



# Effect of short-time hydrothermal pretreatment of kitchen waste on biohydrogen production: Fluorescence spectroscopy coupled with parallel factor analysis



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## HIGHLIGHTS

- Short-time hydrothermal pretreatment (SHP) enhanced hydrogen production potential.
- The results showed that DOM blue shifted during the anaerobic fermentation process.
- The protein component degradation were characterised by EEM with parallel factor analysis.
- The optimal 90 °C-SHP was presented in the aspect of the economic analysis.

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## ABSTRACT

The enhancement of bio-hydrogen production from kitchen waste by a short-time hydrothermal pretreatment at different temperatures (i.e., 90 °C, 120 °C, 150 °C and 200 °C) was evaluated. The effects of temperature for the short-time hydrothermal pretreatment on kitchen waste protein conversion and dissolved organic matter characteristics were investigated in this study. A maximum bio-hydrogen yield of 81.27 mL/g VS was acquired at 200 °C by the short-time hydrothermal pretreatment during the anaerobic fermentative hydrogen production. Analysis of the dissolved organic matter composition showed that the protein-like peak dominated and that three fluorescent components were separated using fluorescence excitation–emission spectra coupled with the parallel factor model. The maximum fluorescence intensities of protein-like components decomposed through the parallel factor analysis has a significant correlation with the raw protein concentration, showed by further correlation analysis. This directly impacted the hydrogen production ability.

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## 1. Introduction

Hydrogen has a higher energy yield compared to any known fuel and has been widely recognised as a clean energy carrier of the future (Lee and Chung, 2010; Nathao et al., 2013). Traditional hydrogen production methods such as water electrolysis (Zeng and Zhang, 2010) are cost-intensive because of high-energy requirements and low hydrogen production efficiency. Recently, bio-hydrogen production from organic wastes has drawn much attention; human resources such as fossil fuels are saved, waste is reused, and sustainable hydrogen supplies can be obtained

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(Chong et al., 2009). Kitchen waste is a main component of municipal solid waste which has accounted for 20–54% in most areas of Asia (Yasin et al., 2013). Because of the characteristics of high content of starch, protein, grease and a small quantity of cellulose and hemicellulose, kitchen waste is a good candidate as the substrate to produce biogas by anaerobic fermentation.

The high lipid and salinity content, which is a primary characteristic of kitchen waste in China, has a negative effect on bacteria activity during the anaerobic fermentation bio-hydrogen production process (Lalman and Bagley, 2000; Palatsi et al., 2009). Previous studies have mostly focused on the substrate transfer limitation, such as lipid accumulation. Compared to traditional pretreatment methods (Elbeshbishy et al., 2011), such as physical crushing, acid, alkali and freeze draw, among others, a hydrothermal pretreatment could release the inhibition of the long-chain

fatty acids resulting from the excess lipids and enhance bio-hydrogen production potential (Jia et al., 2013; Lamoolphak et al., 2008; Ma et al., 2011). However, most hydrothermal pretreatment technologies require high temperature and pressure as well as long reaction times, resulting in much greater energy consumption and processing costs. In this paper, methyl silicone oil is involved in the short-term hydrothermal pretreatment (SHP) for effective thermal conductivity and liquid mobility. The experiment is carried on in heated oil bath. The heat left is collected by water heat recovery heat exchanger to save money and time spent on equipment and energy. In the meantime ensuring the effectiveness and improving the treatment of anaerobic fermentation gas energy efficiency. Follow-up studies focuses on continuous heat treatment method as pre-treatment, and kitchen waste heat pre-treatment improvement of heat transfer efficiency and other issues. Few studies have been published on the influence of kitchen waste hydrothermal pretreatment on the organic matter variation using short reaction times and ordinary pressure to enhance the bio-hydrogen production potential. Furthermore, quickly monitoring the organic matter, especially protein transformation, is a problem during the hydrothermal pretreatment and the anaerobic fermentative hydrogen production (AFHP) process.

The purpose of this paper was to investigate the effect of the SHP under different temperatures on the AFHP process. A novel fluorescence technique consisting of three-dimensional excitation–emission matrix (EEM) spectra, a parallel factor (PARAFAC) model and a correlation of key indicators was applied to monitor the efficiency of using the kitchen waste and transfer mechanism. The kinetics of the bio-hydrogen production were analysed to determine important parameters during the process and varied soluble metabolite composition in response to the SHP.

## 2. Methods

### 2.1. Materials

The kitchen waste was collected from a dining hall near the experiment site and smashed into small particles (1–3 mm) after manually separating the nonbiodegradable matters. The chemical and physical characteristics of the kitchen waste were as follows: a total solid (TS) proportion of 21.3%, a volatile solid (VS) proportion of 19.86%, a soluble chemical oxygen demand (SCOD) of 132.8 g/L, a total chemical oxygen demand (TCOD) of 420.0 g/L, a total organic carbon (TOC) of 32.54 g/L, a total nitrogen (TN) of 0.714 g/L and a moisture concentration of 78.7%.

The seed sludge was enriched from an anaerobic reactor used for pig manure anaerobic fermentation. The seed sludge was filtered using a sieve to remove coarse matter and stones and then diluted by an equal volume of distilled water. The characteristics of the seed sludge were as follows: a SCOD of 1.181 g/L, a TS concentration of 16.69%, a VS/TS% of 11.97% and a moisture concentration of 83.31%.

### 2.2. Pretreatments

200 g kitchen waste (moisture concentration of 78.7%) and 100 mL distilled water were added to a 500 mL stirred tank reactor. Then, the mixture was heated to the designated temperature (i.e., 90 °C, 120 °C, 150 °C or 200 °C) and reacted for a short 30 min. Methyl silicone was used in the oil bath as the heat transfer material with kitchen waste under atmospheric pressure. Floatable oil was separated by centrifugation, and then volume was measured.

After the SHP process, the mixture was neutralised to pH 7.0 with either a 1 M HCl or NaOH aqueous solution. Each

experimental condition was performed in triplicate. Control bottles were also prepared using the kitchen waste without prior treatment.

### 2.3. Experimental design

Serum bottles (500 mL) were used as reactors for the AFHP. The pre-treated kitchen waste (15 g) and sludge (25 g) ( $VS_{\text{kitchen waste}}/VS_{\text{sludge}}$  ratio of 1) were added to the bottles, which were placed in a thermostatic water bath oscillator at 150 rpm and  $35 \pm 1$  °C. The total volume was increased to 300 mL using distilled water and the initial pH was adjusted to 6.0. The reactor's headspace was filled with nitrogen gas for 5 min. The biogas samples were taken at intervals of 6 h. The hydrogen volume was recorded through displacement of water in a 1 L serum bottle. The batch experiment ceased when no biogas production was observed for 3 consecutive days. Biogas production was monitored daily and samples from the mixed liquor were taken 1 times a day for chemical index analyses.

### 2.4. Model analysis

The modified Gompertz model (Eq. (1)) was applied to analysis the anaerobic system operating parameters over time during the experiment:

$$H = P \times \exp \left\{ - \exp \left[ \frac{R \times e(\lambda - t)}{P} + 1 \right] \right\} \quad (1)$$

where  $P$  is a maximum hydrogen production potential (mL),  $R$  is a hydrogen production rate (mL/h),  $\lambda$  is the time of lag phase (h), and  $e$  is a constant of 2.71828. Both the Gompertz model operation and the Pearson correlation analysis were performed using Origin 8.0 (Origin Inc., USA). In this study, the data before the peak point of the bio-hydrogen profiles were fitted by the Gompertz equation because of its suitability for the profile of no gas consumption.

### 2.5. Analytical methods

The cumulative biogas production was examined by a Milli Gascounter (Ritter MGC-1, Germany). The biogas composition was examined by a gas chromatograph (Perkin Elmer Clarus 500, NJ, USA) provided with a thermal conductivity detector (TCD) and a 2-m high-porosity polymer bead-packed column. The temperatures of the column oven, the injection port and detector were set at 150 °C, 50 °C and 150 °C, respectively. Argon was used as the carrier gas at a flow rate of 30 mL/min.

The TCOD, SCOD, TS, VS and pH were measured according to the APHA standard methods (Association et al., 1915). The SCOD was measured in samples that were filtered with membranes (0.45-micrometre pore size). The raw protein was measured by the Kjeldahl method (Hanon-9860, China) (Bradstreet, 1965), and the lipid content was analysed by the Soxhlet extraction method (Hanon-SX500, China) (Hawthorne et al., 2000). The sample was dewatered at 4000 rpm for 10 min to obtain the solid phase and water–oil mixture. The amount of floatable oil was adjusted based on the amount per weight unit of dry kitchen waste.

### 2.6. EEM spectra scan

Before a fluorescence analysis was performed, the TOC was determined using a Multi N/C 2100S TOC/TN analyser (German, Analytik Jena). Hitachi FL-7000 (Japan) fluorescence spectrophotometer was used at a room temperature (approximately 25 °C) on the performing of EEM spectroscopy. The excitation and emission spectra were synchronously scanned at wavelengths of

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