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Hydrothermal and alkaline hydrothermal pretreatments plus anaerobic digestion of sewage sludge for dewatering and biogas production: Bench-scale research and pilot-scale verification



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ABSTRACT

To test the feasibility and practicability of the process combing hydrothermal pretreatment for dewatering with biogas production for full utilization of sewage sludge, hydrothermal/alkaline hydrothermal pretreatments and in turn anaerobic digestion of the filtrates obtained after dewatering the pretreated sludge were performed at bench- and pilot-scales. The hydrothermal temperature fell within the range of 140 °C-220 °C and the pretreatment time varied from 30 min to 120 min. For the alkaline hydrothermal pretreatment the pH value of the sludge was adjusted to 9.0–11.0 by adding Ca(OH)₂. The results showed that the dewaterability of the sewage sludge was improved with increasing pretreatment temperature but the impact of the pretreatment time was not significant. The addition of Ca(OH)₂ gave better performance on the subsequent mechanical dewatering of the pretreated sludge compared to pure hydrothermal pretreatment, and the higher the pH value was, the better the dewaterability of the pretreated sludge was. The conditions of 180 °C/30 min and 160 °C/60 min/pH = 10.0 (for hydrothermal and alkaline hydrothermal pretreatments, respectively) resulted in relatively good results in the theoretical energy balance, which were verified in the pilot-scale tests. Based on the data from the pilot tests, the alkaline hydrothermal process realized self-sufficiency in energy at the cost of a proper amount of CaO.

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1. Introduction

The treatment/disposal of increasingly huge amounts of sewage sludge has become an impending problem in China. Technically, reducing the volume of sewage sludge is the first step, but this has to encounter great difficulties in sewage sludge dewatering due to its special properties including hydrophilic nature and structural stability (the biomass having supra-colloidal structures with cells as the nucleus) (Mikkelsen, 2003). However, unless being effectively dewatered in advance, the final disposal of sludge (by such methods as incineration and land-filling) is either impractical or costs too much. Therefore, it is crucial to explore economical methods to improve the dewaterability of sludge.

Many pretreatment approaches, such as thermal/hydrothermal treatment (Neyens and Baeyens, 2003), ultrasonic treatment (Ruiz-Hernando et al., 2010), alkaline treatment (Ruiz-Hernando et al., 2015) and their combinations have been studied to facilitate sludge dewatering. The principle of these methods for improving sludge dewaterability is to change the "bound" water in the sludge into free water (Hii et al., 2014). Among the above methods, hydrothermal treatment has been widely used to cope with all types of water-rich bio-wastes including various sludges (Nevens and Baeyens, 2003), antibiotic residues (Zhang et al., 2014) and the like. For hydrothermal processing, high pressure steam is employed because of its various virtues (Akiya and Savage, 2002). During hydrothermal processing, the extracellular polymeric substances (EPSs) are disintegrated/decomposed to release the bound water

Abbreviations: EPS, extracellular polymeric substances; COD, chemical oxygen demand; TS, total solid; VS, volatile solid; SS, suspended solid; UASB, up-flow anaerobic sludge blanket; HRT, hydraulic retention time; SRT, sludge retention time; BMP, biochemical methane potential.

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and the walls of the cells are destroyed to spill the intracellular water, which greatly improve the dewaterability of the above sludge-like biowastes as well as their biodegradability (Neyens et al., 2004). Furthermore, the presence of alkalis in hydrothermal processing has been found to have additional effects on facilitating sludge dewatering and biogas production (Guan et al., 2012). Also, bivalent/trivalent cations (such as Ca^{2+}) were found to exhibit positive effects on the dewaterability of sludge (Higgins and Novak, 1997; Neyens et al., 2003) by various ways, such as forming hydroxybases or changing such the sludge properties as surface charge, viscosity and floc strength (Pevere et al., 2007).

In this study, bench-scale experiments were initially employed to investigate the impacts of hydrothermal and alkaline hydrothermal pretreatments on the sewage sludge dewatering and also the biogas production from the filtrate (obtained after mechanically dewatering the pretreated sewage sludge) by anaerobic digestion, and then the results were compared to optimize the hydrothermal conditions. Finally, pilot-scale tests at the optimized conditions were conducted to evaluate the processes of hydrothermal/alkaline hydrothermal pretreatments plus anaerobic digestion for full resource reuse of sewage sludge in terms of the material and energy balances.

2. Materials and methods

2.1. Material and analysis

The sludge used in this work was obtained from a municipal sewage treatment plant in Xiamen, China and stored at 4 $^{\circ}$ C in a refrigerator for further tests. China standard methods (GB/T 5009-2003) were employed to analyze the raw and pretreated sludge including parameters such as COD, TS, VS and SS. The properties of the raw sludge including its proximate and ultimate analysis results are presented in Table 1.

A pH meter (Mettler Toledo FE20) and a portable rapid 3-N (NH₄⁺-N, NO₂⁻-N, NO₃⁻-N) detection instrument (Shenzhen Sinsche Technology, S-3N analyser) were used to measure the pH value and the ammonia nitrogen concentration of the raw and pretreated sludge samples, respectively. A zeta meter (Nano-ZS, size range: ~2000 μ m) and a laser diffraction particle analyzer (LS 13320) (capable size range: ~10000 nm) were adopted to measure the size distribution of the insoluble particles in the liquid obtained after (alkaline) hydrothermal pretreatment and the soluble particles in its resulting filtrate solution (after passing through a filter membrane with pore size of 0.45 μ m), respectively. The zeta potential of the filtrate from the raw and pretreated sludges was determined

Table 1

Key parameters of the solid and filtrate liquid samples of the raw and hydrothermally pretreated sludge.

using a Malvern zetameter Nano ZS (Malvern Instruments Ltd., UK) after adjusting their pH values to 7.0 \pm 0.2. The viscosity was measured by a Brookfield Viscometer (model LVF). The methane content of the biogas was tested with the Agilent Micro 3000 - GC with a thermal conductivity detector (TCD) using argon as the carrier gas and the temperatures for the injector and detector were 100 °C and 150 °C, respectively. The solid content was determined according to the following two methods: 1) centrifugation at a separation factor of 8000 for 15 min; and 2) filter-pressing at 1.60 MPa for 3 h using a bench-scale filter-press.

To ensure the data accuracy, all of the measurements were repeated three times, and their average values were adopted for data analysis.

2.2. Bench-scale hydrothermal pretreatment and anaerobic digestion

2.2.1. Hydrothermal pretreatment

All bench-scale hydrothermal pretreatment experiments with sewage sludge were completed in an electrically heated autoclave having an inner volume of 2.0 L. Each hydrothermal test was started with loading 1000 g of wet sewage sludge (water content of 85 wt %) and 200 g of deionized water into the reactor (the water addition ratio being 20 wt%). For the alkaline hydrothermal tests, Ca(OH)₂ was added at the same time to adjust the pH of the wet sewage sludge to a desired value (the corresponding average dosage of $Ca(OH)_2$ for per gram dry sludge was 0.051 g for pH = 9.0, 0.072 g for pH = 10.0, 0.090 g for pH = 10.5 and 0.105 g for pH = 11.0, respectively). After the autoclave was sealed, the hydrothermal pretreatment was implemented as the followings: heating the loaded material to a preset temperature with heating rate of 5 °C/ min; maintaining the temperature for a desired time; terminating the heating while starting to cool the autoclave to room temperature by an electric fan in 1 h; reducing the pressure of the equipment to atmosphere pressure; and opening the autoclave to collect the products. During (alkaline) hydrothermal pretreatment, stirring at 220 \pm 2 RPM was continuously run to avoid any serious temperature gradient inside the reactor. The pretreated sludge was collected in a glass bottle and kept at 4 °C in a refrigerator for further analysis. The pretreatment temperature varied from 140 to 200 °C, and the pretreatment time ranged from 30 to 120 min. Additional information about the equipment and the typical procedures for the hydrothermal test can be found elsewhere (Li et al., 2015).

Different conditions	Proximate analysis (wt %, air dried)			Ultimate analysis (wt%, air dried)					NH ₄ ⁺ -N (mg/L)	рН		COD (mg/L)
	Ash	VM	FC	С	Н	Ν	S	0		BPT ^a	APT ^b	
Raw material	44.71	48.23	7.07	24.67	4.65	4.51	0.95	20.52	320 ± 10	7.23 ± 0.02		270 ± 5
140 °C, 30 min	51.73	40.95	7.32	22.04	4.18	3.75	0.82	17.48	1130 ± 31	7.23 ± 0.02	6.53 ± 0.03	34149 ± 57
160 °C, 30 min	58.11	35.56	6.33	20.87	3.87	3.19	0.88	13.09	1513 ± 23	7.23 ± 0.02	6.32 ± 0.02	44552 ± 43
160 °C, 60 min	57.37	37.59	5.04	20.65	3.69	3.10	0.82	13.40	1520 ± 15	7.23 ± 0.02	6.08 ± 0.00	45157 ± 19
160 °C, 60 min, pH = 9.0	60.56	34.34	5.10	19.69	3.33	2.66	0.76	13.00	1529 ± 10	9.00 ± 0.04	7.78 ± 0.04	45500 ± 53
160 °C, 60 min, pH = 10.0	62.69	32.31	5.00	18.64	3.18	2.40	0.72	12.37	1595 ± 12	10.00 ± 0.03	9.02 ± 0.02	50800 ± 25
160 °C, 60 min, pH = 10.5	64.9	30.14	4.96	17.73	2.84	2.20	0.70	11.63	1780 ± 14	10.50 ± 0.01	9.89 ± 0.04	55350 ± 20
160 °C, 60 min, pH = 11.0	66.7	28.41	4.89	16.63	2.78	1.88	0.71	11.30	1820 ± 31	11.00 ± 0.05	10.43 ± 0.01	60500 ± 30
180 °C, 30 min	60.56	33.58	5.86	20.34	3.65	2.84	0.86	11.75	1687 ± 17	7.23 ± 0.02	6.01 ± 0.02	46004 ± 24
200 °C, 30 min	62.19	31.82	6.00	20.17	3.44	2.52	0.89	10.79	2050 ± 29	7.23 ± 0.02	5.74 ± 0.04	48545 ± 51
220 °C, 30 min	65.41	27.95	6.64	18.77	3.08	2.20	0.86	9.68	2615 ± 18	7.23 ± 0.02	6.47 ± 0.03	50238 ± 23

^a Before pretreatment.

^b After pretreatment.

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