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Research paper

Effective ammonia recovery from swine excreta through dry anaerobic digestion followed by ammonia stripping at high total solids content

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ABSTRACT

This study investigated ammonia recovery from swine excreta through dry anaerobic digestion (AD) and ammonia stripping. The effects of different operation conditions including temperature (25–55 °C), total solids (TS, 20%–35%) and initial pH (7.0–12.0) were assessed on ammonia production in terms of organic nitrogen (organic-N) conversion and process kinetics through 8 days' trials. All experimental data fitted well to the pseudo first-order kinetic models during dry AD and the maximum organic-N mass conversion ratio was estimated to be 65.1% when dry AD was conducted at 55 °C, 20% TS, and initial pH 10.0. Changes in biogas production and its composition signaled the bioactivity of the bacteria involving in ammonia production from swine excreta during dry AD. Finally, greater than 90% of the total ammonia nitrogen (TAN) in swine excreta was efficiently recovered by air stripping at TS about 20%. Restated, the combination of short-term dry AD with ammonia stripping achieved about 60% of total nitrogen recovery efficiency.

1. Introduction

Anaerobic digestion (AD) is a promising technology for pollution control and energy (biogas, with CH_4 as major component) recovery from livestock excreta. And dry AD with a total solids (TS) content of 20%–40% is an advantageous option over traditional wet AD which typically has a much lower TS content of 1%–5%, since the former requires less space, energy input and disposal cost [1]. Nevertheless, dry anaerobic decomposition of nitrogen (N) rich swine excreta inevitably encounters elevated concentrations of ammonia [2]. Excess ammonia, especially the unionized form, exerts negative impacts on methanogens in terms of microbial activity and CH_4 yield, which is reported as the primary cause of digestion failure [3]. What's more, volatile ammonia in the biogas could cause engine erosion and air pollution [3,4]. For these reasons, ammonia control and recovery are necessary for the improvement of dry AD performance.

To find an efficient way to release ammonia from swine excreta is of priority for ammonia recovery. Recently, numerous strategies have been employed to improve organic matter disintegration including anaerobic fermentation at designated initial pHs [5,6], thermal [7], ultrasonic [8], thermo-chemical [9] and ozone [10] pretreatments. Among these methods, anaerobic fermentation of organic substrates has the advantages of easy operation, high efficiency, low energy input and less chemicals consumption. By proper control of experimental conditions, AD of swine excreta can be restricted to hydrolysis and acidogenesis phases which can be purposefully used for the production of ammonia (i.e. ammonia fermentation) or other small molecular substances like volatile fatty acids (VFAs). Restated, all the above-mentioned attempts on the enhancement of organics disintegration were carried out on sludge samples. For instance, enhancement in sludge hydrolysis and VFAs accumulation was observed after waste activated sludge (WAS) being anaerobically fermented at pH 10 for 8 days [5,6]. Although Lin et al. [11] claimed that ammonia was principally derived from the acidification of swine excreta after comparing the effect of different initial pHs on mesophilic hydrolysis and acidification, all their experiments were performed at TS around 5.8% by diluting the raw swine excreta with distilled water. Up to now, most works focused on controlled AD of organic substances for the production of ammonia or soluble organic substances like VFAs from sludge or livestock excreta at low TS contents. Very few trials have been done on ammonia release from excreta through dry AD [12], a more promising technology compared to traditional wet AD when process efficiency, energy recovery, secondary pollution, and handleability of the digestate are taken into consideration [1].

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As for the recovery of ammonia, air stripping coupled with acid absorption seems to be a simple and effective option. To maximize the recovery efficiency of ammonia, two major parameters (i.e. temperature and pH) need to be taken into consideration since they are closely related to free ammonia concentration [13]. It is noteworthy that ammonia stripping from digestate in liquid state has been previously trialed and proven to be effective, ammonia stripping from digested organic matters with a high TS content, however, has received little attention probably due to its low fluidity.

The current work aimed to investigate the feasibility of ammonia recovery from swine excreta through dry AD followed by air stripping. The process kinetics of ammonia production were discussed and compared among different operation conditions (mainly including operation temperature, solids content and initial pH). Also, the performance of dry ammonia stripping was tested. This study is expected to provide useful information for process design and optimization of ammonia recovery from swine excreta by dry AD technology combined with air stripping.

2. Methods

2.1. Raw swine excreta

The raw swine excreta used in this study was sampled from a farm near Tsukuba campus, Ibaraki, Japan in November 2014. The finishing pigs were raised in traditional pig houses paved with chopped straw bedding (particle size about 5–10 mm). The main daily feed ingredients were corn flour, soy flour, soybean curd residue and fishbone. An excreta layer of approximately 10 cm was collected directly from the floor with a shovel. The obtained raw swine excreta containing straw particles was homogenized and stored at 4 °C before the experiments. Its main characteristics were as follows: TS (mass ratio of dry matter to fresh matter) 37.1 ± 0.2%, volatile solids (VS, mass ratio of volatile matter to dry matter) 77.6 ± 0.2%, total nitrogen (TN) 30.2 ± 2.1 g kg^{-1} of VS, total Kjeldahl nitrogen (TKN) 29.8 ± 1.5 g kg^{-1} of VS, total ammonia nitrogen (TAN) 10.6 ± 0.3 g kg^{-1} of VS, total organic nitrogen (TON) 19.2 ± 1.1 g kg^{-1} of VS, mass ratio of organic carbon (C) to N 18.0 ± 0.5, pH 8.6 ± 0.1, and total alkalinity as $CaCO_3$ 173.5 ± 1.2 g kg^{-1} of TS, respectively.

2.2. Experimental conditions

In order to study the effects of different temperature, TS content and initial pH conditions on dry ammonia fermentation, three runs at ambient (25 °C, room temperature), mesophilic (35 °C) and thermophilic (55 °C) temperatures, four runs at TS content of 20%, 25%, 30% and 35%, and six runs at initial pH of 7.0, 8.0, 9.0, 10.0, 11.0 and 12.0 were conducted, respectively. Solid $Ca(OH)_2$ and 218.8 kg m^{-3} HCl solution was used for pH adjustment in this study. The main operation conditions applied in these trials are summarized in Table 1.

All the batch experiments were carried out by employing 100 cm^3 serum bottles as reactors. For each test, at least 12 reactors were prepared: (1) 90 g mixture of excreta and deionized water at the designed TS content was introduced into each bottle without extra inoculum. (2) The vials were flushed with N_2 for 2 min individually before being sealed with rubber stoppers. (3) Then the reactors were incubated anaerobically in a temperature-controlled thermostat for 8 days during which three reactors were sacrificed for sample analysis in every two days. (4) Gas production and gas composition were measured periodically.

For preparation of ammonia-rich digestate, swine excreta was loaded into 3 identical anaerobic reactors (each with a working volume of 1 L) and incubated for 8 days under the optimal AD condition determined in the above 100 cm^3 serum bottle tests, and the digestate was used for ammonia stripping experiments. An air-recirculating system was used for ammonia stripping and recovery as illustrated in Fig. 1. A 100 cm^3 glass vessel installed with motor-driven propeller was loaded with 75 g digestate. Air was pumped into a temperature-controlled vessel (placed in a water bath) for warming up and humidification, flushed through the digestate and then purged into hydrochloric acid solutions (54.7 kg m^{-3}, 0.5 L) to entrap the stripped ammonia. The gas was circulated at a gas flow rate of 0.1 L min^{-1} throughout the experiment to guarantee sufficient absorption of ammonia. Stripping temperature and initial pH were tested at two levels (35 °C, 55 °C and initial pH of 10.0 and 11.0) for 3 h, respectively.

2.3. Analytical methods

The contents of TS, VS, TN, and TKN in the raw excreta were determined in accordance with standard methods [14]. Total alkalinity was determined by titration method to an end point pH of 4.3. Contents of total organic C and N in the solid samples were analyzed by an elemental analyzer (Perkin-Elmer 2004 CHN, USA). pH was measured directly in the digestate using a semi-solid pH meter (Testo 206, Germany).

For the measurement of soluble products, 4 g well mixed sample (fresh matter) was suspended with 40 cm^3 deionized water. The suspension was centrifuged at ~7300 g and 4 °C for 20 min, and the resultant supernatant was used for measurements of TAN and protease activity. TAN was analyzed according to standard methods [14]. TON was calculated by subtracting TAN from TKN.

Hydrolysis has long been recognized as the rate-limiting step for protein disintegration. To investigate the key enzyme activity related to protein hydrolysis and subsequent ammonia production, protease activity was measured on day 6 and at the end of each experiment according to the procedure described by Jang et al. [15]. One unit of protease activity (U) was defined as the amount of protease in the digestate capable of hydrolyzing casein to 1 μg tyrosine within 1 min at the respective cultivation temperature.

Biogas production during ammonia fermentation was recorded directly by reading the scale on the gas tight syringe connected to the reactor and then normalized to standard temperature and pressure (293 K, 101.3 kPa). Gas composition was determined using a gas chromatography (GC-8A, SHIMAZU, Japan) equipped with a thermal conductivity detector (80 °C) and a Porapak Q column (60 °C). Nitrogen (N_2) was used as the carrier gas.

2.4. Calculation

Concentration of free ammonia nitrogen (FAN) was calculated according to the following Equation (1) [13]:

$$\mathrm{FAN} = \mathrm{TAN} \times \left(1 + \frac{10^{-\mathrm{pH}}}{10^{-\left(0.09018 + \frac{2729.92}{T}\right)}}\right)^{-1} \quad (1)$$

where T is the temperature (K).

Ammonia-N yield refers to the amount of TAN produced during the fermentation and was defined by Equation (2). Average organic-N conversion rate (g d^{-1} kg^{-1} of VS or g d^{-1} kg^{-1} of TON) and organic-N mass conversion ratio (%) were employed to assess the efficiencies of ammonia release under different fermentation conditions according to Equations (3) and (4), in which the former was calculated based on initial VS or TON level of the swine excreta used in the experiments, respectively.