Experimental continuous sludge microwave system to enhance dehydration ability and hydrogen production from anaerobic digestion of sludge

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ABSTRACT

Dehydrating large amounts of sludge produced by sewage treatment plants is difficult. Microwave pretreatment can effectively and significantly improve the dewaterability and hydrogen production of sludge subjected to anaerobic digestion. The aim of this study was to investigate the effects of different microwave conditions on hydrogen production from anaerobic digestion and dewaterability of sludge. Based on an analysis of the electric field distribution, a spiral reactor was designed and a continuous microwave system was built to conduct intermittent and continuous experiments under different conditions. Settling Volume, Capillary Suction Time, particle size, and moisture content of the sludge were measured. The results show that sludge pretreatment in continuous experiments has equally remarkable dehydration performance as in intermittent experiments; the minimum moisture content was 77.29% in the intermittent experiment under a microwave power of 300 W and an exposure time of 60 sec, and that in the continuous experiment was 77.56% under a microwave power of 400 W and an exposure time of 60 sec. The peak measured by Differential Scanning Calorimeter appeared earliest under a microwave power of 600 W and an exposure time of 180 sec. The heat flux at the peak was 4.343 W/g, which is relatively small. This indicates that microwave pretreatment induced desirable effects. The maximum yield of hydrogen production was 7.967% under the conditions of microwave power of 500 W, exposure time of 120 sec, and water bath at 55°C. This research provides a theoretical and experimental basis for the development of a continuous microwave sludge-conditioning system.

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Introduction

In 2014, more than 3500 sewage treatment plants were built and put into operation in China, which provided new daily treatment capacity of 140 million m³ (Qu et al., 2014). Sludge, a by-product of sewage treatment, contains not only organic matter, nitrogen, phosphorus, and potassium; but also biodegradable substances, heavy metals, salts, pathogens, and parasites. It is difficult to dehydrate and has problematic biochemical properties. Because a large quantity of sludge produced on a daily scale, covering a wide area, if not handled properly, it will cause serious secondary pollution of the environment (Hu et al., 2005; Wang et al., 2014). In order to facilitate its transport and resource recovery, sludge needs to be conditioned using physical, chemical, and biological...
The application of the DSC for moisture testing of sludge is still wide variety of organic, inorganic, polymeric, metallic, semi properties, phase transition, and crystallization kinetics of a temperature control programs, and is widely used for study thermal used for measuring thermal effects of samples under temper using Differential Scanning Calorimeter (DSC). This technique is transition temperature of internal materials) was determined relationship between temperature and heat flow (related to the thermal mechanical dehydration (Colin and Gazbar, 1995). The relation larger the volume of bound water, the more difficult the measure the degree of difficulty of mechanical dehydration: the...

Previous studies have experimentally studied the effect of microwaves on the characteristics of sludge. Several research teams (Eskicioglu et al., 2009; Hong et al., 2006; Park et al., 2004) proved that microwave pretreatment is capable of cracking sludge flocs and biological cells, and thereby capable of releasing organic matter and transforming them into their soluble phase. Liang et al. (2012) applied microwave radiation to condition sludge, and discovered that microwave can significantly improve the dehydration performance of sludge under appropriate conditions. Zhou et al. (2013a, 2013b) carried out research on microwave conditioning of sludge and found that dehydration performance of sludge exhibited significant changes after microwave conditioning, affecting properties such as soluble chemical oxygen demand, particle size of sludge, and viscosity. Wojciechowska (2005) applied microwave to condition sludge and found the specific resistance to filtration of mixed sludge and anaerobic digested sludge, was reduced by 27% and 26%, respectively. Eskicioglu et al. (2008) found that at 90°C, dehydration performance of sludge was improved by about 40% (pretreatment with microwave at 90°C for 10 min), and that the Capillary Suction Time (CST) of sludge with total solid content of 5.8% was significantly decreased. Water distribution and the mechanical dehydration performance of sludge are closely associated, and can be used directly to measure the degree of difficulty of mechanical dehydration: the larger the volume of bound water, the more difficult the mechanical dehydration (Colin and Gazbar, 1995). The relationship between temperature and heat flow (related to the thermal transition temperature of internal materials) was determined using Differential Scanning Calorimeter (DSC). This technique is used for measuring thermal effects of samples under temperature control programs, and is widely used for study thermal properties, phase transition, and crystallization kinetics of a wide variety of organic, inorganic, polymeric, metallic, semiconductor, pharmaceutical, and biological materials. Although the application of the DSC for moisture testing of sludge is still relatively rare, its excellent performance has made it widely used in studies of crude oil, high-concentration oil-water emulsions, and drilling (Clausse et al., 2005; Dalmazzone et al., 2006, 2010; Garti et al., 2000; Le Parlouer et al., 2004; Kovalchuk and Masalova, 2012; Zhu et al., 2011). The DSC has also been applied in the food industry (Chen et al., 2010). In this study, the DSC was used to measure the bound water content of sludge to characterize its dehydration performance (Zhou et al., 2014).

Carrère et al. (2010) reviewed studies on the effect of different pretreatment methods on anaerobic fermentation of sludge. Pino-Jelicic et al. (2006) and Hao et al. (2011) found that the amount of biogas produced from sludge increased when the sludge was pretreated using microwave radiation. Shen et al. (2009) compared the effect of heat pretreatment, microwave pretreatment, and chloroform pretreatment on the anaerobic fermentation of organic waste, and found that the microwave pretreatment is the most suitable method for improving hydrogen production from anaerobic reaction.

Current microwave sludge pretreatment research has been focused on the use of intermittent microwave conditioning, research based on continuous microwave conditioning is still relatively rare. Because the production of sludge in sewage treatment plants is continuous, it is necessary to develop a continuous microwave conditioning device for sludge, if this conditioning apparatus is to be used at industrial scale. Based on previous work on intermittent microwave conditioning, a continuous microwave conditioning device was created for these experiments. It was used to compare the effects of intermittent and continuous microwave conditioning on dehydration performance of sludge. Additional experiments were performed to assess the effects of microwave pretreatment on hydrogen production from anaerobic digestion.

1. Method

1.1. Design of continuous microwave conditioning system

To obtain the device needed for continuous sludge conditioning, a main reactor was designed and resonant-cavity was numerical simulated. According to the characteristics of the processing materials and microwave conditions, a continuous conditioning system was established, as shown in Fig. 1. It included four Fig. 1 – Continuous microwave sludge conditioning system.
The electric field distribution inside the cavity was observed as reactor with a spiral coil structure. This places the pipe within a strong electric field able to achieve efficient and uniform microwave pretreatment of sludge.

1.4. Experimental materials and equipment

The sludge is an excess sludge taken from Huang cun, Daxing District sewage treatment plant, which follows the inverted Anaerobic-Anoxic-Oxic (A2O) process. When the supernatant was removed after standing for 24 hr, the water content of the sludge was 99.24%. The continuous microwave sludge pretreatment system had input power of 0–700 W at a frequency of 2450 MHz, and a processing capacity of 0.5–500 mL. A Lange BT300-2J peristaltic pump was adopted as the feed device (flow range of 0.07–1600 mL/min), and a #17 hose was used. The suction filtration method was used to dehydrate the sludge after pretreatment at a suction filtration pressure of 70 kPa. The moisture content was measured using an MA150 infrared moisture analyzer, and CST was measured using a Triton Electronics Type 319 Multi-CST analyzer. The particle size was measured using a Beckman Kurt Multisizer3 particle count and particle size analyzer. The hydrogen gas production was determined using a SP-3420A gas chromatograph, a thermal conductivity detector with a 5A molecular sieve, and a 3 m × 3 mm chromatographic column, with TCD detector.

1.5. Experimental procedure

1.5.1. Microwave conditioning experiments

Intermittent and continuous microwave conditioning sludge experiments were conducted under experimental setting of the microwave power was 300, 400, 500, 600, or 700 W, at exposure times of 30, 60, 120, 180, or 240 sec. The settling volume (SV) percentage of the conditioned sludge was measured after it settled for 30 min in a measuring cylinder. The CST values and particle size of the conditioned sludge were measured, and the moisture content of the sludge was measured after suction filtration. Intermittent and continuous microwave conditioning sludge experiments were conducted. During the experiments, to ensure that the amounts of sludge used in the two experiments were the same, the amount of sludge was controlled by adjusting the pump flow rate in the continuous experiments. Because the volume of the continuous-type reactor was 142.37 mL, it was assumed that the desired flow rate of the reaction was 142.37 mL/min, and the maximum flow rate was 1600 mL/min when the peristaltic pump was set at 600 r/min.

\[
Q_m/n_m = (Q/t)/60/n
\]  \hspace{1cm} (1)

In Eq. (1), \(Q_m\) is the maximum flow of the pump (mL/min); \(n_m\) is the maximum speed of the pump (r/min); \(Q\) is the flow (mL/min); \(t\) is the conditioning time (sec); and \(n\) is the speed (r/min).

When the conditioning time was 30, 60, 120, 180, or 240 sec, the speed of the pump calculated using Eq. (1) was 107, 53, 27, 18, or 13 r/min, respectively.

1.5.2. Anaerobic digestion experiments

Each experiment required 200 g of sludge and 200 g of wastewater, which contained anaerobic bacteria, packed in 500 mL suction filtration bottles. These were placed in the microwave.
conditioning device, at which the microwave power was set to 300, 400, 500, or 600 W, and the exposure time set to 60, 120, 180, or 240 sec. The conditioned sludge was charged with nitrogen for 1 min to drive off oxygen in the bottle, and then a suction-filtration bottle was placed in a water bath at 55°C. The anaerobic digestion time was 72 hr, and the gas produced was collected using aluminum foil gas collecting bag which has characteristics of chemical stability, simple operation and reliably sealing.

2. Experimental results and discussion

2.1. Settling Volume

After the sludge was conditioned by microwave, 100 mL sludge samples were taken, and after standing for 30 min, the SV (mL) of sludge was measured (Tables 1 and 2). After comparison, it was found that the SV value from the continuous sludge conditioning experiment was less than that from the intermittent conditioning experiment, under the same conditions. At short exposure times (30–120 sec), the difference between continuous and intermittent experiments was not significant. The mechanisms by which microwaves act on sludge, include thermal and non-thermal effects. The thermal effect is due to intermolecular friction and dielectric loss, electricity is converted into heat, so the system heats up rapidly. The non-thermal effects generally refer to the polarization of macromolecules in the electromagnetic field that generates directional arrangement. This is likely to cause structural damage, bring about changes in the particle size and density of the sludge, and lead to changes in its settlement characteristics.

2.2. Capillary Suction Time

The CST value of the sludge was measured after microwave conditioning. The CST trends from the intermittent and continuous sludge conditioning experiments were shown in Fig. 4a and b respectively. Under conditions of low microwave power (300–500 W), the results from the intermittent experiments were lower than the results from the continuous experiments. The CST values in the continuous experiments were small, with short exposure time at high microwave power (600–700 W).

Fig. 3 – Electric field distribution of cavity: (a) A–A section, (b) B–B section, (c) C–C section, (d) 3D model.
Fig. 4a shows that most of the CST values fluctuated in a narrow range from 11 to 13 sec. Fig. 4b shows that the minimum CST was 9.3 sec at a microwave power of 600 W and an exposure time of 60 sec. When the microwave power was 400 W and the exposure time 240 sec, the maximum experimental value was obtained (16.6 sec). CST can indicate dehydration performance of sludge to a certain extent. Likewise, the CST of the continuous and intermittent experiment was found to be low and showed little difference under short exposure time (30–60 sec). It is generally believed that the smaller the CST value of the sludge, the more easily the sludge can be dehydrated. However, sludge dehydration will be affected by the characteristics of the sludge, so using CST value alone has certain limitations. To better understand the change in sludge flocs after microwave conditioning, it is necessary to characterize their particle size, bound water content, and the moisture content of the sludge.

### 2.3. Particle size

Floc characteristics have been used in research to measure the dehydration performance of sludge. In this work, the

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particle size of the sludge was measured using a Beckman Coulter Multisizer3 after microwave conditioning. Among the data on the available mean diameters (d_{10}, d_{25}, d_{50}, d_{75}, and d_{90}), the data for d_{50} was selected and reported in Tables 3 and 4. Microwave power and microwave exposure time affect the sludge particle size distribution; at the same microwave power, the particle size of the sludge generally presented a downward trend with increasing microwave exposure time. The d_{50} of the intermittent experiments was roughly in the range 9–13 μm, whereas, that of the continuous experiment was mostly in the range 6–13 μm, indicating that sludge particles were broken up to a greater degree in the continuous microwave conditioning experiments.

2.4. Measurement result from Differential Scanning Calorimeter

DSC is used to characterize the moisture content of the pretreated sludge; for example, the condensation heat of pure water indicated by integration with the DSC peak area was 334.6 J/g. The condensation heat of the sludge was 168.1 J/g under the experimental condition of 600 W microwave power and 30 sec exposure time; the moisture content of the sludge was 99.58% (not dehydrated). Therefore, the real condensation heat of water in the pretreated sludge was 49.55% (i.e., 49.55% of the water was still unfrozen in the vicinity of 0°C, thus, the bound water content of the sludge was 117.48 kg/kg DS by conversion). Similarly, when the microwave power was 600 W and the exposure time was 60, 120, 180, or 240 sec, the condensation heat of the sludge, the real condensation heat of water in the sludge, and the bound water content of the sludge were shown in Table 5, indicating bound water contents of 49.55%, 63.65%, 53.96%, 52.31%, and 55.43%, respectively. Moreover, the peak occurred latest when the exposure time was 240 sec, and produced a relatively large heat flow at the peak (4.684 W/g). The first peak occurred earliest when the exposure time was 180 sec, for which the heat flow at the peak (4.343 W/g) was relatively small. The sooner the peak occurred, the better was the microwave effect.

2.5. Moisture content

The moisture content of sludge pretreated by microwave exposure and suction filtration was measured to visually show changes in the sludge after microwave conditioning. Changes in the moisture content of the sludge during the intermittent experiments were shown in Fig. 5a; those of the continuous experiments were shown in Fig. 5b.

As shown in Fig. 5a, compared with the original moisture content of the sludge, there was a significant decline after

| Table 3 – d_{50} of sludge after continuous pretreatment (μm). |
|---|---|---|---|---|---|---|
| Exposure time (sec) | 300 W | 400 W | 500 W | 600 W | 700 W |
| 30 | 9.83 | 11.39 | 10.71 | 11.68 | 12.09 |
| 60 | 10.42 | 11.42 | 11.17 | 11.46 | 11.54 |
| 120 | 11.45 | 10.58 | 11.04 | 10.62 | 11.35 |
| 180 | 11.16 | 10.94 | 10.69 | 9.447 | 11.12 |
| 240 | 10.81 | 10.37 | 10.54 | 9.434 | 3.585 |

| Table 4 – d_{50} of sludge after intermittent pretreatment (μm). |
|---|---|---|---|---|---|---|
| Exposure time (sec) | 300 W | 400 W | 500 W | 600 W | 700 W |
| 30 | 10.97 | 12.15 | 12.22 | 4.201 | 10.01 |
| 60 | 6.519 | 11.47 | 11.77 | 11.57 | 11.61 |
| 120 | 6.056 | 10.97 | 10.68 | 10.8 | 10.43 |
| 180 | 9.794 | 10.35 | 10.8 | 10.43 | 10.62 |

| Table 5 – Bound water content of sludge. |
|---|---|---|---|---|
| Exposure time (sec) | CST (sec) | Condensation heat (J/g) | Bound water content (%) | Moisture content (%) |
| 30 | 6.8 | 168.1 | 49.55 | 88.85 |
| 60 | 6.5 | 121.1 | 63.65 | 88.90 |
| 120 | 8.4 | 153.4 | 53.96 | 88.34 |
| 180 | 7.5 | 158.9 | 52.31 | 88.17 |
| 240 | 8.5 | 148.5 | 55.43 | 84.47 |
intermittent microwave conditioning. The lowest moisture content (77.29%) was achieved under the conditions of 300 W microwave power and 60 sec exposure time, and the moisture content decreased by 21.95% compared with that of the original sludge (99.24%). In this case, the dehydration performance was significantly improved. As shown in Fig. 5b, the lowest moisture content of 77.56% in the continuous experiment was obtained at a microwave power of 400 W and an exposure time of 60 sec; the moisture content decreased by 21.68%, compared with the original sludge. When the microwave conditioning was performed at high power (600–700 W), the moisture content of the continuous experiment appeared to be lower than that of the intermittent experiment during shorter exposure time (60 sec). When the power was 600 W, the moisture content of the continuous experiment was 78.52%, a reduction of 2.2%, compared with intermittent experiments (80.72%) under the same conditions. When the power was 700 W, the moisture content of the continuous experiment was 79.51%, a reduction of 1.45% from that of the intermittent experiment (80.96%) under the same conditions. In summary, the moisture contents of the continuous and intermittent experiments were found to be low and showed little difference under short exposure times (30–60 sec), in other words, sludge pretreated in continuous experiments has equally remarkable dehydration performance as intermittent experiments.

The main reason for the high moisture content at long exposure times is that the radiation effect is stronger and results in smaller sludge particle size. This makes suction filtration difficult and causes the moisture content to remain high. This conclusion can be confirmed by comparing suction time in the experiments.

2.6. Hydrogen production from anaerobic digestion

To improve the hydrogen production from anaerobic digestion, the activity of methane bacteria must be inhibited and methane production reduced. After reviewing the literature, we found that anaerobic digestion can be conducted in a wide range of temperatures (40–65°C). Increasing the temperature of the water bath can, to a certain extent, effectively inhibit the activity of methane bacteria but the activity of hydrogen producing microorganisms is not weakened, so the temperature of anaerobic digestion experiments was set to 55°C. For sludge, hydrogen production from anaerobic fermentation was 1.045% at a water bath temperature of 55°C without microwave conditioning.

Fig. 6 shows the gas chromatogram of anaerobic digestion at a microwave power of 300 W with exposure times of 60, 120, 180, and 240 sec (the first peak was hydrogen, the second peak was oxygen, the third peak was methane). Different microwave conditions might influence hydrogen and methane production during anaerobic fermentation. The hydrogen content of the sludge pretreated under different microwave conditions was determined and shown in Table 6.

Table 6 shows that microwave power and exposure time affected hydrogen production by anaerobic fermentation. The hydrogen production from anaerobic digestion without microwave conditioning was less than that from conditioned sludge. The hydrogen production was 7.967% under the condition of microwave power of 500 W and exposure time of 120 sec, higher than that from sludge without conditioning by 6.922%.

3. Conclusions

In microwave conditioning experiments utilizing a newly constructed continuous microwave conditioning system, the effects of different microwaving conditions on hydrogen production from anaerobic digestion and dehydration performance of sludge were studied. SV, CST, $d_{50}$, and water content of various sludge pretreatments were compared and analyzed. From the experimental results, the following conclusions were drawn.

(1) Through analysis of the electric field distribution inside the cavity, a more reasonable microwave reactor was designed and successfully built. This continuous microwave conditioning device laid the foundation for further experiments and system optimization.

(2) Microwave technology can improve the dehydration performance of the sludge. Intermittent and continuous experiments significantly improved the dehydration performance of the sludge. The moisture contents of the continuous and intermittent experiment were found to be
Table 6 – Hydrogen production after anaerobic digestion under different microwave conditions(%).

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