



Effect of phosphoric acid as a catalyst on the hydrothermal pretreatment and acidogenic fermentation of food waste



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ABSTRACT

The hydrothermal method was applied to food waste (FW) pretreatment with phosphoric acid as a catalyst. The content of soluble substances such as protein and carbohydrate in the FW increased after the hydrothermal pretreatment with phosphoric acid addition ($\leq 5\%$). The SCOD approached approximately 29.0 g/L in 5% phosphoric acid group, which is almost 65% more than the original FW. The hydrothermal condition was 160 °C for 10 min, which means that at least 40% of energy and 60% of reaction time were saved to achieve the expected pretreatment effect. Subsequent fermentation tests showed that the optimal dosage of phosphoric acid was 3% with a VFA yield of 0.763 g/g VS_{removal}, but the increase in salinity caused by phosphoric acid could adversely affect the acidogenesis. With an increase in the quantity of phosphoric acid, among the VFAs, the percentage of propionic acid decreased and that of butyric acid increased. The PCR-DGGE analysis indicated that the microbial diversity could decrease with excessive phosphoric acid, which resulted in a low VFA yield.

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1. Introduction

Food waste (FW) is a suitable organic waste to recycle for high-added-value products by bioengineering. For instance, Kim et al. (2011) added sewage sludge to FW to enhance the hydrogen fermentation performance. Qiang et al. (2012) obtained methane from FW using high-solid mesophilic fermentation. However, the productivity and reuse pathway of biogas were limited. Nonetheless, FW fermentation for volatile fatty acids (VFAs) is now given more attention because of its wider application and higher yield relative to biogas (H. Chen et al., 2013; Y.G. Chen et al., 2013). VFAs are valuable chemical compounds and have diverse uses in the industry market. VFAs are often offered as substrates for biodiesel production (Christophe et al., 2012), electricity generation in microbial fuel cells (Z. Chen et al., 2012), biological nutrition removal (D.B. Wang et al., 2013), and biodegradable plastic production (H. Chen et al., 2013).

Wang et al. (2014) have demonstrated that high VFA yield (0.918 g/g VSS_{removal}) can be obtained by inoculating an anaerobic activated sludge to FW with a 20-day facultative fermentation at pH 6.0. Because the recalcitrant part of FW, such as hemicelluloses

and cellulose, limits the fermentation process, the VFA-producing efficiency should be higher if some pretreatment methods are used. Pretreatments may be commonly grouped into mechanical, thermal, chemical, biological treatments and their combination. Among these methods, thermal-chemical treatments, which include hydrothermal, steam explosion, liquid hot water and CO₂ explosion steam, are most commonly used to break hemicelluloses and cellulose (Di Girolamo et al., 2013). So far, hydrothermal tech has been successfully applied in biomass recycling including agricultural straws and waste plants (Li et al., 2013). And this is a mature, also worth spreading technology. Furthermore, the hydrothermal treatment is preferred because of its safety, high efficiency and low pollution level. Many studies have proved hydrothermal treatment to biomass can improve its biomethane potential (Fernández-Cegri et al., 2012). Thus, Yin et al. (2014) applied a hydrothermal pretreatment to FW to improve the VFA production under an identical fermentation condition. More soluble carbohydrates dissolved with the hemicellulose and cellulose degradation as a result of hydrothermal pretreatment. The optimal hydrothermal condition for fermentation was 160 °C for 30 min. After only 15 days of fermentation, the VFA yield achieved the aforementioned uniform level.

However, the energy cost of the hydrothermal treatment must be decreased. In addition, the secondary reaction must be inhibited at high temperature (Yin et al., 2014), which implies that the

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temperature and treating time should be reduced. Generally, adding some catalysts during the hydrothermal process can simultaneously promote hemicellulose and cellulose degradation with a shorter time and a lower temperature (Kumar et al., 2009). In addition, some reports have described the application of a catalyst to the hydrothermal process to avoid some undesirable reactions (Kruse and Dinjus, 2007a,b). The universal catalysts are acid and alkaline compounds such as vitriol, hydrochloric acid, phosphoric acid, and caustic soda (Takata et al., 2013). For the subsequent fermentation, the catalyst in the hydrothermal process should be nontoxic or harmless to the microbes. Phosphoric acid, which has 3 mole H^+ per mole, can increase the number of ionized products of water more than other acids. It is among the most efficient pretreatment processes, which has been studied to improve the enzymatic hydrolysis of lignocellulosic materials (Zhang et al., 2007). Furthermore, phosphate salts, which are obtained by neutralizing phosphoric acid at the end of the process, can be used as buffers in the fermentation process and nutrients for the microorganisms (Lenihan et al., 2010; Orozco et al., 2011). Thus, it is the most suitable catalyst for the hydrothermal treatment and subsequent VFA production stage.

However, until now, the application of the hydrothermal pretreatment with a catalyst for FW and its effects on the fermentation to produce VFAs has not yet been reported. The goal of this paper is to investigate the effect of phosphoric acid addition on the FW hydrothermal pretreatment and whether it can improve the VFA yield during the fermentation process.

2. Methods

2.1. Food waste and seeding sludge

FW was collected from a campus cafeteria at Zhejiang Gongshang University, Hangzhou, China. Before use, hard objects in the FW were removed. Then, the FW was cut into small particles with a knife. An anaerobic activated sludge was collected from an up-flow anaerobic sludge bed (UASB) of the Xihu Brewery, which was set as the seeding sludge. Prior to inoculation, the seeding sludge was re-activated under its culture conditions. The characteristics of the food waste and seeding sludge used in this experiment are shown in Table 1.

2.2. Hydrothermal pretreatment with phosphoric acid

The hydrothermal pretreatment of the FW was performed in airtight pressure digestion vessels with volumes of 80 mL. Approximately 30 g of chopped FW was placed into each vessel without adding water. The vessels were operated at 160 °C for 10 min in an air-dry oven with 0%, 1%, 3% and 5% phosphoric acid (w/w) (in wet weight of the FW) as the catalyst. The time was measured from when the air-dry oven reached the set temperature. The vessel was cooled to ambient temperature after 10 min of pretreatment.

Table 1
Characteristics of the food waste and inoculums used in the experiment. The data was based on percentage of dry matter except pH and SCOD.

Parameter	Food waste	Seeding sludge
pH	6.1	6.8
TS (%)	23.7	3.72
VS/TS (%)	93.2	87.0
SCOD (g/L)	17.6	—
Total carbohydrate (%)	39.4	1.30
Total nitrogen (%)	2.21	4.30

2.3. Fermentation of the pretreated FW to produce VFAs

The fermentation experiments were performed as described by Yin et al. (2014). Briefly, after they were hydrothermally pretreated with phosphoric acid, the pretreated FWs were fermented in brown wide-mouth bottles with working volumes of 500 mL. FW without hydrothermal pretreatment was used as a control. The total solid (TS) content in each reactor was adjusted to 7%, and the solid was composed of 80% pretreated FW and 20% inoculum (dry weight, with VS/TS = 82%). Then, the temperature of the experiments was maintained at 30 ± 2 °C. The pH value was controlled at 6.0 by adding 4.5 M HCl or NaOH during the whole period of fermentation. Meanwhile, all reactors were mechanically stirred using magnetic stirrers throughout the experiments. Each reactor was duplicated, and the redox potential (ORP) was controlled between −100 and −200 mV by operating under a non-strict anaerobic condition. The fermentation tests ran for 15 days.

2.4. Analytical methods

After the pretreatment, the soluble chemical oxygen demand (SCOD), soluble protein, carbohydrate, lipid, volatile solid (VS) and TS were detected as previously described (Wang et al., 2014). During the fermentation process, bits of fermentation broth were removed and centrifuged at 10,000 rpm for 10 min. Then, the supernatant was filtered through a filtration membrane with a pore size of 0.45 μ m to determine the VFA content and composition, SCOD, soluble protein, carbohydrate and ammonia nitrogen (NH_4^+-N) concentration (Yin et al., 2014). Volatile fatty acids (VFAs, C2–C5) including Ac, Pr, n-Bu, iso-Bu, n-Va and iso-Va were determined by gas chromatography using a GC7890-II (Tianmei Co., Shanghai, China) equipped with a 3 m \times 2 mm stainless steel packed column filled with GDX-103 as the stationary phase and a flame ionization detector (FID). The temperatures of the column, injector and detector were 180 °C, 230 °C and 250 °C, respectively. Likewise, the quantities of carbon in the VFAs and VSS were referenced from Wang et al. (2014). The adenosine triphosphatase (ATPase) activities of the fermentation microbes were assayed using an ATPase assay kit (Jiancheng Bioengineering Institute, Nanjing, China). The final experimental data were expressed as the average of duplicate reactor tests.

2.5. Microbial diversity analysis

A DNA Isolating Kit (MO Bio Laboratories, Inc., Carlsbad, CA, USA) was used to extract the genomic DNA from the collected samples. Then, the V3 region of 16S rRNA of the extracted DNA was amplified using a polymerase chain reaction (PCR), which was previously described (J.H. Wang et al., 2013) using a universal eubacterial primer pair (357F-GC, 5'-GC clamp-CCTACGGGAGG CAGCAG-3' and 518R, 5'-ATTACCGCGGCTGCTGG-3'). The PCR products were separated by denaturing gradient gel electrophoresis (DGGE) using the Dcode™ universal mutation detection system (Bio-rad Laboratories, Hercules, CA, USA). Polyacrylamide gels with 40–60% of vertical denaturing gradient were first electrophoresed with 40 μ L of PCR product at 90 V and 60 °C for 20 min and subsequently 160 V for 6 h. Then, the gel was stained by silver as previously described (Bassam et al., 1991).

The revealed microbial diversity index and similarity in the DGGE gel were analyzed using Quantity One (version 4.6.2; Bio-Rad Laboratories, Hercules, CA, USA) software. Lanes and bands were applied to the image of the gel by the software with additional manual fine-tuning of the band designations. The relative band densities were calculated using the software; the densities were necessary to determine the Shannon Diversity Index (SDI). Raw, manually adjusted peak areas were used because the

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