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Research paper

Enhanced dry anaerobic digestion of swine excreta after organic nitrogen being recovered as soluble proteins and amino acids using hydrothermal technology

Weiwei Huang[a,b,1], Ziwen Zhao[a,1], Tian Yuan[a], Yang Yu[a], Wenli Huang[c], Zhongfang Lei[a,*], Zhenya Zhang[a,**]

[a] Graduate School of Life and Environmental Sciences, University of Tsukuba, 1-1-1 Tennodai, Tsukuba, Ibaraki 305-8572, Japan
[b] Key Laboratory of Coal Gasification and Energy Chemical Engineering of Ministry of Education, East China University of Science and Technology, No. 130 Meilong Road, Xuhui District, Shanghai 200237, China
[c] MOE Key Laboratory of Pollution Process and Environmental Criteria, College of Environmental Science and Engineering, Nankai University, No. 94 Weijin Road, Nankai District, Tianjin 300071, China

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ABSTRACT

This study investigated the feasibility of N recovery from swine excreta (SE) as value-added soluble proteins and amino acids using hydrothermal (HT) technology for improving dry anaerobic digestion (AD) performance. The effect of temperature on characteristics and transformation behaviors of N species in SE subjected to HT pretreatment at different temperatures (110–210 °C for holding 30 min) was investigated. After HT pretreatment at 150 °C, 58% of the total nitrogen (TN) content in SE became soluble, with 19.03%, 1.55% and 0.56% of TN being converted into soluble proteins, amino acids and urea, respectively. Temperatures higher than 170 °C favored the breakdown of these proteinaceous compounds, leading to accelerated formation of ammonia. After soluble proteins and amino acids being separated by water extraction, CH_4 yield (322.13 $cm^3\ g^{-1}$ of VS_{fed}) from the solid residue pretreated at 150 °C was improved by 51% in comparison to that from raw excreta (213.70 $cm^3\ g^{-1}$ of VS_{fed}), achieving 3 days' shorter in effective CH_4 production period. HT technology could be an effective pretreatment alternative for the recovery of soluble proteins and amino acids and subsequently enhanced dry AD of SE.

1. Introduction

Dry anaerobic digestion (AD) operated at a total solids (TS) content ≥ 15% is now attracting more attention in animal excreta management worldwide. Compared to traditional wet AD (TS < 10%), dry AD has the advantages of smaller reactor space, less energy input and water consumption, and easy handling of the digestate [1]. When dealing with N-rich animal excreta, however, this process inevitably encounters ammonia buildup due to proteins and urea decomposition. Excess ammonia could inhibit methanogenesis, leading to volatile fatty acids (VFAs) accumulation, system buffering capacity breakdown and consequently digestion failure [2].

To mitigate ammonia inhibition so as to improve AD performance, many strategies have been proposed as comprehensively reviewed by Rajagopal et al. [2] and Chen et al. [3]. One option is to lower the N content. Ammonia stripping has been trialed by using various configurations and different gas mediums, resulting in significant increase in CH_4 yield [4–8]. Recently, the feasibility of ammonia stripping from SE at dry state (TS ~ 20%) was justified in terms of technical and kinetic aspects [5]. In order to more efficiently recover ammonia, different methods have also been employed to accelerate organic-N decomposition and ammonia release. Short-term dry fermentation was previously attempted for efficient ammonia accumulation in animal excreta [4,5]. Characteristics of ammonia release during hydrothermal disintegration of chicken excreta was also investigated, through which ammonia release efficiency is more competitive than other pretreatment processes including thermochemical, ultrasonic and controlled fermentation processes [9].

Despite the high efficacy of coupling ammonia stripping with different pretreatment procedures for lowering N content in animal

* Corresponding author.
** Corresponding author.
E-mail addresses: lei.zhongfang.gu@u.tsukuba.ac.jp (Z. Lei), zhang.zhenya.fu@u.tsukuba.ac.jp (Z. Zhang).
[1] These authors contributed equally to this study.

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wastes, this hybrid process could sometimes fail to meet economic needs. Firstly, ammonia stripping needs heating cost, alkali consumption and enclosed equipment. Secondly, as ammonia is a cheap industrial product in abundant supply, the stripped ammonia from animal excreta might not have market advantages in terms of purity and price. Therefore, in this study, an alternative is directed toward the recovery of N as more valuable products like soluble proteins and amino acids. However, up to the present, little attempt has been tried to improve dry AD of SE through N recovery as soluble proteins and amino acids.

Hydrothermal (HT) technology is an attractive process for organic solids solubilization. Previously, it was investigated for the hydrolysis of proteinaceous materials like fish meat [10], silk fibroin [11] and yeast cell [12] into organic acids or amino acids. Recently, its reclamation of macro/micro nutrients from animal excreta was also examined [13,14]. After HT treatment at 160–200 °C, Perera et al. [14] found that ≥ 50% of the total nitrogen (TN) in chicken excreta was solubilized, in which 80% was organic-N. Ekpo et al. [13] also reported that organic-N was the dominant N species in the liquid extracts of swine and chicken excreta after HT treatment at 170–250 °C, and ammonia-N increased gradually with temperature. Nevertheless, more detailed information is necessary on the compositions of soluble organic-N, especially that of proteinaceous amino acids, and their variations under different HT temperatures.

In this study, the feasibility of N recovery as soluble proteins and amino acids was evaluated by using HT technology for dry AD of SE rich in organic-N. Specifically, the effects of HT temperature on N solubilization and accumulation of soluble proteins and amino acids were investigated. After their separation by water extraction, CH_4 production from dry AD of the solid residue was examined. Results from this study could provide new concepts for resources recovery and ammonia inhibition control during dry AD of organic-N rich animal excreta.

2. Materials and methods

2.1. Raw materials

Fresh raw swine excreta (RSE) was collected manually from the concrete floor of a pig finishing unit located in Ibaraki, Japan. No bedding material was used in the pig house. The collected RSE was mixed vigorously to achieve homogeneity and stored at 4 °C before the experiments. Inocula used in this study was taken from a lab-scale mesophilic AD reactor (operated at 37 °C with working volume of 4 L) fed with SE. The characteristics of RSE and the inocula based on 5 parallel analyses were as follows. (1)The RSE had a TS (based on fresh mass) of 25.02 (± 0.11) %, volatile solids (VS, based on fresh mass) of 20.06 (± 0.12) %, total alkalinity of 18.34 (± 0.53) mg g^{-1} as $CaCO_3$, total ammonia nitrogen (TAN) of 0.62 (± 0.02) mg g^{-1}, TN of 4.67 (± 0.15) mg g^{-1} with C/N ratio of 16.76 (± 0.37) and pH of 6.91 (± 0.08). (2) The inocula used in this work had a TS of 19.59 (± 0.21) %, VS of 13.05 (± 0.16) %, total alkalinity of 39.23 (± 3.40) mg g^{-1}, as $CaCO_3$, TAN of 2.04 (± 0.06) mg g^{-1}, TN of 5.09 (± 0.10) mg g^{-1} with C/N ratio of 11.23 (± 0.33) and pH of 7.57 (± 0.07).

2.2. Hydrothermal pretreatment

All HT experiments were conducted in a stainless steel cylindrical reactor (with a working volume of 200 cm^3) equipped with a motor driven propeller (OM Lab-tech MMJ-200, Japan). For each HT experiment, 140 g RSE at TS of 25% was loaded into the reactor. Under a constant agitation of 100 r min^{-1}, the reactor was heated up to the designated temperature (110, 130, 150, 170, 190, or 210 °C) and maintained at this temperature for 30 min. At the end of HT operation, the reactor was cooled with a fan to room temperature (around 25 °C) before opened for analysis of related parameters. Three replicates were performed for each HT temperature condition.

2.3. Water extraction for N nutrient recovery and dry anaerobic digestion of the solid residues

A so-called quick-wash tactic [15] was employed for the extraction and recovery of soluble N nutrients (proteins, amino acids, ammonia, etc.) from the HT pretreated SE. Specifically, the pretreated SE was added with deionized water (5 cm^3 water → 1 g SE) and mixed thoroughly. The mixture was then centrifuged at 7013 × g for 20 min and filtered through filter paper (0.45 μm). The obtained liquids were characterized and the digestibility of solid residues was then tested via 30 days' dry AD.

As for dry AD experiments, 5 glass bottles (101 mm in diameter and 230 mm in height with a working volume of 1000 cm^3) labeled as R-RSE, R-110, R-130, R-150 and R-170 were prepared. The numbers, i.e. −110, −130, −150, and −170 denoted the HT pretreatments were conducted at 110, 130, 150 and 170 °C, respectively. Each bottle was loaded with 300 g excreta sample (untreated RSE or solid residues obtained after water extraction of the HT pretreated SE at 110–170 °C) and 150 g inocula, mixed thoroughly, capped with a silicon stopper, flushed with N_2 and finally kept airtight. A bottle loaded with inocula only was also prepared as control. The bottles were incubated in a water bath with temperature controlled at 37 ± 2 °C. Biogas (CH_4 + CO_2 + H_2) production was quantified by water displacement method, and CH_4 content was analyzed with a GC-8A/TCD (Shimadzu, Japan). The amount produced from the inocula (control) has been subtracted from the reported CH_4 production from SE which was expressed based on per gram of volatile solids (VS) fed to the reactor (g of VS_{fed}).

In addition to daily CH_4 yield (cm^3 d^{-1} g^{-1} of VS_{fed}) and accumulative CH_4 yield (cm^3 g^{-1} of VS_{fed}), effective CH_4 production period (τ_e, d) and averagely effective CH_4 production rate (r_e, cm^3 d^{-1} g^{-1} of VS_{fed}) were also used to assess dry AD performance of SE, which were defined as the duration for achieving 80% of the total CH_4 production and the average CH_4 production rate during τ_e, respectively.

2.4. Sample analysis

Sample preparation and analysis of TS, VS, organic C and N contents, pH, TN, TAN, soluble organic carbon (SOC), VFAs, soluble carbohydrates and proteins were performed according to the procedures described elsewhere [9]. Total alkalinity was determined by titration method to an endpoint of pH 4.3. Amino acids and urea were quantified by an amino acid analyzer (JEOL JLC-500/V2, Japan). Total VFAs were recorded as the sum of acetic, propionic, iso-butyric, n-butyric, iso-valeric, and n-valeric acids. Ortho-P concentration in the separated liquid after water extraction was determined with molybdenum blue method [16]. All sample analysis was repeated at least twice.

2.5. Statistical analysis

Concentrations of TS, VS, TN, total alkalinity and the soluble components in SE were calculated based on fresh mass. All results from the HT experiments were mean values of six repetitions (triplicate HT experiments × replicate sample analysis). During dry AD, results of the solid characteristics were presented as mean values of three repetitions. Statistical analysis was performed using Microsoft Excel 2013 software, and significance in statistical difference was assumed at $p < 0.05$.

3. Results and discussion

3.1. Hydrothermal pretreatment on swine excreta

3.1.1. Changes in solids content and alkalinity

Compared with RSE, the HT pretreated SE samples had a darker color which became increasingly darker with the increase in HT temperature. Fig. 1a depicts the solids content and system pH in different