



## Effects of thermal hydrolysis on organic matter solubilization and anaerobic digestion of high solid sludge



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### HIGHLIGHTS

- THP positively affected the organics solubilization of high-solid sludge.
- THP of high solid sludge greatly determined the performance of anaerobic digestion.
- HTHP significantly sped up the gas production rate and increased the total gas volume.
- The SRT could be reduced after the thermal hydrolysis at 140–160 °C.

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### ABSTRACT

Anaerobic digestion of sludge not only decreases the mass/volume of sludge, but also increases the production of energy-rich biogas. The effects of a thermal hydrolysis process (THP) on the solubilization of main organics of sludge, as well as the performance of the followed biochemical methane potential (BMP) tests under mesophilic condition (35 °C), were systematically evaluated. Organic compounds such as proteins and carbohydrates which have relatively high yield coefficients are solubilized efficiently during the THP. Good linear relations were obtained between the amounts of soluble chemical oxygen demand and soluble proteins/carbohydrates. The viscosity of sludge dramatically reduced after the THP, particularly at higher temperatures. In contrast to the treatments by low-temperature THP (60–90 °C), the treatments by high-temperature THP (120–160 °C) accelerated the digestion rate and increased the biogas yield in the following BMP tests. Meanwhile, the solid retention time could be reduced from 18–20 days to 12–14 days when high solid samples were pre-treated under THP at 140–160 °C based on the corresponding results of acceleration of methane production after the treatment. As a result, this study elucidated the appropriate operational conditions for high solid sludge (TS > 10%) under THP, and their mechanisms of the acceleration of biogas production.

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### 1. Introduction

Anaerobic digestion is applied popularly to sewage sludge stabilization. It has many advantages such as reduced mass/volume of the sludge, production of energy-rich biogas, and improved sludge dewaterability [1]. Moreover, compared to aerobic digestion, anaerobic digestion costs less, and need smaller environmental footprints [2]. Anaerobic digestion includes four major steps: hydrolysis, acidogenesis, acetogenesis, and

methanogenesis. In the first step, hydrolysis, both the solubilization of particulate matter and biological decomposition of organic polymers to monomers/dimers occur slowly, thus making it the rate-limiting step of the overall processes [12].

When it comes to the hydrolysis of sludge integrations, the release of intracellular and extracellular matters becomes the most important. Thermal pretreatment processes thus have been studied to promote sludge hydrolysis by destroying the integrations, which results in the reduction of solid retention time in digestion process by finally accelerating methane production. In addition, the sludge after enhanced hygienization of thermal processes can not only comply with the European Union (EU) policy on the elimination of pathogens [10], but also reach class A bio-solid standard of the US Environmental Protection Agency (EPA) [15], making it more suitable for subsequent land application.

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Since the pretreatment under high temperature (>100 °C) and high pressure (10 M) consumes more energy and requires strictly for the equipments, pretreatment under low temperature for organic matters has attracted much attention recently. Wang et al. [16] tested the effectiveness of low-temperature pretreatment (60–100 °C) on the mesophilic anaerobic digestion of sludge and found that it significantly increased (30–52%) the methane production. The generation rate of methane production sped up more after the pretreatment at 60 °C compared to that observed for both at 80 °C and 100 °C. However, the thermal pretreatment time adopted for all the experiments in the study was only 30 min; the superiority of higher temperature might not have been well elucidated. Climent et al. [6] reported that the optimal pretreatment for secondary sludge with 70 °C was for 9 h rather than for 24 h, 48 h, and 72 h. Meanwhile, different raw sludge and/or different pretreatment conditions might lead to inconsistent or even contrary results [16,4]. And most studies were carried out either at one temperature for different pretreatment times or over a narrow temperature range for a determined pretreatment time, and sometimes the results also led to the contrary conclusions, it is necessary to conduct a systematical analysis of thermal hydrolysis over a broader range of temperature and pretreatment time using same sludge, thereby to comprehensively elaborate the effects of THP on organic matter solubilization of sludge and the performance of the following anaerobic digestion.

Once the temperature exceeds 200 °C during the thermal-pressurized pretreatment, the Maillard reactions will happen, by which melanoidins will be produced in the reactions between reducing sugars and amino acids. However, melanoidins are high-molecular-weight heterogeneous polymers that are difficult to degrade or even inhibit the degradation of other organics [9]. Abe et al. [1] reported that biogas production by anaerobic digestion of sludge with thermal pretreatment at 200 °C decreased by 33% in comparison to the gas yield from the digestion of sludge with thermal pretreatment at 170 °C, and even less than the control in which the fed sludge was not pretreated.

Moreover, the concentrations of total solids (TS) in sludge in the previous studies were mainly 3–10%, and dewatered sludge with TS ≈ 20% had been rarely studied. In comparison to low solid anaerobic digestion, high solid anaerobic digestion (TS > 10%) is more attractive because of the relatively smaller reactor volumes, lower energy requirements for heating, less material handling, etc [13]. Nowadays, centralized processing of collected dewatered sludge becomes promising to some small-scale wastewater treatment plants (WWTPs) especially in the developing countries such as China [8,9]. Although high solid anaerobic digestion of dewatered sludge without pretreatment (feeding TS, 20%) was feasible in the laboratory-scale reactors [8], the rheological behavior of sludge, which is crucial for the design of anaerobic digestion plants, could not be reflected from laboratory-scale experiments because of the scale limitation. The Cambi thermal hydrolysis process (THP) has been applied to several full-scale projects (165–180 °C, 10–30 min, TS = 15–20%) [14], in which the sludge with 12% TS after THP can be handled in the same way as raw sludge with 5–6% TS.

At present, there are still lack of systematical evaluations on the solubilization of organics (soluble proteins, carbohydrates, and volatile fatty acids) and variations in viscosity of high solid sludge after different operational conditions for THP. In this study, the difference of those soluble substances after THP were compared, and further the biogas production of those pretreated sludge in mesophilic batch experiments after THP were checked respectively, by which the appropriate operational conditions for THP and their mechanisms of the acceleration of biogas production were elucidated as the reference for practical applications.

## 2. Materials and methods

### 2.1. Sludge characteristics

The dewatered/high solid sludge samples were obtained from a municipal WWTP in Shanghai, China. The samples were stored at 4 °C in laboratory before the experiments. The main characteristics of the sludge used in the experiments are listed in Table 1.

### 2.2. Pretreatment conditions

The effects of thermal pretreatment depend on treatment temperature and time. In this study, low-temperature thermal hydrolysis process (LTHP: 60 °C, 70 °C, 80 °C, and 90 °C) with different duration time (1 h, 2 h, 4 h, 8 h, 12 h, 24 h, 36 h, 48 h, 60 h, and 72 h) and high-temperature thermal hydrolysis process (HTHP: 120 °C, 140 °C, 160 °C, and 180 °C) with different duration time (15 min, 30 min, 45 min, 60 min, 75 min, 90 min, 105 min, 120 min, 150 min, and 180 min) were carried out to study the comprehensive effects.

In the LTHP, four beakers, each containing 500 g of sludge, were heated in water baths at the different controlled temperatures of 60, 70, 80, and 90 °C for 3 days, respectively. The beakers were covered with lids to prevent water evaporation and magnetically stirred to ensure temperature homogeneity. Around 10 g samples each were taken from the beakers for the measurements at the determined time.

In the HTHP, corresponding to the sampling time, each group contained 10 sealed 100 ml Teflon jars with 70 g sludge (30% safety volume guarantee). The different groups of jars were heated in an oven at the different temperatures (120, 140, 160, and 180 °C, respectively) and taken out one by one at different sampling time. A thermocouple attached to the surface of the jar and a thermode-tector were used to measure the surface temperature of the jars, which was approximately as same as the temperature of the sludge inside the jar.

The raw and pretreated sludge samples were analyzed for TS, VS, viscosity, SCOD, VFAs, proteins, carbohydrates, NH<sub>4</sub>-N, and pH.

### 2.3. Biodegradability batch tests

The waste activated sludge (WAS) was thermally treated at different temperatures for 24 h (low temperature) or 3 h (high temperature) before mixing with the seed sludge. The seed sludge was pre-incubated to remove its residual biodegradable organics. The pre-incubation was carried out at 35 °C in a water bath for 2 days. The inoculum-to-substrate ratio was set to 2:1 VS (a mixture of 300 g seed sludge and 45 g pretreated WAS). The methane production from only the inoculum was measured, which was subtracted from the total methane production. The main equipment for BMP test was a 500 mL flask incubated at 37 °C in a shaking water bath with 120 rpm. The headspace of each flask was flushed with nitrogen gas for 1 min and immediately sealed using butyl

**Table 1**  
Characteristics of the dewatered sludge.

Parameter	Value
TS (% w/w)	16.7 ± 0.5
VS/TS	70.5 ± 0.1
TCOD (g/L)	166.0 ± 2.3
SCOD (g/L)	7.55 ± 0.5
Dissolved VFAs (g/L)	1.45 ± 0.1
Dissolved NH <sub>4</sub> -N (g/L)	1.10 ± 0.1

TS, total solids; VS, volatile solids; TCOD, total chemical oxygen demand; SCOD, soluble chemical oxygen demand; VFAs, volatile fatty acids.

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