# Enhancement of hydrolysis and acidification of solid organic waste by a rotational drum fermentation system with methanogenic leachate recirculation

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## Abstract

A cascade process of a rotational drum fermentation system with leachate recirculation from a methanogenesis to the acidogenic reactor was constructed to enhance the hydrolysis and acidification of solid organic waste. Using fresh soybean meal as substrates, two process configurations-Cascade process 1 and 2, without and with leachate recirculation, respectively-were employed to perform the experimental estimation under mesophilic condition and a total HRT of 20 days. An apparent first-order hydrolysis rate constant of  $9.0 \times$  $10^{-3}$ /d for Cascade process 1 at pH values of 4.5-4.6 and  $15.8 \times 10^{-3}$ /d for Cascade process 2 at pH of 4.6-5.2, were obtained. The apparent VS degradation ratios ranged from 16.5% to 21.1% and total VA (as acetic acid) from 14.5 g/L to 16.7 g/L. Occupying ratios for ionized VA decreased from 40.5% to 35.3% for Cascade process 1 and increased to 68.5% for Cascade process 1 and to 72.6% for Cascade process 2 whereas propionic acid and butyric acid ratios increased in acidogenesis of Cascade process 2. The leachate recirculation promoted hydrolysis of substrate in Cascade process 2, where apparent hydrolysis rate constant and VS degradation ratio were higher than that of Cascade process 1. *Keywords:* leachate from methanogenic process, cascade process, hydrolysis rate constant, volatile solid degradation ratio, unionized VA, ionized VA, VA spectrum

## 1. Introduction

With some superiorities over the conventional one-phase process (Cohen et al., 1979), the two-phase anaerobic digestion process has been widely used in treatment of industrial wastewater (Bull et al., 1984), dairy-industry wastes (Ghosh et al., 1994), municipal solid wastes (Sans et al., 1995; Chugh et al., 1999) and solid food wastes (Argelier et al., 1998; Traverso et al., 2000). It was carried out in two physically separated reactors with responsibility for hydrolysis/acidogenesis and methanogenesis respectively. With the aim to accelerate degradation of complex organic compounds and obtain the maximum concentrations of intermediary products, hydrolysis/acidogenesis is recognized as a rate-limited step where the substrate consists of particles (Eastman et al., 1981). Its performance was significantly affected by environmental and operational parameters such as pH, temperature, substrate, process configuration and concentrations of intermediaries (Veeken et al., 1999).

In the two-phase anaerobic digestion of solid waste, metabolic intermediary products such as volatile acids (VA) in the acidogenic reactor were prone to accumulate due to the absence of methanogenic microorganisms which consume the intermediaries directly (Eastman et al., 1981). According to the surface based kinetics model (Sanders et al., 2000), where it was assumed that the substrate was spherical particles and was degraded from outside, the metabolic intermediaries could easily attach onto the surface of the particles and prevent the microorganisms entrance for further reaction. In addition, Veeken and his workers (2000) observed that the accumulation of metabolic intermediaries such as VA restricted both methanogenesis and acidogenesis. Especially, unionized volatile acid (UVA) would inhibit not only the former (Kroeker et al., 1979), the latter (Garcia et al., 1991) but also the hydrolysis rate of particulate matter (Llabres-Luengo et al., 1988). Simultaneously, physical separation of acidogenic and methanogenic phases suppressed the syntrophic acidogenic reactions due to excessive hydrogen-forming where there was a deficiency of methanogenic microorganisms (Fox et al., 1994).

Some investigations, such as in situ removal of VA and alteration of process configurations, were conducted to alleviate the inhibition of high-concentration VA. Generally, the methods for in situ removal, which removed the excess VA physically by extraction, electrodialysis and adsorption (Sun et al., 1999; Aljundi et al., 2005; Hirata et al., 2005), were used to recover VA as products from the fermentation broth. And the attempts to alter process configurations, such as leachate recirculation, solid recycle process and cascade process, were made to ease the inhibition by providing a predominant environment for microorganisms.

Leachate recirculation, which was operated by recycling the effluent from a methanogenic process to acidogenic or hydrolysis process, can not only alleviate the inhibition of excess VA, but also establish the balance between each step in the two-phase degradation process. Veeken et al. (2000) designed a batch-wise solid state digestion (BSSD)

setup with leachate recirculation to evaluate the effect of leachate recirculation on solid state digestion of biowaste and concluded that the apparent first order hydrolysis rate constant increased from 1.0 to  $17.0 \times 10^{-3}$  /d as the leachate recirculation rates increased from 1 to 100 m<sup>3</sup>/m<sup>3</sup>/d. Moreover, Jiang et al. (2005) developed a solid recycle (SR) process with leachate recirculation from a methanogenic reactor to acidogenesis using a rotational drum fermentation system (RDFS) to evaluate the effects of leachate on acidogenic performance. Their results showed that the leachate recirculation elevated the pH levels and improved acidogenic performance of the solid recycle process such as increasing VA concentration and VS degradation ratio.

Another configuration, the which process cascade process, separated hydrolysis-acidogenesis into detached reactors may be appropriate to suppress inhibitions caused by high-concentration products as well. The separation of the hydrolysis and the acidogenic phase could establish the concentration gradients of pH and reactants for each step, hence upgrading the hydrolysis of solid particles (Jiang et al., 2003). Argelier et al. (1998) established a cascade process for food solid waste to lessen the inhibition of VA on fermentative activities. They succeeded in obtaining effluent with 42 g/L of VA at steady-state conditions at a loading of 12.5 kg-COD/m<sup>3</sup>/d and a hydraulic retention time of 12.5 days. Utilizing the rotational drum fermentation system, Jiang et al. (2003) introduced a cascade process and a solid recycle process to achieve a higher TVA concentration and hydrolysis rate.

The results indicated that the separation of hydrolysis and acidogenesis strongly affected acidogenic performance in terms of the VA constituents and the distribution of ionized and unionized VA.

The purpose of this work was to enhance the hydrolysis and acidification of solid organic waste in a cascade process with leachate recirculation by a rotational drum fermentation system. The objectives were to: (1) construct a cascade process using a rotational drum fermentation system that recycles the leachate from a methanogenic to acidogenic process, and (2) evaluate the effects of leachate on acidogenic performance via parameters such as pH, VA production, hydrolysis rate constant and VS degradation ratio, in comparison with a single cascade process.

#### 2. Materials and methods

## 2.1. Substrate and feeding sludge

Fresh soybean meal or Okara (approximately 20% total solids), a food process by-product from soybean curd (Hisaki Tofu, Shimane, Japan), was used as substrates. The chemical characteristics of the soybean meal were identical to the ones shown previously by Kitamura et al. (1998).

Mesophilic anaerobic sludge from a municipal sewage treatment center (Shimane, Japan) was inoculated in the fermentors as the seeding sludge.

#### 2.2. Process configuration

The cascade process configuration is shown in figure 1.  $\alpha$  represents the recirculation ratio of leachate to feeding rate for fermentors II and IV, respectively. In the case of  $\alpha$ =0, the process is named as Cascade process 1 whereas the process is termed Cascade process 2 as  $\alpha$ =1.

Each process consists of two fermentors for pseudohydrolysis and pseudoacidogenesis, respectively. Either fermentor I or II is a component of Cascade process 1. Feedstock was fed to fermentor I, and then the effluent from fermentor I was fed to fermentor II. Finally, sludge from fermentor II was excreted as an effluent of Cascade process 1. Cascade process 2 comprises fermentor III and IV. The same feedstock was fed to fermentor III, then its effluent was diluted by leachate from a methanogenic process as feedstock without loss of hydrolytic and acidogenic microorganisms fed to fermentor IV.

# Figure 1.

The methanogenic fermentor (Jiang et al., 2005) was fed daily with synthetic wastewater (Chang et al., 1982) and operated at mesophilic condition with an HRT of 10 days. CH<sub>4</sub> content in the biogas and the VA concentration in the effluent were 64-72% and 1900mg/L, respectively, whereas the TS was close to zero. The liquid fraction of the effluent obtained by centrifugation at 3000 rpm for 3 minutes was recycled to acidogenic process as a "leachate"

of methanogenic process.

## 2.3. Experimental apparatus and procedure

A rotational drum fermentation system (Jiang *et al.*, 2002, 2003) was employed. The aluminum oxide milling balls (diameter = 30 mm) were added to pseudohydrolytic fermentors (I and III) to occupy 10% of 3.6 L fermentor volume. However, to avoid the adverse effect of ball rotation on biomass growth, there were no milling balls in the pseudoacidogenic fermentor (II and IV). The RDFS and the methanogenic fermentor were both operated in an incubator at a constant mesophilic temperature (36 to 38 °C).

Prior to starting the experiments, a preheated mixture of the fresh anaerobic sludge and the substrate were inoculated into every fermentor at a weight ratio of 1:1 (anaerobic sludge to feedstock). The substrate was fed daily by the draw- and- fill method at a total HRT of 20 days for each process. Semicontinuous rotation was conducted at 30-min intervals and for 15 min throughout the experimental period, with the drum rotation at around 12 rpm.

The experiment conditions are summarized in Table 1.

## Table 1

## 2.4. Measurement and analysis

Total solids (TS), pH, volatile acids, and volatile solids (VS) were determined according to the sewage test procedure (Japan Sewage Association, 1997). A capillary electrophoresis analytical system (HP, 3DCE) was employed to monitor the VA spectrum during the steady-state period. Biogas composition analysis was performed for  $CH_4$  and  $CO_2$  content using a gas chromatograph (SHIMADZU, GC-14A) equipped with a thermal conductivity detector (TCD).

## 3. Results and discussion

Table 2 shows the values of pH, pH gradient, TVA, TVA gradient, ionized and unionized VA distribution, and VA spectrum for all fermentors. Table 3 shows the VS, VS gradient, VS degradation ratios and hydrolysis rate constants. The results were derived from averaging the consecutive one-month data during the steady-state period.

## 3.1. VA production

Low values of pH (4.6-5.2) in all fermentors indicated that the acidogenesis was prevailing. The results of fermentor I and III are the same. In comparison with the fermentor I, II, III, the pH value in fermentor IV increased due to leachate recirculation.

A high VA concentration (12.6-16.7 g/L) was obtained in all fermentors. The highest VA concentration (16.7 g/L) occurred in fermentor II followed by fermentor IV (14.5 g/L). The lowest VA concentration existed in fermentor I and III (12.6 g/L).

As shown in tables 2 and 3, both cascade processes were favorable to establish the gradients of reactants' (substrates and products) concentration and pH. The values of pH decreased from 6.5 to 4.6 in fermentors I and III, from 4.6 to 4.5 in fermentor II, and from 5.2

to 5.1 in fermentor IV. Simultaneously, with Okara as substrates, VS content decreased from 188 to 168 g/L in fermentors I and III, from 168 to 157 g/L in fermentor II, and from 84 to 74 g/L in fermentor IV. As a result of VS degradation, the VA concentration increased in all fermentors, from 0 in Okara to 12.6 g/L in fermentor I and fermentor III, from 12.6 to 16.7 g/L in fermentor II, and from 7.3 (averaging the VA concentration of fermentor III and the leachate ) to 14.5 g/L in fermentor IV. The greater change of VA concentration in fermentor IV than that in fermentor II was due to the addition of leachate lowered VA concentration and heightened the value of pH of feedstock from fermentor III, thus enhancing acidogenesis. This phenomenon implied the adverse effects of products on the acidogenesis.

From the viewpoint of VA generation, Cascade process 2 with leachate recirculation was more favourable than Cascade process 1 with no addition.

VA generated from fermentor I (12.6 g/L) and fermentor II (4.1 g/L) provided 75% and 25 % VA respectively for Cascade process 1. Fermentor III (6.3 g/L, 43.4% total process VA) contributed less VA to Cascade process 2 than fermentor IV (7.2 g/L, 49.7% total process VA). Compared with fermentor II, higher VA contribution ratio of fermentor IV was also due to the recirculation of leachate from the methanogenic process. It helped elevate the pH level (4.6 to 5.2) approaching the optimum (6.0-6.5) proposed by Kisaalita et al. (1987) and reduce the VA concentration of influent for fermentor IV. These phenomena proved that the leachate promoted the acidogenesis.

#### Table 2

VA can exist in solution in two principal forms: unionized VA (UVA) and ionized VA. The concentrations of UVA were calculated based on the total VA (TVA, g/L) by the following equilibrium expression (Bujoczek *et al.*, 2000):

$$UVA = VA(10^{(pKa-pH)})/(1+10^{(pKa-pH)})$$
(1)

where  $pK_a$  is the dissociation constant value of the acids in water,  $pK_a=4.762$  for acetic acid at 35°C (Weast, 1981).

The distributions of ionized and unionized VA were given in Table 2. The occupying ratios of ionized VA increased as pH increased. The highest ratio of ionized VA to total VA was obtained in fermentor IV (68.5% TVA), followed by fermentors I and III (40.5% TVA), whereas the lowest ratio occurred in fermentor II (35.3% TVA). It can be seen that the results were affected strongly by the leachate recirculation. The occupying ratios of ionized VA in Cascade process 2 were elevated by manipulating the pH levels after leachate recirculation.

The observed VA spectrum is shown in Table 2. Acetic acid was predominant in fermentors I, II and III, followed by succinic acid, while no butyric acid and propionic acid was detected. Due to the leachate recirculation, no succinic acid was detected whereas propionic acid and butyric acid were observed in fermentor IV. Other volatile acids were measured at negligible concentrations.

The effect of leachate in this study is revealed by the shift in the VA spectra, in which the

occupying ratio of acetic acid was decreased from 94.3% to 72.6% in Cascade process 2. Existence of propionic and butyric acids implied that the leachate recirculation accelerated the degradation of long-chain VA such as succinic acid. Higher propionic acid content observed in Cascade process 2 would be fatal for methanogenic process. The decreased occupying ratio of acetic acid in fermentor IV (72.6%) demonstrated that the higher ionized VA (68.5% TVA) was prone to be converted to biogas by methanogenic microorganisms. In addition, the absence of amino acids from hydrolysis of protein in all fermentors can be explained by the presence of carbohydrates, which suppress the protein degradation (Yu et al., 2001).

# 3.2. Hydrolysis rate constant

Based on first-order hydrolysis kinetics, the hydrolysis phase can be expressed using Equation (2) (Borzacconi et al., 1997).

$$\frac{dS}{dt} = -K_h S \tag{2}$$

where  $K_h$  is the particulate matter hydrolysis rate constant (per day), and S is the substrate concentration in solid phase (g-VS/L).

Alternatively, Equation (2) can be transformed into Equation (3) by a definite integral:

$$\ln(\frac{S_0}{S_T}) = K_h T \tag{3}$$

where  $S_0$  is the initial substrate concentration (g-VS/L) at T=0;  $S_T$  is the substrate concentration in fermentor withdrawal at time T (g-VS/L); and T is the reaction time (day). In the steady state operation, the microorganism is in the exponential growth period, therefore, the reaction time is equivalent to the HRT.

## Table 3

Substituting the experimental data into Equation (3),  $K_h$  for each fermentor was calculated (Table 3). The highest  $K_h$  ( $12.4 \times 10^{-3}$  /d) in fermentor IV was because of the leachate recirculation. The second highest value of  $K_h$  ( $11.3 \times 10^{-3}$  /d) occurred in fermentor I and III, whereas the lowest value ( $6.8 \times 10^{-3}$  /d) occurred in fermentor II. The lowest value in fermentor II was because of the adverse effect of high products concentration on hydrolysis. The leachate recirculation in Cascade process 2 elevated the  $K_h$  of fermentor IV ( $12.4 \times 10^{-3}$  /d) in contrast to the one in fermentor II in Cascade process 1 ( $6.8 \times 10^{-3}$  /d).

Apparent hydrolysis rate constants (K<sub>0</sub>, K<sub>0</sub>') of two processes were deduced by Equation (4) for Cascade process 1 and (5) for Cascade process 2.

$$K_0 = \frac{1}{T_0} (K_1 T_1 + K_2 T_2) \tag{4}$$

where  $T_0$  is the apparent HRT for Cascade process 1(day);  $K_1$  and  $K_2$  are the hydrolysis rate constants for fermentor I and II (per day), respectively; and  $T_1$  and  $T_2$  are the HRTs for fermentor I and II (day), respectively.

$$K_{0}' = \frac{1}{T_{0}'} \left( K_{3} T_{3} + K_{4} T_{4} \right)$$
(5)

where  $T_0$ ' is the apparent HRT for Cascade process 2 (day);  $K_3$  and  $K_4$  are the hydrolysis rate constants for fermentor III and IV (per day), respectively;  $T_3$  and  $T_4$  are the HRTs for fermentor III and IV (day), respectively.  $T_0$  and  $T_0$ ' can be deduced by Equation (6) and (7).

$$T_0 = \frac{1}{1+\alpha} T_1 + T_2 \tag{6}$$

$$T_0' = \frac{1}{1+\alpha} T_3 + T_4 \tag{7}$$

Where  $\alpha$  is the recirculation ratio of leachate to feeding rate for fermentor II ( $\alpha$ =0) and IV ( $\alpha$ =1) (Fig. 1).

According to the experimental conditions,  $K_0$  of 9.0  $\times 10^{-3}$  /d and  $K_0$ ' of 15.8  $\times 10^{-3}$  /d for the two processes were obtained by substitution of the experimental data into Equation (4) and (5). The first order hydrolysis rate constant of Cascade process 2 was raised remarkably by the leachate recirculation. This result indicated that the leachate recirculation strongly affected the cascade process.

## 3.3. VS degradation ratio

The VS degradation ratio (R, %) was determined by Equation (8).

$$R = \frac{S_{in} - S_{out}}{S_{in}} \times 100 \tag{8}$$

where R is the VS degradation ratio (%);  $S_{in}$  is the initial substrate concentration (g-VS/L); and  $S_{out}$  is the substrate concentration of withdrawal from fermentors (g-VS/L).

Substituting the experimental parameters of each fermentor to Equation (8), R of each fermentor can be calculated and was shown in Table 3. The highest VS degradation ratio (11.7%) was observed in fermentor IV. The second highest VS degradation ratio (10.6%) was found in fermentors I and III. The highest R of fermentor IV was owing to the leachate recirculation. R of fermentor II was lower than that in fermentor I due to the lower pH

gradient and the inhibition resulting from excessive VA concentration.

(9) for Cascade process 1 and Equation (10) for Cascade process 2.

$$R_0 = R_1 + R_2 - R_1 R_2 \tag{9}$$

where  $R_0$  is the apparent VS degradation ratio for Cascade process 1 (%);  $R_1$ ,  $R_2$  are the VS degradation ratios for fermentors I and II (%), respectively.

$$R_0' = R_3 + R_4 - R_3 R_4 \tag{10}$$

where  $R_0$ 'is the apparent VS degradation ratio for Cascade process 2 (%);  $R_3$ ,  $R_4$  are the VS degradation ratios for fermentors III and IV (%), respectively.

Substituting the values of  $R_1$ ,  $R_2$ ,  $R_3$  and  $R_4$  to Equation (9) and (10),  $R_0$  of 16.5% for Cascade process 1 and  $R_0$ ' of 21.1% for Cascade process 2 were obtained. The VS degradation ratio for Cascade process 2 was higher than that of Cascade process 1.

Based on the results of VA production, hydrolysis rate constants and VS degradation ratios, it is shown that the leachate recirculation promoted the hydrolysis and upgraded acidogenic performance in Cascade process. The VA concentration, the VS degradation ratio and the hydrolysis rate constant of Cascade process 2 were higher than that of Cascade process 1. It is likely that the recirculation of leachate raised the pH levels due to its neutralization, and, in turn, upgraded the distribution of ionized and unionized VA. In

comparison with the solid recycle process with leachate recirculation developed by Jiang et al. (2005), the cascade process was favorable according to hydrolysis rate constants and VS degradation ratios, which were increased from 14.4 to  $15.8 \times 10^{-3}$ /d and from 19.4 to 21.1%, respectively.

# 4. Conclusions

In the acidogenic process of solid organic waste, the volatile acids were prone to accumulate and inhibit both hydrolysis and acidogenesis. The cascade process of a rotational drum fermentation system with methanogenic leachate recirculation alleviated the inhibition by establishing the gradients of pH and product concentration for each step. The leachate recirculation elevated pH levels, and in turn, influenced the distribution of ionized and unionized volatile acid. Simultaneously, VA constituents were affected by alteration of process configuration. The occupying ratio of acetic acid decreased whereas propionic acid and butyric acid increased with leachate recirculation. Our results suggest that the cascade process with methanogenic leachate recirculation assist to improve the acidogenic performance of solid organic waste.

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Fig. 1 Cascade process configurations for acidogenesis. a (g/d): feeding rate of Okara;  $\alpha$  (-): recirculation ratio of leachate to feeding rate for fermentor II and IV, respectively; S (g/L): VS concentration in each stream; r<sub>1</sub> (-): recirculation ratio of leachate to volume of corresponding fermentor content for Cascade process.

Experimental co	onditions						
Acidogenic	Fermentor	HRT	Fooding rate (/d)	Loading rate (/L/d)			
process	No.	(days)	reeding fate (/d)	Loading fate (/L/d)			
Cascade 1	Ι	10	120 g Okara	100 g Okara			
	II	10	120 g sludge from I	100 g sludge from I			
Cascade 2	III	10	120 g Okara	100 g Okara			
	IV	10	120 g sludge from III+120 g leachate	50 g sludge from III+ 50 g leachate			
			from methane fermentor	from methane fermentor			
Methane fermentor		10	300 g inorganic wastewater +3 g acetic acid	100 g inorganic wastewater + 1 g acetic acid			

Table 1	
Eurorimontal	aandi

Table 2
Steady-state parameters for two processes

Acidogenic	Fermentor	pH (-)		pН	TVA	(g/L)	TVA VA Di		stribution		VA Spectrum		
process	No.	in	out	gradient	in	out	gradient	Ionized	Unionized	Acetic	Propionic	Butyric	Succinic
				(-)			(g/L)	VA (%)	VA (%)	acid (%)	acid (%)	acid (%)	acid (%)
Cascade 1	Ι	$6.5 \pm 0.1$	$4.6 \pm 0.1$	1.9	0	$12.6 \pm 0.2$	12.6	$40.5 \pm 1.2$	59.5±1.5	96.1	ND	ND	3.9
	II	$4.6\pm0$	$4.5\pm0$	0.1	$12.6 \pm 0.2$	$16.7 \pm 0$	4.1	$35.3 \pm 0.6$	$64.7 \pm 2.4$	94.3	ND	ND	5.7
Cascade 2	III	$6.5 \pm 0.1$	$4.6 \pm 0.1$	1.9	0	$12.6 \pm 0.2$	12.6	$40.5 \pm 1.1$	$59.5 \pm 0.8$	96.1	ND	ND	3.9
	IV	$5.2\pm0$	$5.1\pm0$	0.1	$7.3 \pm 0.3$	$14.5 \pm 0.8$	7.2	$68.5 \pm 1.3$	$31.5 \pm 1.4$	72.6	8.2	19.2	ND

Note: ND represents undetectable.

Acidogenic Fermentor VS (g/L) VS gradient Hydrolysis VS degradation No. (g/L) rate constant process in out  $(K_h, 10^{-3}/d)$ ratio (R, %)  $188.0 \pm 5.3$  $168.0 \pm 4.8$ Cascade 1 20.0 11.3 10.6 Ι Π  $168.0 \pm 4.8$  $157.0 \pm 3.7$ 11.0 6.8 6.6 Cascade 2  $188.0 \pm 5.3$ III  $168.0 \pm 4.8$ 20.0 11.3 10.6 IV  $84.0 \pm 2.3$  $74.2 \pm 2.1$ 9.8 12.4 11.7

Table 3Steady-state parameters for two processes