# Enhancing anaerobic biodegradability and dewaterability of sewage sludge by microwave irradiation

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**Abstract:** Thermal hydrolysis was an effective pretreatment for the sewage sludge by improving dewatering and anaerobic digestion but the heating was a time-consuming process. This study focused on the effects of the microwave irradiation by using high temperature and pressured system on sewage sludge hydrolysis. The results obtained in current research showed that sludge could absorb microwave energy with high efficiency and then be hydrolyzed fast. The solubilization of suspended solid (SS), volatile suspended solids (VSS) and the increasing of the level of soluble chemical oxygen demand (SCOD) in liquid fraction showed that the microwave irradiation could be shortened to 5 min under a temperature range of 80°C-170°C. The highest value of VSS dissolution ratio (36.4%) was obtained at 170°C for 30 min. The COD dissolution ratio was about 25% at 170°C. The improvement of the biogas production from both mixture sludge and waste activated sludge was 20.2% and 25.9% respectively at 170°C for 10 min. The dewaterability of sludge was greatly improved. Subsequently, the sludge volume could be reduced by 60% with microwave irradiation.

**Keywords:** sludge, microwave irradiation, biogas production, dewaterability
**DOI:** 10.3965/j.ijabe.20171002.2871

**Citation:** Gao F, Xia Z, Wang W. Enhancing anaerobic biodegradability and dewaterability of sewage sludge by microwave irradiation. Int J Agric & Biol Eng, 2017; 10(2): 224–232.

## 1 Introduction

Wastewater treatment plants produced a large amount of sludge including primary and waste activated sludge. It was widely known that sludge was not easy to be treated and disposed due to its low biodegradability and high moisture content[1]. The traditional treatment methods such as anaerobic digestion processes, aerobic composting and incineration were normally at a low efficiency or require pretreatment[2]. Therefore, an effective treatment and disposal method of sludge has been one of the major concerns in wastewater treatment sector. Thermal hydrolysis has been recognized for many years as a useful pretreatment enhancing sludge bio-biodegradability and dewaterability[3,4]. Normally, sludge was heated to a temperature of 150°C-190°C for over 30 min by adopting thermal hydrolysis pretreatment[5]. The process was effective to disrupt bacterial cells, hydrolyzed solid fractions in sludge and then release the soluble materials into liquid fraction[6]. The hydrolysis of the organic matters was a dominant characteristic to distinguish the heat treatments with other methods. Small molecular weight materials would be generated during the heating process. As a result, the water affinity of solid, colloid and inorganic materials would be greatly reduced and the dewaterability would be improved significantly. In other hand, the sludge could be readily decomposed into methane by anaerobic digestion processes with thermal hydrolysis pretreatment. In the study of Brooks[4], 40%-60% of volatile suspended solids (VSS) are solubilized from waste activated sludge (WAS) at 170°C. Haug et al.[7] calculated a 25% of

**Received date:** 2016-09-26 **Accepted date:** 2016-12-16
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dropping energy production using the heat treatment compared to the conventional digestion. The high temperature pretreatment required energy input. However, the anaerobic digestion of sludge after heat treatment produce net energy output[5,8]. With the purpose of heating sludge, microwave irradiation might serve as an alternative way by providing rapid and uniform heating throughout the material.

In recently years, microwave irradiation as a novel technique to treat sludge has attracted many interests[9,10]. A uniform microwave field generates energy through the realignment of dipoles with oscillating electric fields to generate heat both internally and at the surface of the targeted material. The sludge is a multiphase medium containing water, mineral and organic substances, proteins, and microorganisms cells[2]. Due to high water content, sewage sludge would absorb microwave irradiation energy with high efficiency and increase its temperature very fast. Techniques such as microwave drying, pasteurization, sterilization, pyrolysis, gasification, microwave-hydrothermal synthesis and organic compound decomposition have been investigated[11-13]. Eskicioglu et al.[10] used microwave to heat sludge up to 96°C and then adopted a batch anaerobic digestion test to evaluate the improvement of biodegradability and found that the biogas production increase of 17% over untreated sludge. As compared to conventional heat treatment, microwave resulted in a more soluble protein and volatile fatty acid. Park et al.[14] reported that microwave treated sludge could increase the methane production by 79% compared to untreated sludge. Wojciechowska[15] used microwave to heat sludge, after 180 s microwave heating specific resistance to filtration of mixed sludge (primary and waste activated sludge) and anaerobic digested sludge decreased 73% and 84%, respectively. Recent literatures reported that hydrolysis of organic materials by combing microwave with hydrogen peroxide and acid would be used to recovery sludge nutrients[16]. Therefore, the effectiveness of microwave has been recognized by researchers. However, most of previous research adopting microwave irradiation used a temperature below 100°C in an atmospheric pressure condition. The high efficiency of microwave irradiation was limited by the temperature.

In present research, a higher temperature microwave pretreatment within a pressure sealed vessels was applied to hydrolyze sludge and to enhance the biogas production in anaerobic digestion. In addition, the dewaterability and the volume reduction of sludge after pretreatment was investigated. The aims of current research were to evaluate the possibility of using high temperature microwave irradiation to pre-treat sludge for subsequent treatment and disposal.

## 2 Materials and methods

### 2.1 Sewage sludge

The sludge was collected from a municipal wastewater treatment plant of Beijing. In this plant, the primary sludge and the waste activated sludge (WAS) were mixed and transported into an intermediate storage tank. The WAS could be obtained before going to the tank. In current research, WAS and the mixture of primary sludge and WAS were sampled and used for experiment. Table 1 provides the characteristics of the WAS and mixture sludge (MS). The sampled sludge was screened in laboratory by using a 3.2×3.2 mm mesh sieve to remove undesired particles and then was stored in a refrigerator at 4°C before using.

**Table 1 Characteristics of sludge**

| | WS* | MS** |
|---|---|---|
| Solid content/% | 2.8 | 4.6 |
| Organic content/% | 70 | 69 |
| pH | 6.07 | 6.29 |

Note: * Waste activated sludge; ** Mixture of primary and waste activated sludge.

### 2.2 Microwave irradiation heating

A microwave oven (2450 MHz, 1000 W, MSD6, Shanghai Sineo Co., Ltd.) with polytetrafluoroethylene vessels was used for providing microwave irradiation energy. This frequency of microwave were widely used in lab scale experiment[17,18]. The microwave irradiation treatment was performed by adopting batch tests. Firstly, the sludge of 30 mL was subjected to a 70 mL PTFE vessel. And then, the sludge was heated to a fixed temperature for some period. Finally, the vessel was chilled in cold water to room temperature to take the sludge out for subsequent analysis. All the test samples were subjected to microwave heating with temperature of

80°C, 120°C, 150°C and 170°C. The microwave heating holding time was 1 min, 5 min, 10 min, 20 min and 30 min. The temperature and pressure of the microwave vessel were measured and controlled automatically. To evaluate the efficiency of microwave energy by sludge, sludge of 100 g, 200 g, 300 g, 500 g, 700 g and 1000 g was added in the sealed vessels, and was heated for 60 s. The temperature was measured immediately after microwave irradiation stop. The energy absorption was calculated by the following Equation (1):

$$p = m \cdot k \cdot c_p (T - T_0)/t \tag{1}$$

where, $p$ is microwave energy absorbed by sludge, J/S; $m$ is amounts of sludge subjected to microwave irradiation, g; $k$ is coefficient of energy, cal=4.2 J; $c_p$ is specific heating of heating material, cal/(g·°C); $T$ is sludge temperature after microwave irradiation, °C; $T_0$ is sludge temperature before microwave irradiation, °C; $t$ is microwave heating time, s.

### 2.3 Conventional heating

The conventional heating experiment was carried out by using 500 mL stainless cylinder reactors which were put in a hot oil batch. The temperature was set at 120°C, 150°C, and 170°C. The sludge of 300 mL was firstly put into the reactor which was then put in the hot oil oven. The reactor was pressured when the temperature increased. The temperature of the cylinder reactor was monitored by using XMTD meter.

### 2.4 Biochemical methane potential test

Biochemical methane potential (BMP) test was used to evaluate sludge anaerobic digestibility before and after microwave irradiation pretreatment. Microwave treated sludge seeded of 30 mL and 150 mL matured inoculums were mixed into a 250 mL serum bottle. There was 60 mL of untreated sludge fed as a control test. Then, the serum bottles were put in a water bath at 35°C. Serum bottle was shaken every 12 h for sufficient blend by hand. The cumulative gas production was measured using a water displacement method. Methane in biogas was measured by a gas chromatograph equipped with a thermal conductivity detector. The BMP test procedure was according to previous report[19].

### 2.5 Chemical analysis

The total COD (TCOD) was tested by the potassium dichromate/ferrous ammonium sulfate method. Sludge particles were kept uniformly suspended by a magnetic stirrer when sampling. The supernatants separated from sludge by centrifuging (LG10-2.4A) at 2775 g for 10 min were used for soluble COD (SCOD) determination. The values of TS and SS were measured by drying sludge slurry at 105°C for 24 h; volatile solid (VS) and VSS were tested by burning it at 600°C for 2 h. For SS and subsequent VSS analysis, before heating, sludge was centrifuged to remove soluble solids as described in SCOD determination.

The reduction of sludge volume after heat treatment (microwave and conventional method) was calculated according to the Equation (2).

$$\phi = 1 - \frac{S_0 \cdot (1-\alpha)}{1-W} \div \frac{S_0}{1-W_0} = 1 - \frac{(1-\alpha)\cdot(1-W_0)}{1-W} \tag{2}$$

where, $S_0$ is suspended solid content in raw sludge, %; $\alpha$ is hydrolysis ratio of suspended solid after microwave irradiation, %; $W_0$ is water content of sludge, %; $W$ is water content of microwave treated sludge, %

## 3 Results and discussion

### 3.1 Temperature increasing of sludge with microwave irradiation

Compared to conventional heating, microwave irradiation was much faster to reach a fixed temperature[15]. It has been known that loaded materials could be heated by high frequency electromagnetic waves. The heating effect arises from the interaction of the electric field component of the wave with changed particles in the material by using microwave irradiation. Energy absorbed by materials was higher as the microwave penetration depth was smaller. Because of the complicated composition of sludge, the absorption of microwave energy would be influenced by organics (such as protein, lipid and carbohydrate) and solid concentration, also by heating load. Water absorbs microwave energy by exponential relationship with the heating load, and the absorption efficiency can reach at 80%[9]. In current research, temperature of sludge increase to 120°C, 150°C and 170°C were only 4 min, 7 min, and 7.5 min, respectively. Normally, more than 30 min is necessary to increase the sludge temperature to

170°C using the conventional heating. As shown in Figure 1, the temperature increasing within 60 s can be represented by the equation of $y=-17.45\ln(x)+136.94$ with $R^2$ of 0.9743. The equation for pure water was $y=-19.548\ln(x)+153.11$ with $R^2$ of 0.9528. The results indicated that the temperature of pure water by absorbing microwave energy was slightly higher than that of sludge. Energy absorbed by both sludge and pure water started to level when the materials in microwave oven were more than 800 g as shown in Figure 2. That indicated the suitable load of heating materials and microwave energy input should be further optimized if large scale microwave reactor was applied.

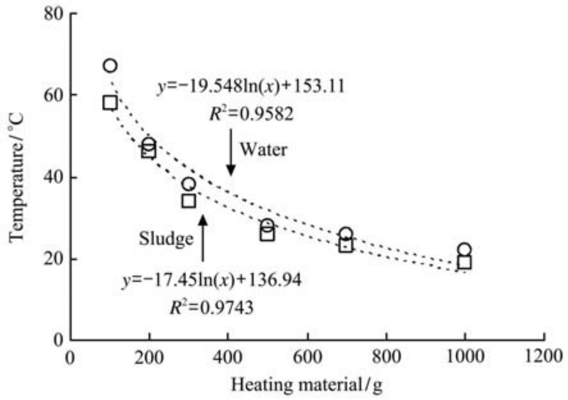



Figure 1 Temperature changes of sludge by microwave irradiation

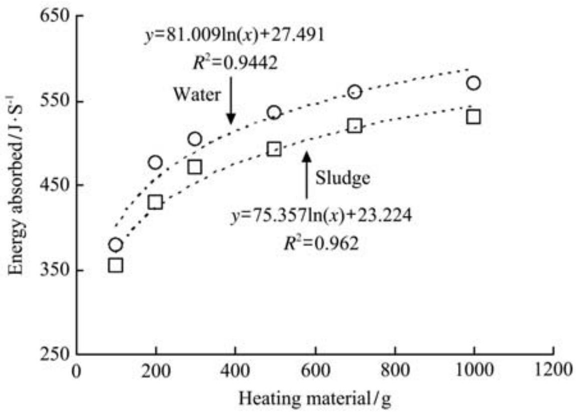


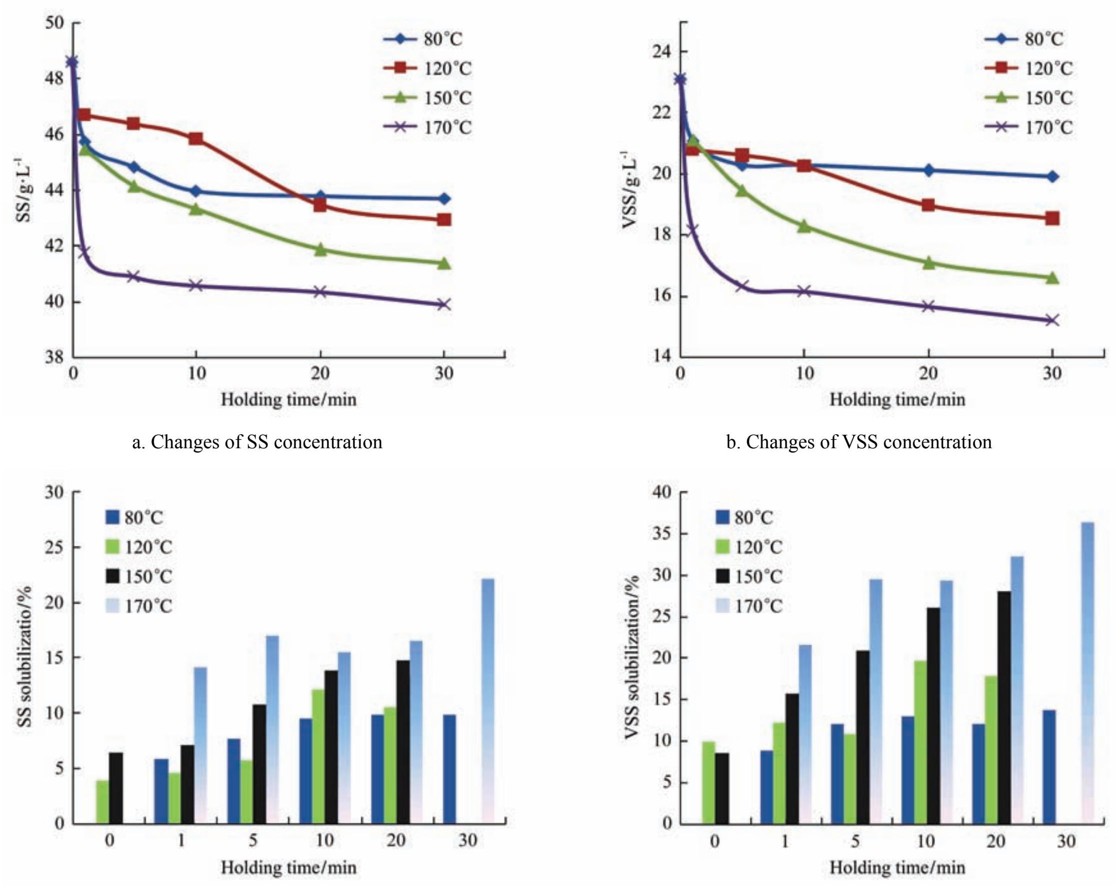

Figure 2 Microwave energy absorption

### 3.2 Hydrolysis efficiency of sludge

The conventional heat treatment, performed by Wang et al.[20], proved that the inorganics dissolved at the lower dissolution ratio compared to organic materials and the main parts of the solid dissolution originated from VSS. The solid fractions in the sludge followed the pathways of dissolution and hydrolysis. First of all, the flocs of microorganism dispersed and disintegrated under high temperature, and then the intracellular materials were released, dissolved and hydrolyzed as follows: the lipids hydrolyzed to palmitic acid, stearic acid and oleic acid; protein to a series of saturated and unsaturated acids, ammonia and some carbon dioxide; the carbohydrate to polysaccharides with a smaller molecular weight and possibly, even to simple sugars[21]. Therefore, VSS was generally taken as a principal parameter to evaluate the hydrolysis of sludge under heat treatments and VSS dissolution depicted the tendency of sludge becoming an inorganic product.

Figure 3 presents the variations of dissolution of SS and VSS at different temperatures and heating time. The holding time from 1 min to 30 min were used at the temperature of 80°C, 120°C, 150°C and 170°C. The VSS dissolution ratios substantially increased with rising the temperature and elongate the holding time. However, the increase of dissolution was not obvious when the holding time was over 5 min. That indicated the microwave irradiation fast penetrate the sludge, disrupt the microbe cells and subsequently released the internal materials. It can be concluded in Figure 3a and Figure 3c, the VSS dissolution were mainly dependent on the temperature. The highest value of VSS dissolution ratio (36.4%) was obtained at 170°C for 30 min. The VSS dissolution did not increase significantly when temperature at 80°C and 120°C. However, the dissolution increased from around 10% to 30% when temperature increased from 120°C to 170°C. The similar results can be founded in the dissolution of SS as shown in Figure 3b and Figure 3d. The dissolution of SS is slightly lower than that of VSS. The highest SS dissolution was 23.5% obtained at 170°C for 10 min and the dissolution of SS greatly influenced the reduction of sludge after microwave irradiation.

Correspondingly, the soluble COD concentration in the supernatants of microwave irradiated sludge would increase sharply. The liquid fraction would be more easily to be biodegraded in anaerobic system. As shown in Figure 3c, the dissolution of SS was much lower than that of VSS and COD because there were some inorganic materials which cannot be hydrolyzed but represented as SS.




a. Changes of SS concentration

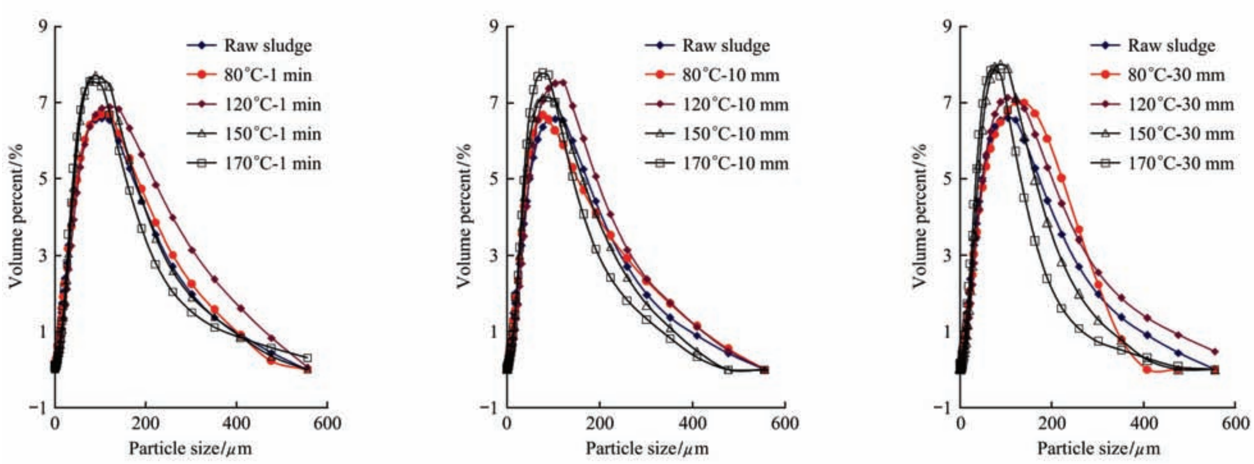
b. Changes of VSS concentration

c. SS solubilization

d. VSS solubilization

Figure 3 Dissolution efficiency of SS and VSS

As shown in Figure 4, the particle size distributions of sludge with and without microwave irradiation were compared. It was significant that the particles became smaller after microwave treatment that indirectly indicated the disruption of sludge and can prove the release of organics into the liquid fraction. The longer microwave irradiation did not give significant improvement of the particles changes. However, the temperature plays an important role comparing the results of 80°C, 120°C, 150°C and 170°C. The results could support that the microbes cells in sludge could be disrupted very fast by using high temperature microwave. Under conventional treatment by using steam heating, the heating time longer than 30 min were normally required[3]. The microwave could provide more advantages than conventional heating using a very short time.




a. Particle size changes by heating 1 min

b. Particle size changes by heating 10 min

c. Particle size changes by heating 30 min

Figure 4 Particle size distributions after microwave irradiation

The hydrolysis efficiency of elements (carbon, hydrogen and nitrogen) in sludge was provided in Figure 5. It can be founded that carbon was difficult to be hydrolyzed (around of 30%) than hydrogen and nitrogen and did not show significant increment with the increasing of temperature. On the other hand, hydrogen and nitrogen were much easier to be hydrolyzed and did not increase with temperature as well. There was more than 60% of nitrogen that was hydrolyzed into liquid fraction under temperature of 80°C-170°C. The hydrogen solubilization efficiency was around 45%. That indicated the protein which contains much nitrogen and it was readily hydrolyzed with microwave irradiation. Theoretically, the higher carbon in materials would result in a higher heat value and would facilitate the incineration of the solid phase sludge after dewatering the microwave treated sludge. In addition, the low nitrogen would provide advantages of reducing $NO_x$ in the combustion fuel gas and the nitrogen released into liquid phase would be further removed by biological process or recovered by physical process as fertilizer. At this point, the microwave pretreatment provides many opportunities for high efficient treatment of sludge.

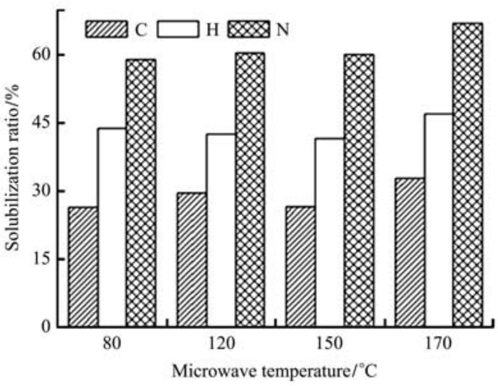



Figure 5 Solubilization of carbon, hydrogen and nitrogen

### 3.3 Effects of sludge concentration

The wastewater treatment plant normally adopt thickening and dewatering process to remove undesired water as much as possible from sludge to reduce the volume and facilities the following treatment like composting and incineration. The higher concentration of solid in sludge would benefit the overall treatment process efficiency. In Figure 6, the SCOD concentration was provided using sludge concentrated at 7%, 9% and 13% at temperature of 170°C. It was obvious that the soluble COD concentration increased proportionally by increasing the sludge concentration. However, the high concentration would negatively affect the heat transfer and microwave energy absorption in the reactor. It can be seen from Figure 6 that, the SCOD concentration gradually increased from 52 g/L at 1 min to 69 g/L at 20 min with the prolonged heating time. As shown in Figure 7, the ammonium was released into liquid fraction depending on the sludge concentration. The ammonium concentration reached to 1.6 g/L using sludge at TS of 13%. A slight decreasing of ammonium with the heating time using sludge of 13% concentration was observed. The possible reason was the volatilization of $NH_3$ during cooling. There was no significant increment comparing the ammonium concentration by using sludge at TS of 7% and 9%. The high ammonium would negatively affect the activity of methanogenes in anaerobic digestion process. In current research, the ammonium which was below 2 g/L would not inhibit the biogas production in anaerobic digestion process. The optimum of sludge concentration in heat treatment should be optimized for the ammonium effects in subsequent anaerobic process.

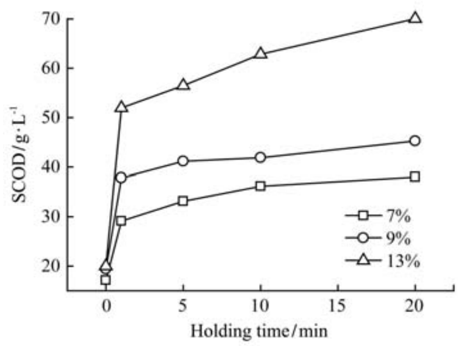



Figure 6 SCOD concentrations of different sludge after microwave irradiation

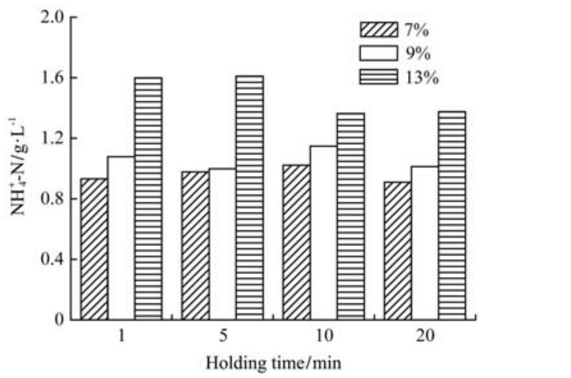
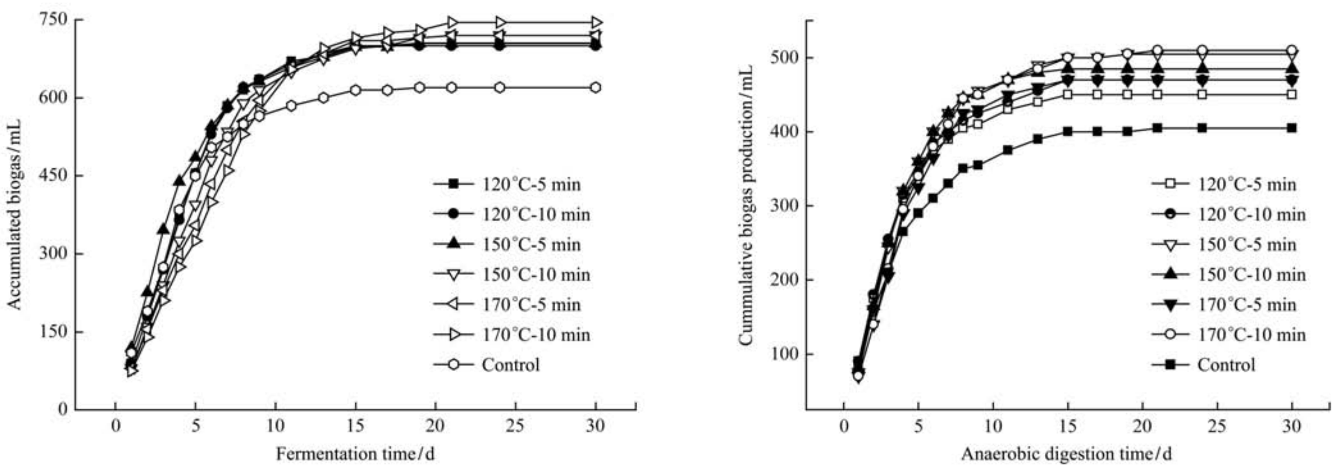



Figure 7 Ammonium concentrations from different concentration sludge after microwave heating

### 3.4 Biodegradability improvement after microwave irradiation

Pino-Jelcic et al.[22] compared microwave irradiation with conventional heating at 60°C-65°C, and found that VS removal ratio of microwave thermal treated sludge anaerobic digestion was 53.9%, as while as 51.3% in conventional thermal treated sludge anaerobic digestion. It showed that microwave was helpful to destroy sludge bacterial cell membrane, destruct more E. coli, and release more intracellular materials which were represented as COD in liquid fraction. Eskicioglu et al.[10] found a higher biogas production from sludge treated by microwave irradiation than that from the samples treated by conventional heating. The BMP test was often performed to evaluate the anaerobic digestibility of various pretreated sludge. Hydrolysis test showed that the VSS dissolution did not increase remarkably with the elongated time further after 5 min, in the same time, VSS dissolution was low at 80°C. Thermal treated sludge used for BMP test was heated at the temperature of 120°C, 150°C and 170°C for 5 min and 10 min. In order to analyze microwave effect on different type sludge, both MS from and WAS were tested. After microwave treatment, total biogas production increased by 12.9% to 20.2% over control after 30 d digestion for mixture sludge. The total biogas production of waste activated sludge was 11.1% to 25.9% higher than that of untreated sludge. The highest biogas production for both mixture sludge and waste activated sludge was obtained from the sludge treated by microwave at 170°C for 10 min. However, as introduced before, the VSS and COD dissolution were greatly increased by using higher temperature. It indicated that there were some refractory materials generated which was not easily converted into methane. The WAS after pretreatment provided lower biogas production in Figure 8b comparing to the MS. In addition, the temperature effects were not significant as well. It was expected that microwave heating as a pretreatment method for MS and WAS would be effective to obtain higher biogas production.




a. Mixture sludge

b. Wastewater activated sludge

Figure 8 Biogas production from sludge after microwave irradiation

Both of the two batches of BMP gas productions were increasing fast for the first 10 d. Then the gas production ratio slowed down until the total gas production did not change. Biogas yield from MS was higher than that of WAS. Normally, MS contain more easily degradable organics than WAS which was mainly composed of bacterial cells. It indicated that the microwave thermal pretreatment improved the sludge anaerobic digestibility for both MS and WAS.

The digesters treating high Mw (4300 kDa) materials resulted in smaller biodegradation rate constants (*k*), indicating that microorganisms require a longer time to utilize high Mw fractions which are most likely cell wall fragments and polymers[10]. As listed in Table 2, microwave irradiation pretreatment produce less high Mw (>10 000) materials in both COD (4.63 g/L) and TOC (1.6 g/L) than conventional heating. That indicated the microwave irradiation would produce more readily biodegradable components which can be converted into methane in anaerobic digestion system.

**Table 2 Liquid fraction concentration after microwave irradiation**

| | | Fraction divided by molecular weight* | | | |
|---|---|---|---|---|---|
| | | >10 000 | 5000-10 000 | 1000-5000 | <1000 |
| Microwave heating | COD/g·L$^{-1}$ | 4.36 | 1.58 | 2.97 | 7.29 |
| | TOC/g·L$^{-1}$ | 1.6 | 0.8 | 0.3 | 3.9 |
| Convention heating | COD/g·L$^{-1}$ | 5.54 | 0.6 | 4.2 | 10.1 |
| | TOC/g·L$^{-1}$ | 2.2 | 0.4 | 0.7 | 5.5 |

Note: * the unit of molecular weight was g/mol.

### 3.5 Sludge volume reduction after microwave irradiation

By combing the thermal pretreatment (170°C/40 min) and anaerobic digestion, the reduction in the final volume of sludge is 65% as TS[23]. Tian et al.[24] found that the microwave irradiation effectively changed the structure of sludge and enhanced the dewaterability. As introduced in Table 3, the moisture content of dewatered sludge was greatly reduced after heating by both centrifuging and belt dewatering.

**Table 3 Water contents of sludge with and without microwave irradiation**

| | Raw sludge | 80°C | 120°C | 150°C | 170°C |
|---|---|---|---|---|---|
| Centrifuging/% | 84±1 | 68±3 | 68±4 | 65±2 | 60±2 |
| Belt dewatering/% | 83±2 | 66±2 | 72±2 | 64±3 | 57±1 |

The raw sludge had a water content of 84% but decreased to 60% and 57% after microwave irradiation, centrifuging and belt dewatering separately. Anderson et al.[25] stated that the physical structure of sludge was irreversibly altered by a thermal pretreatment over 150°C and then the dewaterability of sludge was significantly enhanced. Higher temperature resulted in lower moisture content. The sludge volume reduction originated from the hydrolysis of solid and the reduction of moisture content. The moisture content of sludge after microwave irradiation at 80°C, 120°C, 150°C and 170°C were 76.8%, 80.6%, 73.1% and 65.5%, respectively, using centrifuging at 2775 g. As shown in Figure 3, the solid in sludge (both SS and VSS) was significantly hydrolyzed into liquid fraction. According to the calculation in Equation (2), the sludge volume reduction compared to dewatered raw sludge was calculated. There were 21.3%, 5.7%, 33.9% and 60% sludge volume reduced by microwave irradiation.

## 4 Conclusions

Microwave irradiation was effective to pretreat sludge by using short time and providing comparable efficiency compared to conventional heating process. Solid materials (SS and VSS) in sludge could be dissolved with microwave irradiation and the increasing of the SCOD concentration in liquid phase was obviously founded. The improvement of sludge biodegradability was proved for both mixture sludge and for waste activated sludge by providing higher biogas production. The improved dewaterability and the dissolution of SS contribute to a reduction of sludge volume up to 60%.

## Acknowledgement

This work was finically supported by the projects of National Key Technology R&D Program of China (2014BAC24B01) and Environmental Development Center of Ministry of Environmental Protection (ZZ-2016-01).

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