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Enhancing Anaerobic Digestion of High-solid Sludge with Coupled Ultrasonic and Alkaline Pretreatment: Mechanism Research and a Full-Scale Experiment

Min Ji[1,*] Bo Zhang[1,2], Fen Wang[1], Ruying Li[1], Yingxin Zhao[1]

[1] School of Environmental Science and Engineering, Tianjin University, Yaguan Road 135, Tianjin, 300350, China.

[2] Tianjin Eco-city Water Investment and Construction Co. Ltd, Hexu Road 276, Tianjin, 300467, China

E-mail: jimin@tju.edu.cn; Tel: +0086-22-87402072; Fax: +0086-22-27407956.

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Abstract

Disintegrating sludge with pretreatment technology is an effective way to enhance the anaerobic digestion performance. In this paper, full-scale ultrasonic and alkaline method were applied to pretreat the high-solid sludge. The disintegration mechanism and improvements of anaerobic digestion performance by individual ultrasonic pretreatment, individual alkaline pretreatment and coupled ultrasonic and alkaline pretreatment were discussed. A full scale coupled ultrasonic and alkaline pretreatment test was carried out to evaluate the methane yield and energy balance. The results showed that there was synergetic effect between coupled ultrasonic and alkaline pretreatment. Compared with ultrasonic sequencing batch mode, Ultrasonic continuous mode could accelerate the sludge solubilization rate but inhibit the sludge solubilization degree. In the full scale test, a cumulative biogas yield 0.24-0.28 L/g-VS was observed, which can obtain an energy equivalent to not only offset the energy consumption but also gain net energy harvest.

Keywords: high-solid sludge anaerobic digestion, Ultrasonic disintegration, Alkaline pretreatment,

1. Introduction

Anaerobic digestion (AD) has been widely used to treat the sludge produced in municipal wastewater treatment plants (WWTPs). AD could biodegrade the organic matters in sludge and generate methane, thus reduce the total volume of sludge and recover energy [1]. Most of the full-scale AD projects were carried out with the solid contents of 3% - 5% [2]. AD has been proved to be a promising prospect of application and spreading [3]. However, there were some shortcomings of AD, such as large floor area and high heating cost, which limited its application and development. In order to increase the applicability of anaerobic digestion, high-solid anaerobic digestion (HSAD) might be a feasible solution. HSAD was commonly applied with the solid content higher than 8%, thus it could enhance the space utilization efficiency of anaerobic digester, and, reduce the heating energy demand [4]. So HSAD has drawn more and more attention.

The hydrolysis of organic matters in EPS and cells is the rate-limiting step during AD [5]. High solid content in HSAD led to high viscosity and blocked mass transfer, which worsened the hydrolysis efficiency problem [6]. In order to accelerate the hydrolysis efficiency, pretreatments were usually applied to disintegrate the extracellular polymeric substances (EPS) and microbial cells, which led to the organic matters solubilization from solid to liquid [7]. Many pretreatment methods, such as thermal [8], chemical [9], mechanical [10], physical [11], and coupled methods [12] were applied to enhance the hydrolysis efficiency.

Ultrasound is a widely used mechanical pretreatment method, which can disintegrate sludge effectively by its mechanical shear force [13]. However, the high energy consumption of ultrasound limited its application [14], and it is necessary to optimize the design of ultrasonic reactors [15] and / or couple ultrasound with other pretreatment methods [16]. It was reported that multiple transducers has better performance than single transducer under the same power [8]. Besides, ultrasonic pretreatment was often coupled with other methods such as alkaline [17], microwave [18], ozone [19], and so on, which can achieve a higher disintegration degree and thus reduce the energy demand. Alkaline pretreatment can disrupt sludge flocs and cells, release inner organic matters and accelerate sludge hydrolysis, and consequently improve the performance of subsequent AD [20]. It has been proved that coupled alkaline and ultrasonic pretreatment had a synergetic effect on sludge disintegration [21]. However, few literatures reported the full-scale application of coupled alkaline and multiple-transducer ultrasonic pretreatment.

In this study, a full scale demonstration on coupled ultrasonic and alkaline pretreatment was carried out. The disintegration mechanism of couple ultrasonic and alkaline pretreatment on high-solid sludge (TS=8%) solubilization was discussed, and the improvement of the performance of full scale HSAD by coupled ultrasonic and alkaline pretreatment was evaluated.

2. Materials and methods

2.1 Sludge samples and inoculums

Dewatered sludge was collected from a WWTP in Tianjin, China. The thickened sludge was conditioned with polyacrylamide and dewatered to water content of 80% using a belt filter press. The dewatered sludge was stored at 4°C. Before pretreatment, dewatered sludge was diluted to TS 8%. The inoculum was taken from a full-scale high-solid anaerobic digester in the same WWTP in Tianjin, China. The inoculum was incubated at mesophilic temperature before experiment [22]

The total solid and volatile solid of the diluted dewatered sludge (DDS) and inoculums were listed in Table 1.

Table 1 Characteristics of diluted dewatered sludge (DDS) and inoculums

Parameters	DDS	Inoculums
Total solid (TS, g/L)	80±3	82±4
Volatile solid (VS, g/L)	43±5	34±3

2.2 Lab-scale experiment

For individual $Ca(OH)_2$ pretreatment, different amount of lime (CaO, Technical grade, Rual Co., LTD, Tianjin, China) was first dissolved using tap water with doses of 0.02, 0.04, 0.06, 0.08, 0.1 g/g TS of sludge. Then the solution was used to dilute dewatered sludge to TS 8%. The alkaline pretreatment was implemented in a 1 m^3 tank. The samples were stirred at 80 rpm with a stirrer for 1 hour at ambient temperature.

For individual ultrasonic pretreatment, a multi-transducer ultrasonic path reactor [27] was developed with a total volume of 250 L and a maximum power of 10 kW. Ultrasonic pretreatment was performed in the full-scale ultrasonic reactor. The input power was fixed at 5 kW. By controlling the sludge flow rate, the sludge was exposed to ultrasonic waves for 15, 30 and 45 minutes. The specific energy input can be calculated using Eq. (1 as follows) [23]:

$$E_{spec} = (P \times T)/(V \times TS) \quad (1)$$

Where E_{spec} is specific energy input in kW s/kg of total solids (kJ/kg of total solids), P the ultrasonic power input (kW), T the duration of exposure to ultrasonic waves (s), V the volume of treated sludge (L), and TS the total solids concentration of the sludge (kg/L).

For coupled pretreatment, 120 kg dewatered sludge were first diluted with 180 L $Ca(OH)_2$ solution with different lime doses of 0.02, 0.04, 0.06, 0.08 and 0.1 g/g TS, and then the samples were mixed at 80 rpm with a stirrer for 1 h at ambient temperature. After that, the sludge was pumped into the ultrasonic reactor, where it was disintegrated for 15, 30 and 45 minutes at 5 kW. By diluting dewatered sludge with tap water, a control group was obtained.

2.3 Full-scale test

A novel, efficient and energy saving multi-transducer ultrasonic path reactor (as Fig.1) was developed with a total volume of 250 L and a total power of 10 kW. The sludge could be pretreated with a capacity of 5 m3/h and an energy consumption of 2 kWh/m^3.

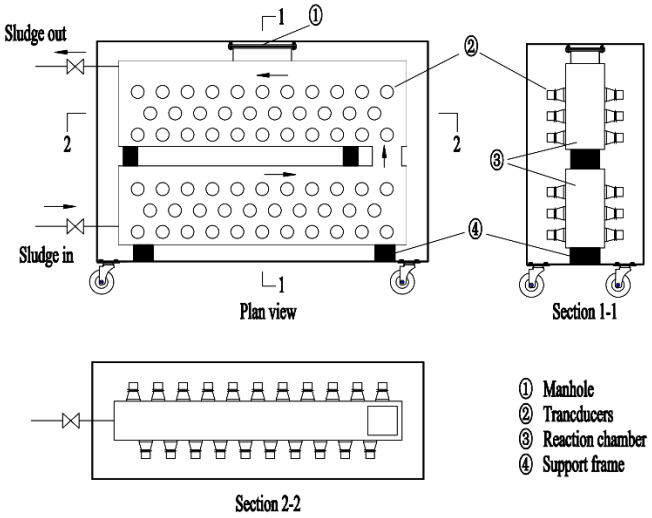


Fig.1 Diagram of ultrasonic reactor

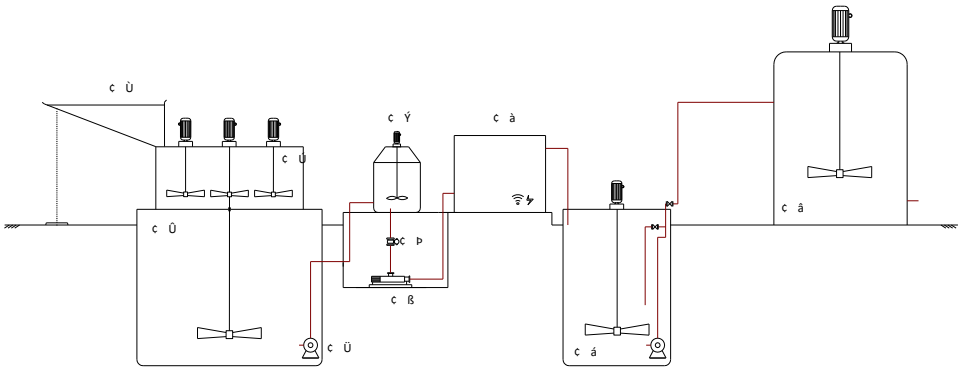
When optimizing the full scale test, ultrasonic pretreatment was applied for 1, 3, 5, 7, 10, 15, 20 min of sequencing batch mode, and 1, 3, 5, 7, 10, 15 min of continuous mode.

Lime pretreatment was implemented in a 3 m^3 tank. For individual $Ca(OH)_2$ pretreatment, different amount of lime (CaO, Technical grade, Rual Co., LTD, Tianjin, China) was first dissolved using tap water with doses of 0.02, 0.04, 0.06, 0.08, 0.1 g/g TS of sludge. Then the solution was used to dilute dewatered sludge to TS 8%. For coupled pretreatment, the dewatered sludge were first pretreated by $Ca(OH)_2$ solution and then mixed at 120 rpm for 1 h. After that, the sludge was pumped into the ultrasonic reactor, where it was disintegrated for 15, 30 and 45 minutes at 10 kW.

By diluting dewatered sludge with tap water, a control group was obtained.

The pretreated sludge was fed into the anaerobic digester with a volume of 200 m^3. The fermentation temperature was 37 °C, and the hydraulic retention time was 15 d.

The flow chart of pretreatment-anaerobic digestion process was shown in Fig. 2.



¢ ÙSludge funnel ¢ ÚAlkaline and Pre-Mixing Pool ¢ ÛMixing Pool ¢ ÜPump ¢ ÝMixing-Buffer Pool
¢ ÞElectromagnetic flowmeter ¢ ßPump ¢ àUltrasonic reactor ¢ áMixing Pool ¢ âAnaerobic digester

Fig.2 Process flow diagram of ultrasonic and alkaline pretreatment and HSAD

2.4 Batch experiment on high-solid anaerobic digestion

The batch experiment on high-solid anaerobic digestion was carried out with an automatic methane potential test system II (AMPTSII, Bioprocess Control, Lund, Sweden). Each digester bottle had a volume of 650 mL. The inoculum and substrate was mixed with a VS ratio 2:1. Nitrogen gas was bubbled into the bottles for 3 min to remove oxygen. The batch experiment was operated for 30 days.

2.5 Analytical methods

Extracellular polymeric substances (EPS), N-acetylglucosamine content and Flow cytometry (FCM) were measured with heating method, Reissig colorimetric method [24] and flow cytometer (FACSCalibur, BD, USA) method, respectively, based on the previous study. TS, VS, COD and total ammonia nitrogen (TAN) were analyzed according to the standard methods [25]. Samples were first centrifuged at 10,000 g for 8 min, the supernatant was filtered through 0.45 μm membrane. Analysis were carried out in duplicate and average values were determined for each set.

3. Results and discussion

3.1 Lab-scale pretreatment

3.1.1 Disintegration mechanism discussion

Most of the organic matters in sludge are located in EPS and microbial cells, the disintegration of EPS and cells caused the release of organic matters [26]. In order to gain some insight into the disintegration process and mechanism, damage to EPS, cell wall and cell membrane was studied. Based on the previous study (data was not shown here) [27], the individual low power ultrasonic pretreatment could hardly disintegrate EPS, cell walls or cell membranes, most of the ultrasonic energy was consumed to transmit in sludge and slightly disintegrated loosely bound EPS (LB-EPS), so there were almost no organic matters solubizalition. The individual 0.04 g/g-TS lime pretreatment could partly disintegrate the EPS but hardly disintegrate cell structure, some of the organic matters located in EPS were released into liquid. When coupled ultrasonic and alkaline pretreatment was applied, EPS, cell wall and cell membrane were all damaged in different extent,

which indicated that the COD solubilization was due to the release of both EPS and intracellular polymers.

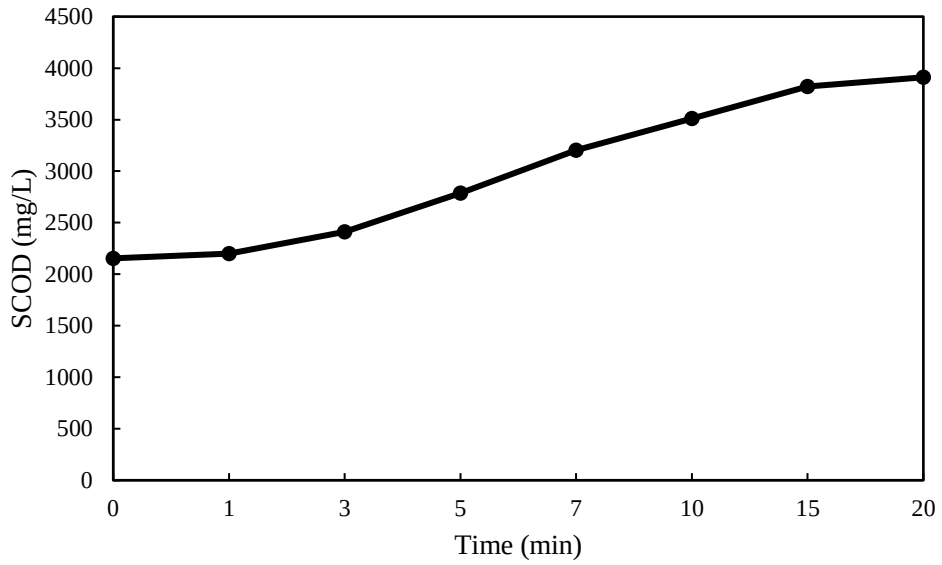
Results indicated that there was synergetic effect when coupled ultrasonic and alkaline pretreatment was applied. This effect could enhance the disintegration degree of EPS, and disintegrate the microbial cell which could not be damaged by individual ultrasonic or alkaline pretreatment.

The reason of the synergetic effect may be as follows: (1) The alkaline pretreatment disintegrated EPS or cells by saponification [28], while ultrasonic pretreatment damaged EPS or cells by its hydraulic shear force [13]. When the lime dose was 0.04 g/g-TS, the saponification of alkali could only swell the EPS and thus release the organic matters from EPS to liquid. While the hydraulic shear force could hardly damage EPS or cells. After alkaline pretreatment, the EPS structure became loose after swelling, hence the EPS were easier to be damaged. Besides that, some flocs lost the EPS layer because of saponification, the cell wall would be exposed directly to the hydraulic shear force caused by ultrasonic pretreatment. Without the EPS protection, the ultrasound could disintegrate these cells easier. (2) The alkaline saponification process was mainly reacted in the first 30 min [29]. After 30 min, the SCOD increased limitedly, while the pH value of the sludge was remained at about 9.0. When coupling lime with ultrasonic pretreatment, the hydraulic shear force could stir or disintegrate the EPS and cells, which could enhance the alkaline saponification under pH 9.0 and lead to further swelling of EPS and cells.

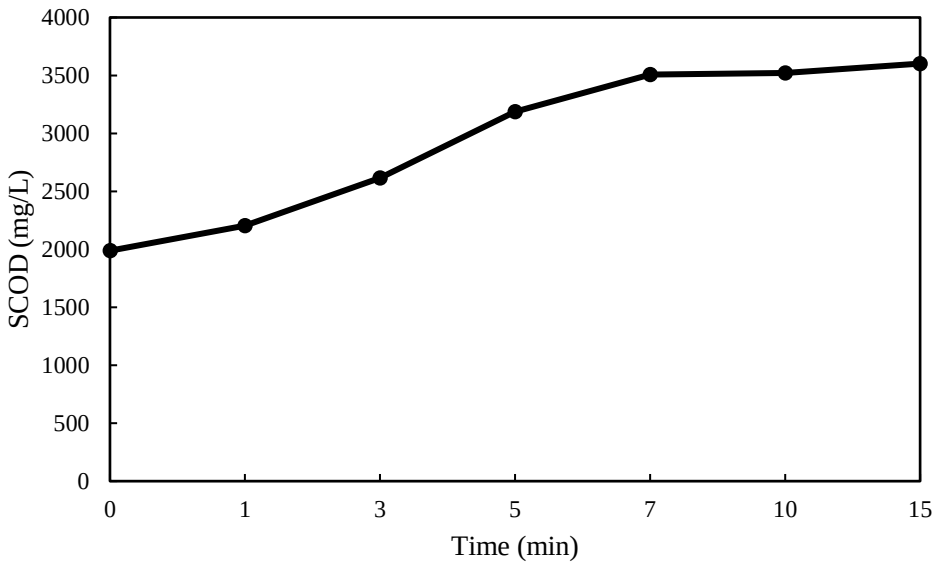
3.2 Optimization of Full-scale Ultrasonic pretreatment operation

3.2.1 COD solubilization

During the full-scale experiment, the COD solubilizaition results after coupled 0.04 g/g-TS CaO and sequencing batch / continuous ultrasonic pretreatment were contrasted (Fig.3). Based on the previous results, 15 min sequencing batch ultrasonic pretreatment could disintegrate sludge effectively, and obtain a synergetic effect with alkaline pretreatment, 30 min or more time had no synergetic effect with alkali. Thus, the sequencing batch ultrasonic pretreatment with duration from 1-20 min were studied. As shown in Fig.3 (a), the SCOD concentration increased with the time increasing. The time 0 meant individual 0.04 g/g-TS CaO pretreatment, and the SCOD concentration was 2154 mg/L. When the coupled pretreatment was applied, the SCOD concentration increased with the ultrasonic duration. The SCOD concentration of sludge after coupled 0.04 g/g-TS CaO and 1 min, 3 min, 5 min, 7 min, 10 min, 15 min and 20 min pretreatment was 2201 mg/L, 2411 mg/L, 2787 mg/L, 3204 mg/L, 3512 mg/L, 3821 mg/L and 3911 mg/L, respectively. In the first 15 min, the SCOD concentration increased obviously with the ultrasonic duration, while further increase of time led to limited SCOD concentration increase. Thus, 15 min is the effective disintegration time of sequencing batch ultrasound when coupled with 0.04 g/g-TS CaO.



(a)



(b)

Fig 3 SCOD concentration of sludge with different duration of sequencing batch (a) and continuous (b) ultrasound

The continuous ultrasonic pretreatment with the duration of 1-15 min was coupled with 0.04 g/g-TS CaO, and the results were shown in Fig.3 (b). The SCOD concentration increased with the disintegration time. After 1 min, 3 min, 5 min, 7 min, 10 min and 15 min, the SCOD concentration was 2204 mg/L, 2616 mg/L, 3188 mg/L, 3508 mg/L, 3520 mg/L and 3602 mg/L, respectively. In the first 7 min, the SCOD concentration increased linearly with the time increasing, and the further increase of time resulted in changeless SCOD concentration. The results indicated that the coupled 0.04 g/g-TS CaO and continuous ultrasonic pretreatment had an effective ultrasonic disintegration time of 7 min.

3.2.2 Anaerobic digestion performance

The flow rate of screw pump used in the process was 5 m^3/h, and the total volume of the two reactors in series operation was 500 L. So the duration of single-circle continuous ultrasonic pretreatment was 6 min.

The effective ultrasonic disintegration time of sequencing batch and continuous ultrasonic pretreatment was 15 min and 7 min, respectively. In order to contrast the disintegration effect on anaerobic digestion of sequencing batch and continuous ultrasonic pretreatment under the same duration and energy input, 6 min and 18 min ultrasonic retention time were applied (Fig.4). Single-circle and triple-circle continuous ultrasonic pretreatment were applied.

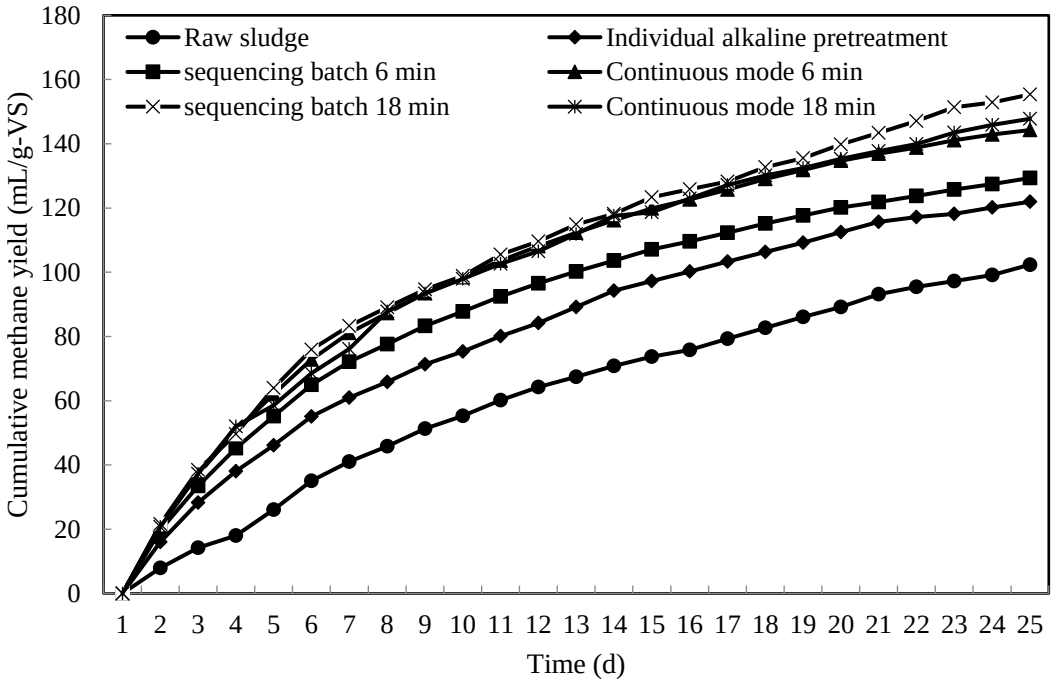


Fig 4 Effect of different operating modes by coupled ultrasonic and alkaline pretreatment on cumulative methane yield

As shown in Fig.4, compared with the raw sludge, the cumulative methane yield of individual alkaline pretreated sludge increased by 19.2%. When coupled alkaline and ultrasonic pretreatment was applied, the cumulative methane yield of 6 min and 18 min sequencing batch ultrasonic pretreated sludge increase by 26.5% and 52.0%, and the cumulative methane yield of 6 min and 18 min continuous ultrasonic pretreated sludge increased by 41.5% and 44.5%. The results showed that the 6 min continuous mode acted better than 6 min sequencing batch ultrasonic mode, and the 18 min sequencing batch mode was better than 6 min continuous ultrasonic mode. It was due to that the sludge mixing of streaming was beneficial to facilitate the uniform distribution ultrasound field [30], so that the ultrasonic continuous mode could accelerate the sludge solubilization in the beginning of 6 min, While with time further increasing, the sludge solubilization degree would not be enhanced.

The higher cumulative methane yield of 18 min sequencing batch mode was 8% higher compared with that of 6 min continuous mode, while the energy input of 18 min sequencing batch mode was 3 times higher. Thus, in the full-scale experiment, 6 min continuous mode was selected for the following experiment.

3.3 Full-scale experiment

The anaerobic digestion performance from the start-up to stable operation was shown in Fig.5.

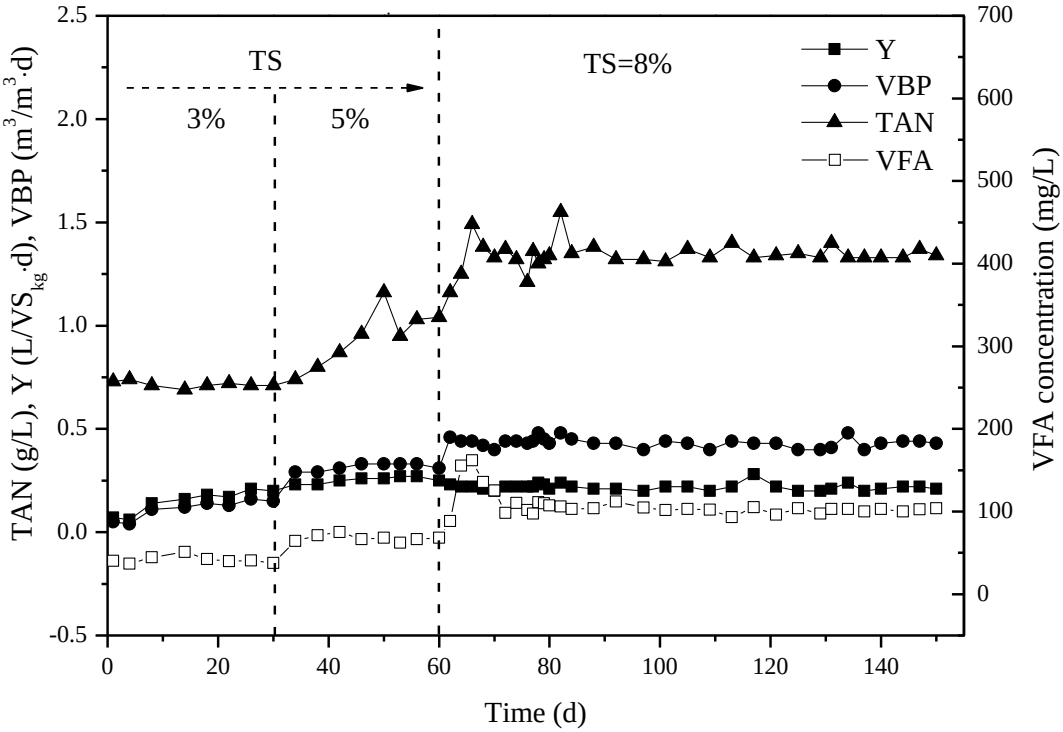


Fig.5 Anaerobic digestion performance of full-scale experiment

As shown in Fig.5, the TS content during the start-up period was 3%-5%, and increased to 8% during stable operation period. The 3%-8% TS sludge were all pretreated by coupled ultrasonic and alkaline pretreatment. When the TS increased to 8%, the yield (Y) of biogas, volumetric biogas production (VBP), TAN and volatile fatty acid (VFA) fluctuated within the range of 0.24-0.28 L/g-VS, 0.4-0.48 $m^3/m^3 \cdot d$, 1.21-1.49 g/L and 103.2-111.9 mg/L, respectively. During the anaerobic digestion process, the VBP, TAN and VFA increased with the TS, while the yield (Y) of methane decreased gradually. Higher VBP was due to the higher amount of organic matters in the reactor, and more organic matters were converted to biogas. Protein was the main organic matter in sludge [31], more protein could be degraded under higher TS and then more TAN was generated [32]. Higher TS also led to higher sludge viscosity [33], which inhibited mass transfer, thus the VFA could not be utilized in time, which led to lower methane yield.

3.4. Energy balance

The total energy consumption of all pretreatment equipment and anaerobic digestion system, and the energy equivalent of biogas production during anaerobic digestion process was shown in Fig.6. The total energy consumption increased with the TS increasing from the start-up period to stable operation period. When the TS was 3%, the total energy consumption was 92.6 kWh, and the energy equivalent of biogas production was approximately equal. With the TS increased to 5%, the total energy consumption was 89.7 kWh, and the energy equivalent of biogas production was about 140-180 kWh, thus the anaerobic system produced more energy than it consumed. When the TS reached 8%, the total energy consumption was 99.5 kWh, the energy equivalent of biogas production was about 190-220 kWh, the 8% TS system could remain more net energy compared with TS of 5%. The energy balance results showed that the pretreatment and HSAD system could achieve net energy harvest.

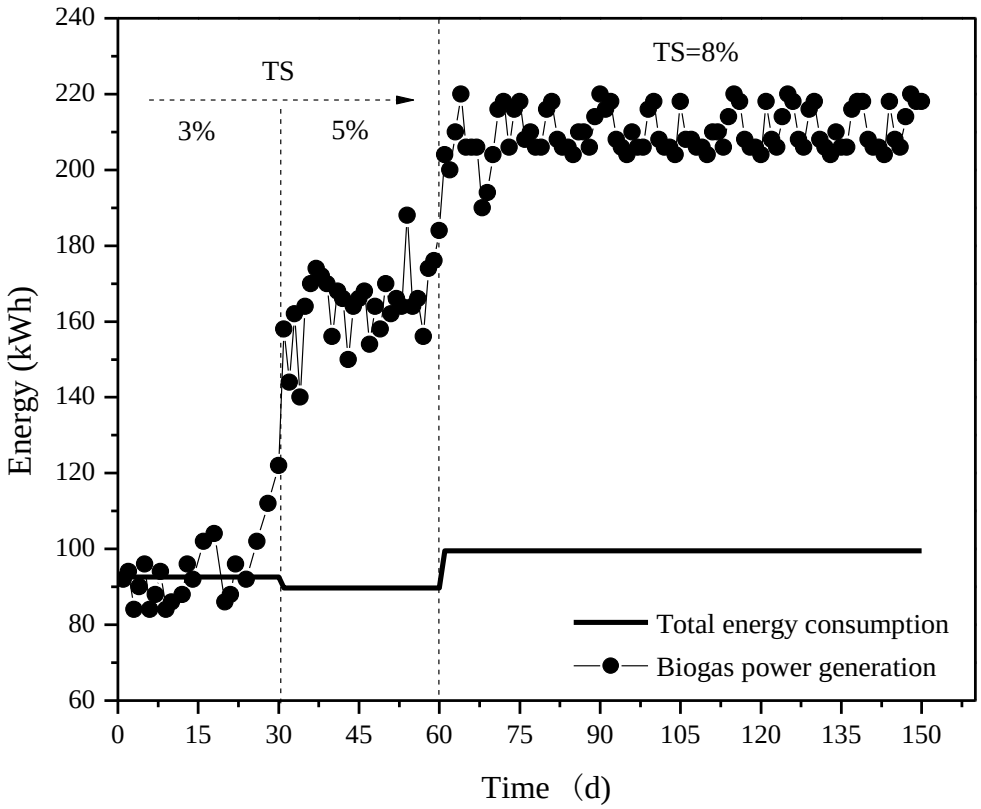


Fig 6 Equipment energy consumption and energy equivalent of biogas production during anaerobic digestion process

4. Conclusions

(1) There was synergetic effect when coupled ultrasonic and alkaline pretreatment was applied, which was due to the different mechanisms of these two methods.

(2) Ultrasonic continuous mode could accelerate the sludge solubilization in the beginning of 6 min, while with time further increased, the sludge solubilization degree would not be enhanced.

(3) Under 8% TS anaerobic digestion, the yield (Y) of methane, VBP, TAN and VFA fluctuated within the range of 0.4-0.48 m^3 $CH_4/m^3 \cdot d$, 1.21-1.49 g/L, 103.2-111.9 mg/L and 0.24-0.28 L/g-VS, respectively. The pretreatment and HSAD system could gain net energy.

Acknowledgements

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