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Effective Excess Sludge Disintegration Increasing by Combined HPI and LTTH

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Abstract

Excess sludge, one of the byproducts of active sludge process which contains organic matters and hazardous substances, has serious environmental pollution risks without treatment. High pressure impingement (HPI) and low temperature thermal hydrolysis (LTTH) could disintegrate sludge effectively. In this study, HPI, LTTH, and combined method of HPI and LTTH were explored to study the disintegrated mechanism of different methods. HPI showed optimal results under 12 min, 14 MPa, 7 cm and 3000 mg·L-1. LTTH showed optimal results under 60 min, 60~65 ℃ and 6000~10000 mg・L-1. Combined method with the order of LTTH first then HPI had significant disintegrated effects. Transmission electron microscope (TEM) was used to observe the disintegrated sludge structure. The results showed that LTTH could help to dissolve extracellular polymers (EPS) in the sludge and HPI could disintegrate sludge cells significantly. LTTH first could enhance the effects of HPI and take less time than single method (HPI or LTTH) to achieve the similar disintegrated effects. LTTH combined HPI has complementary mechanism, which provides a new insight of sludge disintegration.

Keywords: Excess Sludge; Sludge Disintegration; Impingement; Thermal Hydrolysis.

1. Introduction

Activated sludge process (ASP) is widely used in waste water treatment in the world, which will discharge a lot of excess sludge. Excess sludge is rich in organic matters and hazardous substances[1], which will cause serious secondary pollution without properly treatment. At present, the most widely used sludge treatment methods are sanitary landfill and incineration, which can’t remove the organic matters and hazardous substances completely. Sludge disintegration is helpful for recycling organic matters,[2,3] dissolving hazardous substances,[4] and can bring economic effects,[5] which is a promising method of sludge treatment.

Methods of sludge disintegration include physical method, chemical method, biological method and combined method. Physical methods include ultrasonic,[6] microwave,[7] impingement,[8] etc. Most chemical methods are accomplished by adding alkali (NaOH for example) or strengthening oxidant oxidation.[9,10] Biological methods include biological digestion,[11] anaerobic fermentation,[12] etc. The combined methods can effectively utilize the advantages of single disintegration methods, strengthen sludge disintegration, and achieve a better effect.

The main components of excess sludge are various types of bacteria and microorganisms, which are gathered by peptide chains glycan with strands crosslinked.[13,14] In disintegration process, extracellular

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polymers (EPS) and cell walls are obstacle of releasing intracellular substances.[13] Different methods have diverse effects on disintegration of those structures. Low temperature thermal hydrolysis (LTTH) can release EPS and SCOD,[4] provided ligands for copper and cadmium complexation,[15] yield methane and dewater the sludge efficiently.[16] Wilson and Novak found that soluble biopolymers (i.e. proteins and polysaccharides) were strongly affected by thermal hydrolysis at 150 °C and above.[17] Gao et al. found that it was effective in the releasing of SCOD, protein and carbohydrate in EPS when the dosage of alkyl polyglucose was below 0.1 g/g TSS with thermophilic bacteria in 65 °C.[18] Besides, high pressure impingement (HPI) can significantly strengthen the heat and mass transfer process between solid-liquid phase,[19] resulting in interactions of strong collision, extrusion and shearing. Xie L et al. proposed that high pressure jet device had multiple effects on bacterial cells disruption synergistically.[20] Theoretically, the combination of LTTH and HPI may be complementary in disintegration mechanism (LTTH disintegrates EPS and HPI disintegrates cells), which helps to strengthen the effect of sludge disintegration.

In this study, LTTH and HPI were explored respectively in univariate experiments to determine the optimal range of factors. The order of combined method was explored and the structure of disintegrated sludge was observed by Transmission electron microscope (TEM) to explain the mechanism of LTTH, HPI and combined method. This study provides a new sludge disintegration method since the combination of LTTH and HPI can hardly be found in the scientific literatures.

2. Method

2.1. Material

Excess sludge was from the secondary sedimentation tank, Shenshuiwan wastewater treatment plant, Shenyang, China. The sludge concentration, cumulative distribution up to 50% particle size (D_{50}), the soluble chemical oxygen demand (SCOD), Deoxyribonucleic acid (DAN), peptidoglycan (PGN), and protein of raw sludge were 8200±150 mg·L^{-1}, 139±24 μm, 270±50 mg·L^{-1}, 16±4 μg·L^{-1}, 36±6 mg·L^{-1}, and 72±12 mg·L^{-1}, respectively.

2.2. Experimental method

In this study, the optimal parameter ranges of LTTH and HPI were explored by univariate pre-experiments with analyzation of D_{50}, SCOD, DNA, PGN and protein, which were used in the combined treatment. The microstructure of disintegrated sludge was observed by Transmission Electron Microscope (TEM). Combined method was accomplished by changing the order of LTTH and HPI (completing one method and then conducting the other).

In HPI, 5L sludge was inhaled by the high pressure machine and ejected through the nozzle from the reflux drum (Fig.1). After the impingement in the enclosed space, it was collected by the reflux drum and mixed with the raw sludge. In LTTH, 5L sludge was heated by a temperature control heater with power of 1 kW. The heating time was 5-10 min according to different se temperature and timing was calculated at the start of heating. The temperature fluctuation range is ±1 °C, heater was on when temperature was lower than the limit and off when its higher than the limit. Mechanical agitation was continuously applied to uniform temperature field.

The sludge after HPI treatment could be used in LTTH directly, but it needed to be cool down when LTTH was in advance. Because sludge in high temperature might cause damage to high pressure machine.

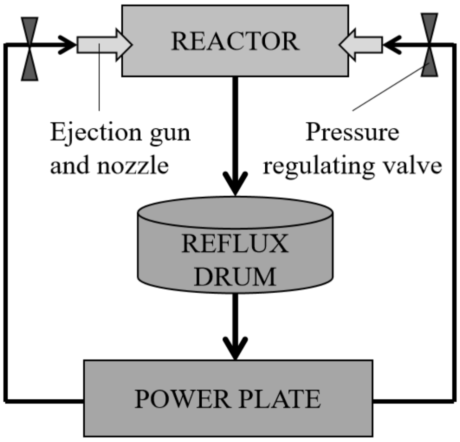

Fig. 1 Experiment system diagram

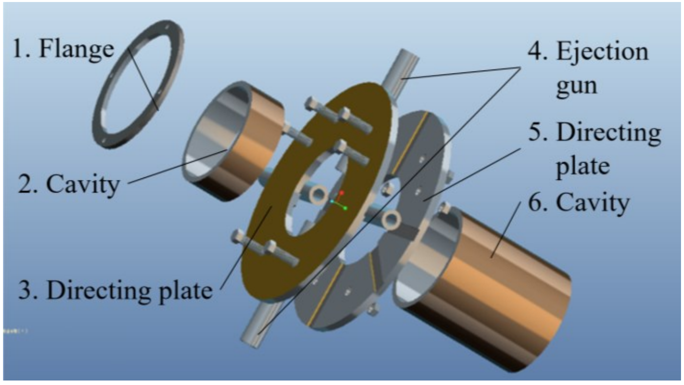

Fig. 2 Exploded view of the reactor

2.3. Equipment

2.3.1. Reactor

A pressure adjustable (1-25 MPa) high pressure machine (LF6000, Sichuan Lifeng) was used as power device. Rod ejection gun was used to facilitate the impingement in the reactor. Rod ejection gun was connected with high pressure machine by quick-change tube and the other side was connected with self-excited nozzle.

The reactor was designed by tutors and master students in the laboratory, with material of hardened and tempered steel, all surfaces were painted with antirust paint. As shown in Fig. 2, flange 1 was connected to the acrylic cover to observe the process inside the reactor. Cavity 2 and 6 constituted the reactor cavity. Directing plate 3 and 5 had the same structure and size, the same bolt holes and strip grooves, which were corn components. The strip groove was used to position and direct the rod ejection gun.

All the components were connected by welding or bolting. To prevent the sludge splashing out of the reactor from the gap between two directing plates, a tube-shaped seal ring was added in the reactor which had groove structure matching the rod ejection gun. The regulating valve could change the pressure in experiments. The rod ejection gun could be moved radially along the directing plates to change the distance between the nozzles.

2.3.2. Nozzle

Different nozzle structure will significantly affect the characteristics of the sludge jet, the degree of self-excited oscillation of the jet, and further affect the sludge cracking effect.[22] In this study, a self-excited nozzle was designed by tutors and master students in the laboratory, which was composed by two components, shown as Fig. 3.

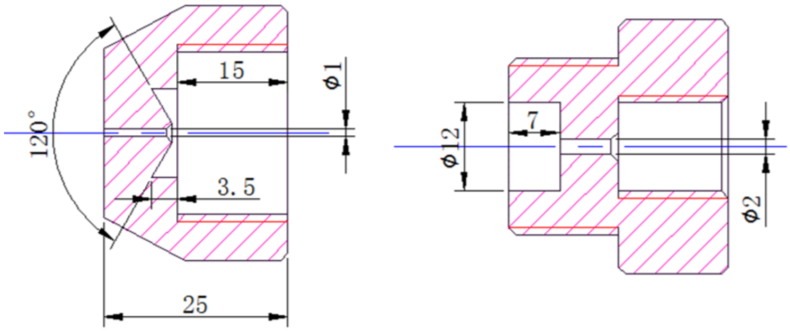

Fig. 3 Self-excited nozzle structure

2.4. Experimental analyzation

After LTTH and HPI, the values of D_{50}, SCOD (王 104), DNA(王 106), PGN(王 107), protein(王 105), the removal ratio of volatile suspended solids (VSS) and total solids (TS) were analyzed to confirm the disintegration effects.[23-25] The values were got by a digital digestion instrument (COD-571-1), an ultraviolet spectrophotometer (UV-5100), a laser particle size analyzer (Bettersize-2000), a type blast drying oven (HN101-0), and a centrifuge (TD5A).

In this study, the growth rate (GR), the ratio of index concentration difference and original index concentration, was used to assess the disintegration effects. The specific calculation formula was as follows:

$$\mathrm{GR} = \frac{A-B}{B} \times 100\% \tag{1}$$

where GR is the growth rate of each index; A is the index concentration after treatment, mg·L^{-1}; B is the index concentration before treatment, mg·L^{-1}.

The ratio of growth rate (RGR), the ratio of index concentration difference and sludge concentration, was used to assess the disintegration effects in different sludge concentration. The specific calculation formula was as follows:

$$\mathrm{RGR} = \frac{A-B}{C} \times 100\% \tag{2}$$

where RGR is the ratio of growth rate of index; A is the index concentration after treatment, mg·L^{-1}; B is the index concentration before treatment, mg·L^{-1}; C is the sludge concentration, mg·L^{-1}.

3. Result

3.1. Impingement

It had been proved that frictional shear, impingement and cavitation effects between sludge jets were the main effects of sludge disintegration.[26] Impingement increased force and cavitation effects in sludge, which was helpful for disintegration. Different impinged time, impinged pressure, impinged distance and sludge concentration were analyzed in univariate pre-experiments with two nozzles collision. The results of univariate pre-experiments were used for orthogonal test design, and the table of L9 (34) was adopted as

Table 1 showed. The actual sludge concentration had slightly difference because it's difficult to control accurately.

Table 1 Factors and levels of orthogonal test by HPI

Factors	Impinged time (min)	Impinged pressure (MPa)	Impinged distance (cm)	Sludge concentration ($mg \cdot L^{-1}$)
Level 1	6	6	3	3000
Level 2	9	10	5	5000
Level 3	12	14	7	7000

Table 2 Orthogonal test table of sludge disintegration by HPI

Number	Sludge concentration ($mg \cdot L^{-1}$)	Impinged time (min)	Impinged distance (cm)	Impinged pressure (MPa)	RGR (%)
1	3000	6	3	6	19.73
2	3000	9	5	10	26.38
3	3000	12	7	14	50.11
4	5000	6	5	14	28.15
5	5000	9	7	6	31.89
6	5000	12	3	10	28.46
7	7000	6	7	10	25.53
8	7000	9	3	14	32.86
9	7000	12	5	6	23.39
T_{i1}	32.07	24.47	27.02	25.34	
T_{i2}	29.50	30.38	26.31	26.79	
T_{i3}	27.60	34.32	35.84	37.04	
k_1	10.69	8.16	9.01	8.45	
k_2	9.83	10.13	8.77	8.93	
k_3	9.20	11.44	11.95	12.35	
Range (R)	4.81	9.52	9.87	12.04	
Order	Impinged pressure > Impinged distance > Impinged time > Sludge concentration				
Optimal level	3000	12	7	14	

The RGR was considered as evaluation index and the experimental results were shown in Table 2. The order of factors' influence on sludge disintegration was impinged pressure > impinged distance > impinged time > sludge concentration according to Table 2, without considering the interaction of factors. And k_i is the average RGR obtained in the third experiment at the level i. The optimal values of orthogonal test were 14 MPa, 7 cm, 12 min and 3000 $mg \cdot L^{-1}$, respectively. The optimal values indicated that higher impinged pressure, longer impinged time, appropriate impinged distance and sludge concentration leaded to better disintegration effects. Test number 3 got the best RGR of 50.11% with the optimal level of each factor, which showed the accuracy of the orthogonal test.

In this study, the variance analysis of results in Table 2 was conducted with IBM SPSS Statistics 23, as Table 3 showed. The significance value of each factor was less than 0.05, indicating that the four factors selected in the orthogonal test had significant influence on the RGR, but the significance of sludge concentration was much smaller than the other three factors. That proved this orthogonal test was reliable.

Table 3 Orthogonal experiment table of sludge disintegrated by HPJIS

Source	Type III sum of square	Degree of freedom	Mean square	F	Significance
Model	17085.11[a]	9	1898.34	843.32	0.0000000
Sludge concentration	60.59	2	30.30	13.46	0.0019732
Impinged time	338.72	2	169.36	75.24	0.0000024
Impinged distance	488.23	2	244.12	108.45	0.0000005
Impinged pressure	295.00	2	147.50	68.53	0.0000043
Error	20.26	9	2.25		
Sum	17105.37	18			

The longer impinged time and higher impinged pressure meant more energy consumption. Considering both disintegration effects and energy consumption, time of 9 min and pressure of 14 MPa were chosen. Because the RGR increased slower and slower after 9 min and 14 MPa, which meant more energy consumption could not improve disintegration effectively.

After ejecting from the nozzle, cavitation in sludge jet was influenced by the distance. Too high distance could help to cavitate completely, but the jet speed would be slow and impinged force became weak. Too low distance would limit cavitation process, which was negative to disintegration effects. 7 cm was appropriate. Lower sludge concentration had better effects, but the difference was very slightly and treatment efficiency with low concentration would be very poor. Besides, sludge concentration had the least significance of four factors. In order to be convenient in the combined method, sludge disintegration in HPI and LTTH should be consistent.

3.2. Hydrolysis

Thermal hydrolysis was recognized with the best potential to meet the objectives.[27] The heated time, temperature and sludge concentration influenced disintegration effects significantly. Many scholars reported about thermal hydrolysis, but the conditions of experiments were quite different. Nazari et al. heated 70 g sludge for 60-300 min with alkaline under 40-80 ℃, and the dissolve ratio of COD increased 20%.[28] Rocher M et al. reported that the bacterial cells in sludge became deactivated at 55℃ and completely deactivated at 60 ℃ in 2 min, because microorganisms cell walls would be destroyed at 60 ℃.[29] Ruiz et al. hydrolyzed sludge 10-30 min at 70-80 ℃ and found the longer time, the better biodegradability.[30] Farno et al. indicated that thermal treatment induced solubilization of organic matters which affected sludge rheology in 60min at 20-80 ℃.[31]

Previous reports showed that the optimal range of heated time and temperature were 40-80 min and 40-100 °C, with quite different volume of sludge. Thus, in this study, sludge concentration was explored firstly.

Consulting the achievements of predecessors, 5L sludge was heated in the condition of 60 min and 60 °C, with concentration of 3500, 6680, 11060, and 14005 mg·L^{-1}. As Fig. 4 showed, the RGR of SCOD, DNA and protein with different concentration had hardly difference. Sludge had been disintegrated adequately in all the four concentration. That indicated that sludge concentration might had no significant impact on disintegration effects. The range of 6000-10000 mg·L^{-1} had better VSS and TS removal ratios, which was the range of raw sludge. Dilution and dewatering were needed to get a lower or higher concentration, which were uneconomical by adding processing steps, wasting water and energy. The range of 6000-10000 mg·L^{-1}was chosen as optimal concentration.

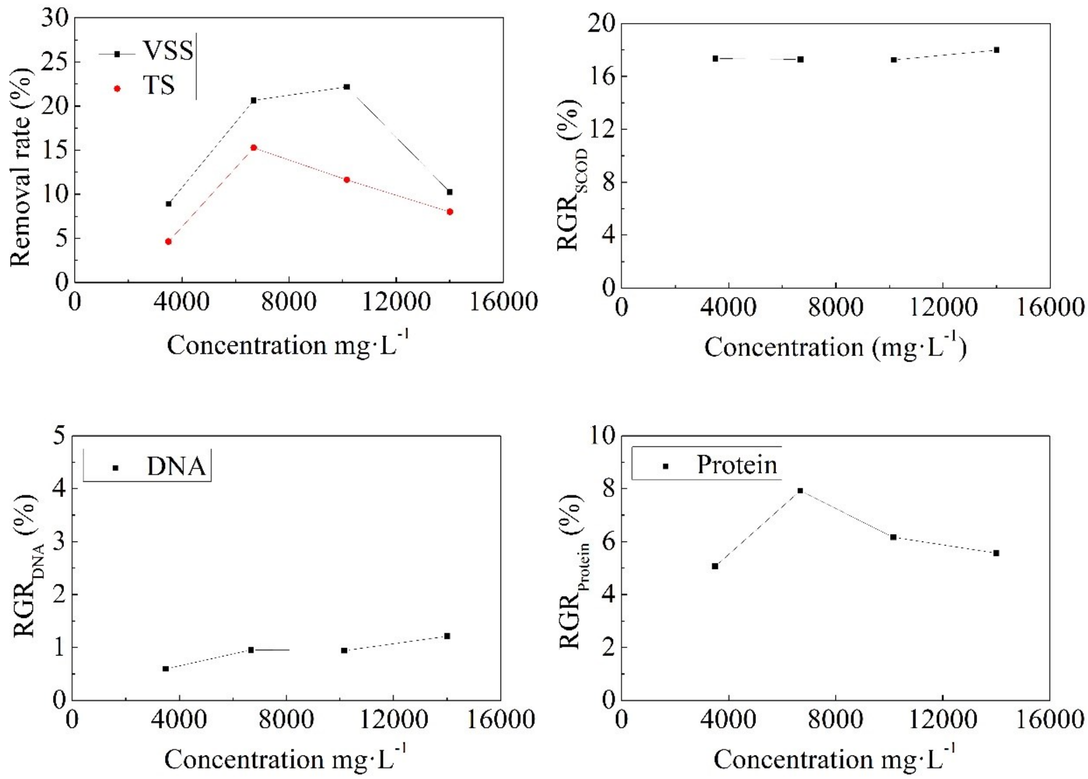

Fig. 4 Removal rates of TS and VSS, RGR of SCOD, DNA, and protein at different concentration

In the research recently, the hydrolysis time of under 1 h was applied widely. But those researches had different raw sludge conditions, temperature and diverse purposes, which could not be summarized an authoritative standard. In this study, 0~120 min was explored in 60 °C and 60 min to discuss the optimal time.

As Fig. 5 showed, with the increase of time, the removal ratios of VSS and TS reached the best at 60 min (TS 18.4%, VSS 25.3%). After that the removal rate decreased because some organic substances polymerized as hydrolysis process extending. SCOD, DNA and protein showed similar results that the contents growth rates decreased obviously after 60 min, which indicated that 60 min was enough for sludge disintegration and more time had no effective impact on disintegration. Ma et al. and Camacho et al. had similar results.

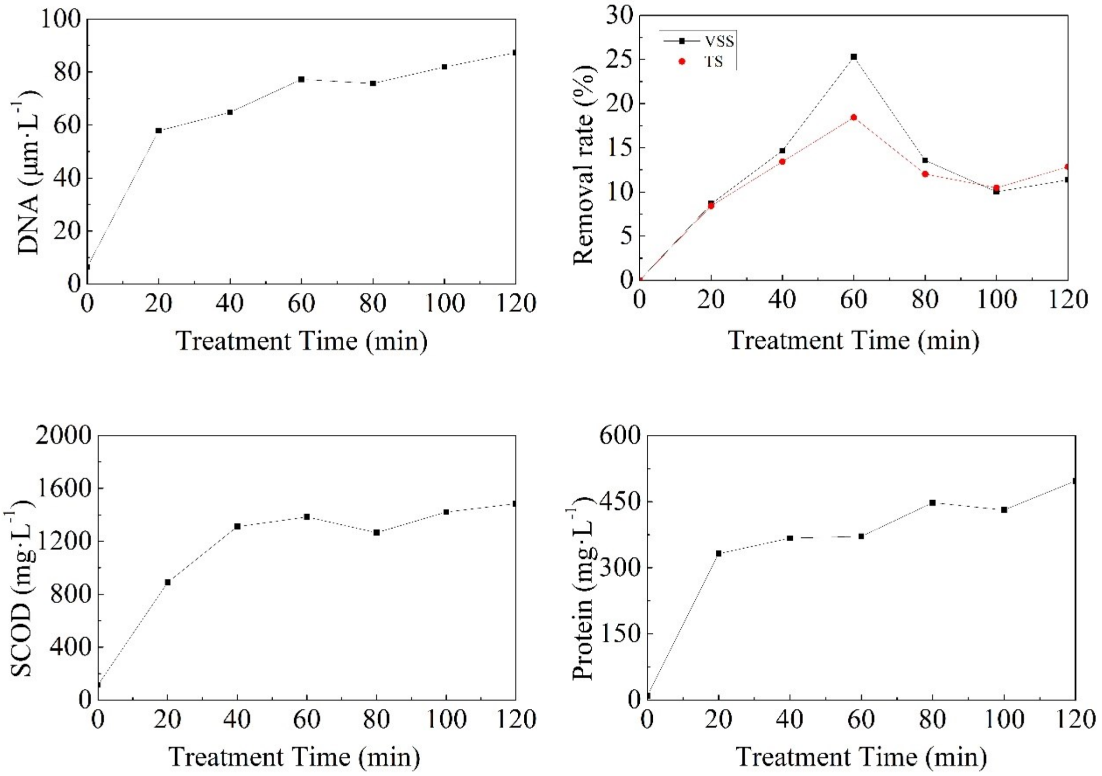

Fig. 5 Removal rates of TS and VSS, SCOD, DNA, and protein levels at different time

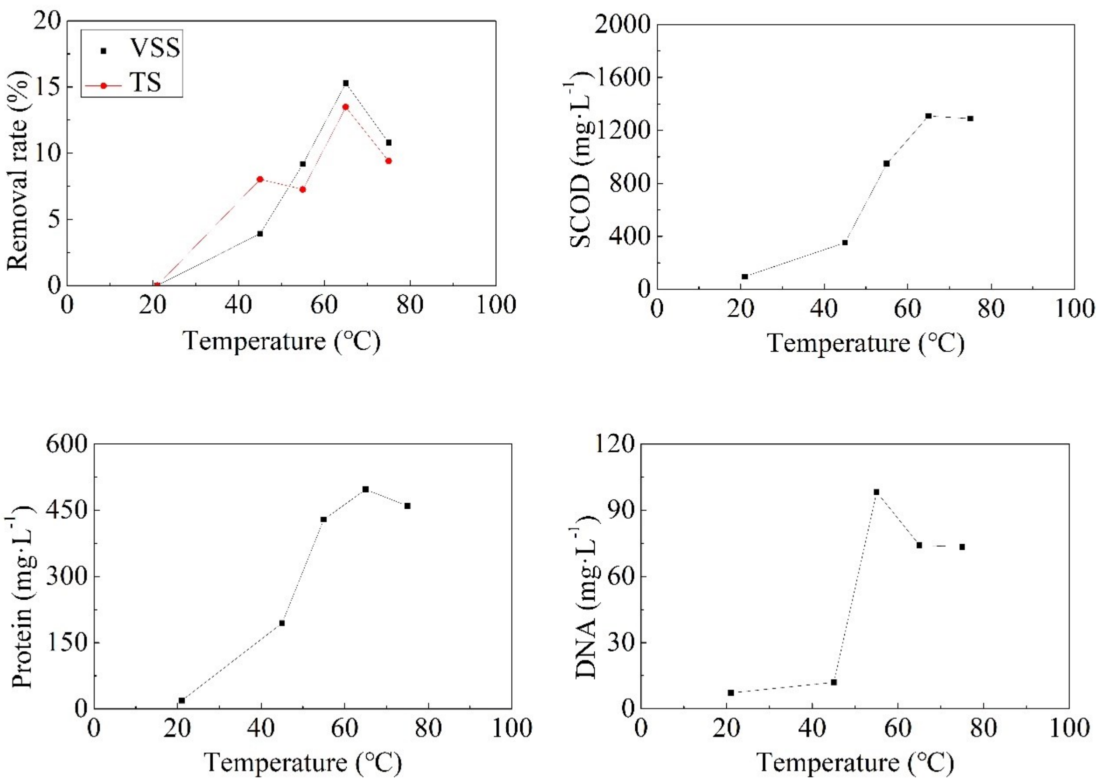

Fig. 6 Removal rates of TS and VSS, SCOD, DNA, and protein levels at different temperature

3.3. Impingement and hydrolysis

In combined experiments, sludge conditions should be as consistent as possible to facilitate convenience of process. Sludge concentrations of HPI and LTTH were quiet different (3000 mg·L^{-1} of HPI, 6000~10000 mg·L^{-1} of LTTH). In HPI, the significance of sludge concentration was the smallest, and the influence of different concentrations had not obvious distinction, which indicated that too low concentration was not necessary. Besides, when concentration was too low, the efficiency of treatment would reduce, which was conflict with economy. Therefore, the sludge concentration of combined experiments was chosen 6000~10000 mg·L^{-1}.

5L sludge was treated with 14 MPa, 7 cm, 8000 mg·L^{-1}, under the time of HPI 9 min and LTTH 60 min. The combined experiments were explored with the orders of HPI first then LTTH, and LTTH first then HPI. It should be noticed that in the order of LTTH first then HPI, sludge should be cooled in ice water to room temperature. TEM was used to observe the sludge conditions at the beginning, the middle and the end of combined experiments. As Fig. 7 showed, amounts of microorganisms polymerized in the form of zoogloeal with the effect of EPS, which contained intact cells. Most bacteria were coccus with diameters of 1 μm.

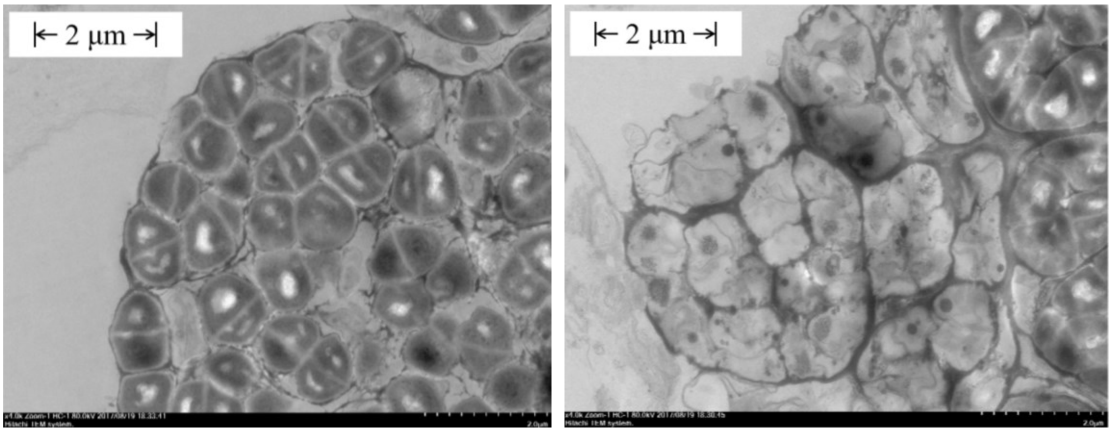

Fig. 7 TEM micrographs of raw sludge

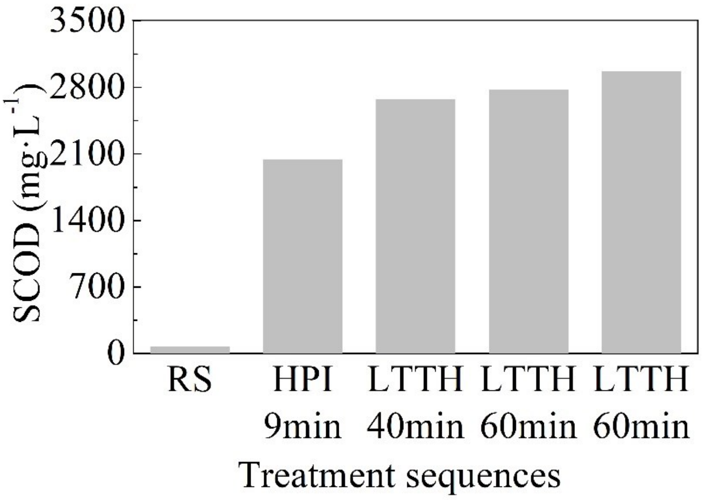

Fig. 8 SCOD levels after HPI + LTTH

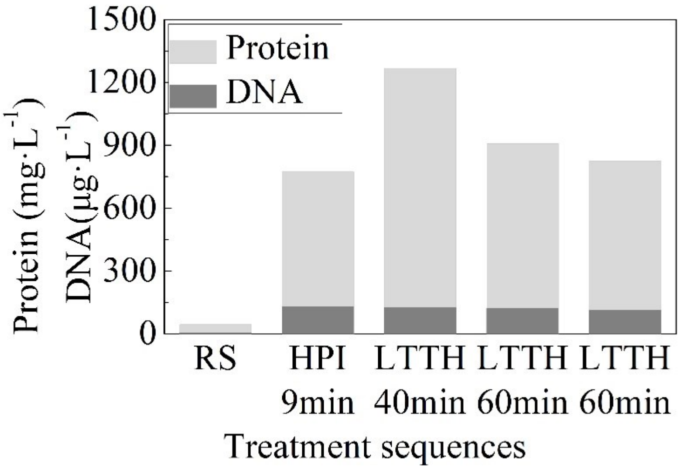

Fig. 9 DNA and protein levels after HPI + LTTH

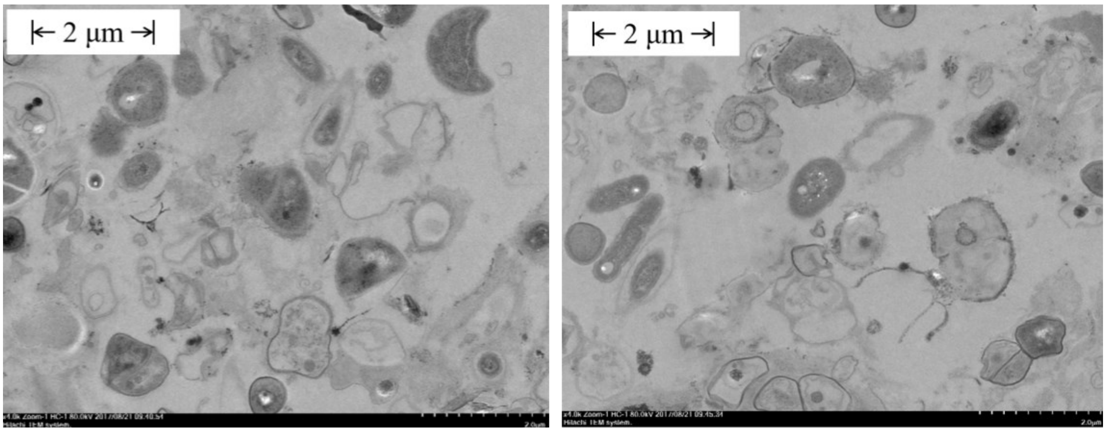

Fig. 10 TEM micrographs of sludge after HPI

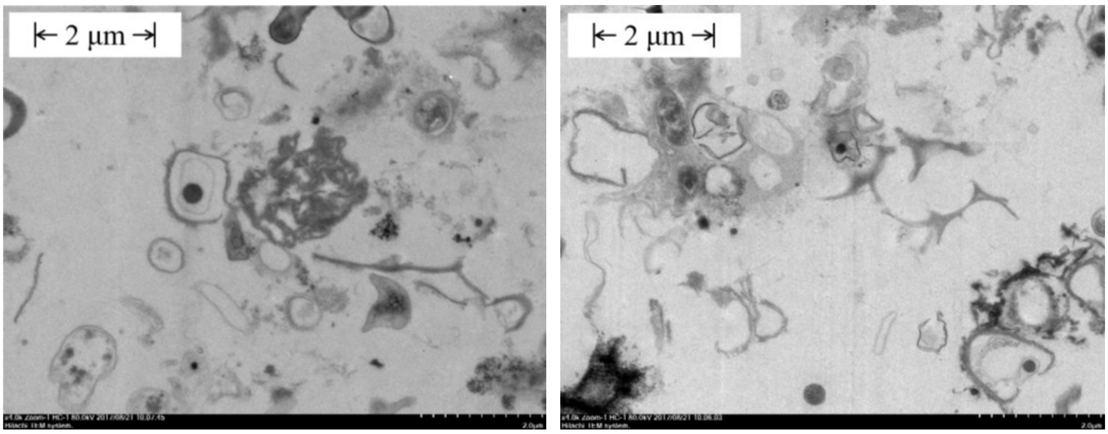

Fig. 11 TEM micrographs of sludge after HPI and LTTH

As Fig. 12 showed, LTTH could disintegrate sludge effectively as SCOD increasing to 1147 mg·L^{-1}. However, the SCOD after HPI in previous experiments was twice than LTTH, which meant that HPI was the main method disintegrating sludge. Then, HPI was explored and SCOD increased rapidly to 2223.7

$mg \cdot L^{-1}$ in 3 min. That's because LTTH disintegrated zoogloea in the sludge which helped HPI to affect cells more significantly. As time increasing, the growth of SCOD became slowly. Fig. 13 shoowed that DNA and protein had similar tend to SCOD, which indicated that longer time of HPI after LTTH wouldn't have better effect. The results showed that LTTH could enhance the effect of HPI significantly.

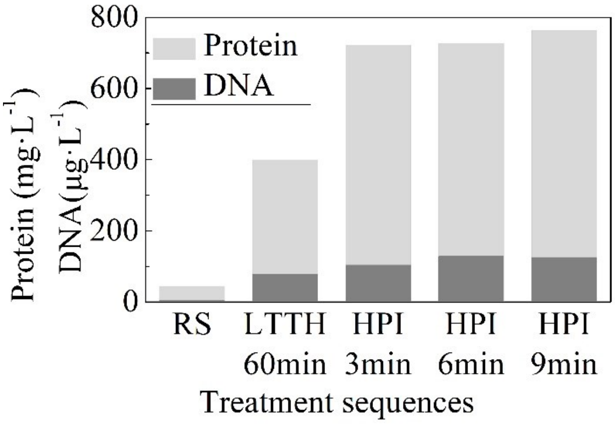

Fig. 12 SCOD levels after LTTH + HPI

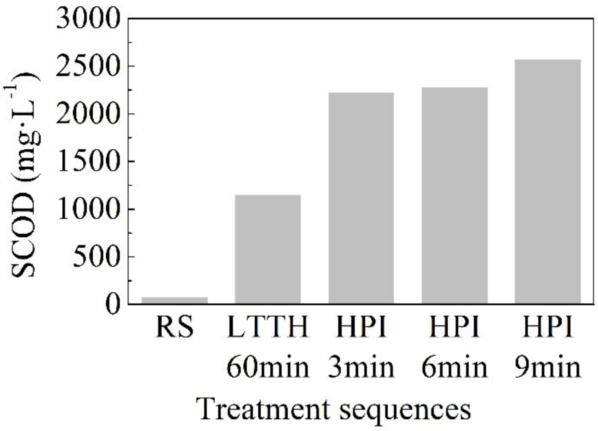

Fig. 13 DNA and protein levels after LTTH + HPI

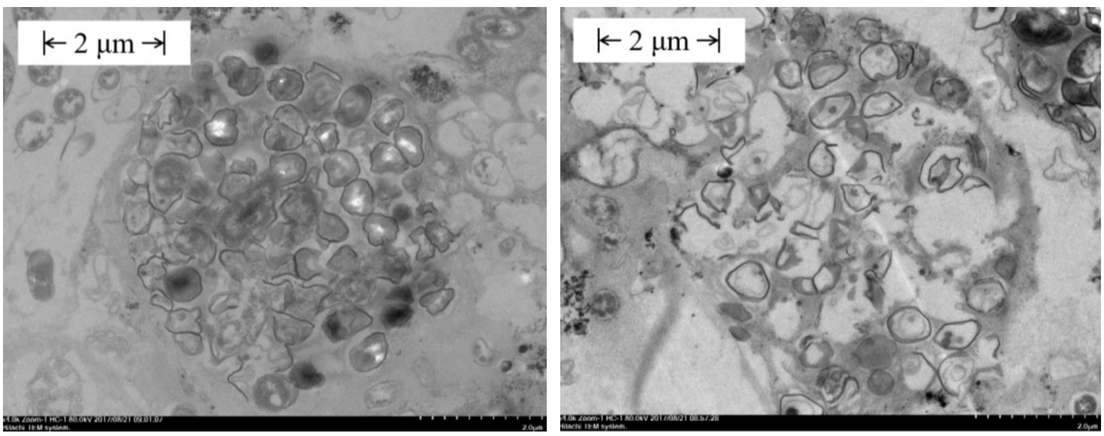

Fig. 14 TEM micrographs of sludge after LTTH

It's clearly in Fig. 14 that most of zoogloea in sludge dissolved and the cells kept intact after LTTH, which proved that LTTH could disintegrate zoogloeal and dissolve EPS in sludge. After HPI, as Fig. 15 showed, most of cells even cytoarchitecture were destroyed, which led to the increase of DNA. Those results verified the previous conjecture that LTTH could dissolve EPS and HPI could disintegrate sludge cells, which was consistent with previous results.

To summarize, LTTH and HPI could enhance the disintegrated effects mutually. The optimal order should be LTTH first then HPI, because the sludge would be easier to disintegrate by HPI after dissolving EPS. And it's clearly to see that the sludge structure would be more fragmented with TEM.

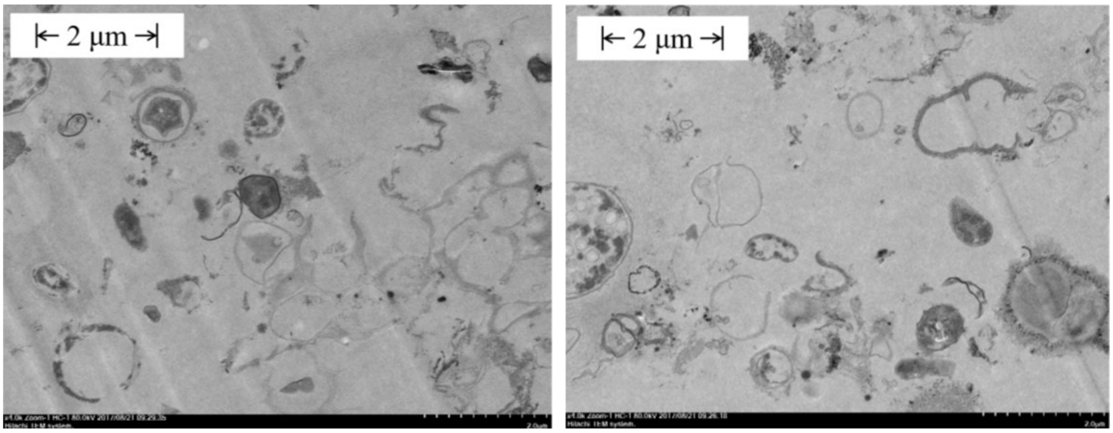

Fig. 15 TEM micrographs of sludge after LTTH+HPI

4. Conclusion

In this study, the experiments of HPI, LTTH and combined method were explored.

HPI could disintegrate sludge effectively, with the optimal parameters of 9~12 min, 14 MPa, 7 cm, 3000 mg·L^{-1}, and the order of factors' influence on sludge disintegration was impinged pressure > impinged distance > impinged time > sludge concentration.

LTTH could disintegrate sludge effectively, with the optimal parameters of 40~60 min, 60~70 ℃, 6000~10000 mg·L^{-1}.

Combined method with LTTH first then HPI had better effect of disintegrate effects. LTTH and HPI could strengthen the sludge disintegration mutually. LTTH could dissolve EPS effectively and HPI could disintegrate cells significantly, that's why sludge disintegration with LTTH first then HPI had better effects. Besides, to achieve the similar disintegrated effects, combined method took less time than single method, which was economical.

Acknowledgements

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