



## Short Communication

# Early warning indicators for monitoring the process failure of anaerobic digestion system of food waste



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## HIGHLIGHTS

- Operation conditions significantly affect the responses of state parameters.
- The recommended early warning indicators are different among digesters.
- None of the single indicators was universally valid for all the systems.
- A combination of total VFA, VFA/TA and BA/TA has a general adaptability.

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## ABSTRACT

To determine reliable state parameters which could be used as early warning indicators of process failure due to the acidification of anaerobic digestion of food waste, three mesophilic anaerobic digesters of food waste with different operation conditions were investigated. Such parameters as gas production, methane content, pH, concentrations of volatile fatty acid (VFA), alkalinity and their combined indicators were evaluated. Results revealed that operation conditions significantly affect the responses of parameters and thus the optimal early warning indicators of each reactor differ from each other. None of the single indicators was universally valid for all the systems. The universally valid indicators should combine several parameters to supply complementary information. A combination of total VFA, the ratio of VFA to total alkalinity (VFA/TA) and the ratio of bicarbonate alkalinity to total alkalinity (BA/TA) can reflect the metabolism of the digesting system and realize rapid and effective early warning.

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## 1. Introduction

Anaerobic digestion (AD) usually features process imbalance under high organic loading rate (OLR) (Méndez-Acosta et al., 2010), so the process monitoring and control are indispensable for achieving a high-efficiency and stabilized performance of the AD system (Bjornsson et al., 2001), while effective process parameters are the basis for ensuring the process monitoring and control. A multitude of studies have explored the process parameters of the AD system. To sum up, the most commonly used parameters include pH, volatile fatty acid (VFA), alkalinity, biogas production, methane content and their combination factors (Bjornsson et al., 2001).

However, previous researches on early warning parameters always focused on a specific parameter, verified its effectiveness

and defined its threshold value (Martín-González et al., 2013; Wang et al., 2009); or studied the responses of multiple parameters under a specific condition, and suggested the optimal parameters for this operating conditions (Nielsen et al., 2007; Kleyböcker et al., 2012; Ahring et al., 1995). Thus, though lots of researches on early warning parameters have been conducted, the proposed early warning indicators are only effective when applied to these specific operating conditions, while less effective in systems with different operation conditions. For example, the ratio of intermediate alkalinity to partial alkalinity (IA/PA) of 0.9 was suggested in order to maintain stable operation in thermophilic reactors treating sewage sludge; however IA/PA of 0.4 was proposed to assure a stable reactor performance when a potato-starch wastewater was treated; and IA/PA below 0.3 was recommended to maintain a stable operation when municipal solid waste was treated (Martín-González et al., 2013). In contrast, Hill et al. (1987) suggested that the ratio of propionate to acetate ( $Pr^-/Ac^-$ ) higher than 1.4 indicated impending digester failure, but Weiland (2008) held that the  $Pr^-/Ac^-$  shall be lower than 1.0 in a stable system; and

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Ahring et al. (1995) suggested that  $\text{Pr}^-/\text{Ac}^-$  was useless in determining process imbalance. Other parameters are also suffering the similar phenomenon. Therefore, the indicators suggested by each study are different, as for the same warning parameter there even existed multi-thresholds. So far there still lacks of an early warning index that is universally applicable.

This study is aimed at comprehensive evaluation of the validity of parameters under different operation conditions, and find out the early warning index which is universal applicable for indicating process failure of food waste (FW) digesters. Three anaerobic digesters with different operation conditions were investigated; and such process parameters including gas production and composition, pH, VFA, alkalinity and their combined indicators were all evaluated.

## 2. Methods

### 2.1. Feedstock and seeding sludge

FW was collected from a school canteen, and with the removals of such coarse impurities as bones and plastics, the collected FW was divided into to 2 portions, and 1/3 of the FW was placed in boiling water for the removal of the surface floating oil, then it was subject to solid–liquid separation through a sieve with  $2 \times 2$  mm lattice, and the obtained solid part was shredded into particles with an average size of 5.0 mm by a Robot-Coupe Shredder. The remaining 2/3 were milled directly without degreasing. Then, the two kinds of materials were all packed into 4-L plastic storage bags, and cryopreserved at  $-18$  °C. One week prior to use, the frozen feedstock was thawed, and stored at 4 °C.

The seed sludge used in this study was respectively the digested sludge taken from Dadukou Municipal Wastewater Treatment Plant, Chongqing, China (MWS); and digested sludge from an anaerobic digester treating piggery waste (PWS) and digested sludge from a 50 L anaerobic digester of FW (FWS). The characteristics of the substrate and seed sludge were listed in the [Supplemental materials \(SM\) Table S1](#).

### 2.2. Digesters operation

Three complete-mix anaerobic digesters each with a working volume of 20 L were operated at  $36 \pm 1$  °C. Motorized automatic stirring was provided at the top, with an agitating frequency of  $20 \text{ min h}^{-1}$  and a rotary speed of 40 rpm. Different substrate pre-treatment modes, seeding sludge sources and retention time were respectively adopted for the three digesters, and the specific operating conditions of the various digesters are shown in [Table 1](#). The digesters were operated at a semi-continuous mode; substrate was added once daily and before which manual sampling was conducted at the sampling opening. For an efficient injection, one token of the substrate was mixed with twice volume of retrieved sludge from the operating reactor.

During the experiment, the total solids (TS) and volatile solids (VS) of digestate were measured every three days, and the other

parameters measured daily. VS removal rate ( $\text{VS}_r$ ) was calculated by the same equation as reported by [Koch et al. \(2009\)](#).

### 2.3. Analytical methods

TS and VS were measured according to standard methods ([APHA et al., 1998](#)). pH was measured using a pH meter (Horiba, B-212). Total VFA, total alkalinity (TA) bicarbonate alkalinity (BA), intermediate alkalinity (IA) and partial alkalinity (PA) were analyzed according to [Anderson and Yang \(1992\)](#). Biogas production was measured using wet gas meters. Biogas composition was determined by a Biogas 5000 portable instrument (Geotech, UK). Ammonia-nitrogen was analyzed using a DR-2800 spectrophotometer (HACH, USA). Individual VFA were analyzed on a gas chromatograph equipped with flame ionization detector.

## 3. Results and discussion

### 3.1. Digester performance

OLR, methane yield and  $\text{VS}_r$  are usually used for the evaluation of the anaerobic digester performance ([Nagao et al., 2012](#)). Perturbations of OLR were introduced, and steady state was determined by constant methane yield and  $\text{VS}_r$  in this paper. The date when sharp declines appeared in methane yield or  $\text{VS}_r$  was defined as the date of the process failure. [Fig. 1](#) manifests the responses of methane yield and  $\text{VS}_r$  along with the OLR during the experiment. It can be seen that the fluctuations of  $\text{VS}_r$  were slight throughout the experiment, with the specific values being  $87.5 \pm 3.9\%$ ,  $88.9 \pm 2.8\%$  and  $86.6 \pm 3.4\%$  in Reactor A, B and C, respectively. Thus they are within the recommended range of 81–92% ([Nagao et al., 2012](#)) throughout the experiment, indicating that it is infeasible to use the  $\text{VS}_r$  for performance description.

However, the fluctuations in methane yield were much more obvious. Approximately 50% reductions appeared on day 66 and 72 in Reactor A and C, respectively, and the same reductions occurred on day 34 and 62 in Reactor B. Accordingly, the corresponding dates of process failure were defined, which was indicated by the vertical dotted line in [Fig. 1](#) and [SM Figs. S1–S3](#).

### 3.2. Response of process parameters

As with methane yield, process parameters will respond to process perturbations, and once response from a process parameter is faster than that of methane yield, then the process parameter can be used as early warning indicator. The curve diagrams of the responses of various process parameters along with OLR in three reactors were shown in [SM Figs. S1–S3](#); and [Table 2](#) summarized the specific early warning information of various parameters. Among then the dates of the process imbalance for IA/PA, VFA/TA and BA/TA were, respectively defined as the date when the ratios exceeded their critical value of 0.3, 0.35 and 0.8 ([Martín-González et al., 2013](#); [Méndez-Acosta et al., 2010](#)). The

**Table 1**  
Specific operating conditions of three digesters.

Digester	Inoculum	Substrate	Stage	OLR ( $\text{g VS L}^{-1} \text{d}^{-1}$ )	Retention time <sup>a</sup> (d)
A	MWS	Pre-treated FW	Gradual overload	4, 5, 6, 7, 7.5, 8, 8.5	10
B	PWS	Raw FW	Gradual overload	4, 5, 6, 7, 7.5	7
			Recovery stage	1, 4	10
			Sudden overload	4–7.1 <sup>b</sup>	1
			Recovery stage	1	8
C	FWS	Raw FW	Gradual overload	2, 3, 4, 5, 6	15

<sup>a</sup> Retention time of each OLR.

<sup>b</sup> Increased 10% daily.

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