into the reactor. The pump was operated in the mode of 4 min on and 1 min off.

2.3. Analysis methods

pH, alkalinity, COD, ammonia, and TS were determined according to the Japanese Standard Testing Method for Wastewater (JSWA (Japanese Standard Methods of the Examination of Wastewater), 1997). Bicarbonate alkalinity was measured by titration at pH 5.75 using 0.1 mol/L HCl (Lahav and Morgan, 2004). Volatile fatty acids (VFA) concentration was determined by an Agilent-6890 gas chromatograph equipped with a DB-WAXetr capillary column and an FID detector. The biogas composition, CH₄ and CO₂, was measured by a Shimadzu GC-8A gas chromatograph equipped with a thermal conductivity detector. The elemental

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