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Combined alkaline and ultrasonic pretreatment of sludge before aerobic digestion

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Abstract

Alkaline and ultrasonic sludge disintegration can be used as the pretreatment of waste activated sludge (WAS) to promote the subsequent anaerobic or aerobic digestion. In this study, different combinations of these two methods were investigated. The evaluation was based on the quantity of soluble chemical oxygen demand (SCOD) in the pretreated sludge as well as the degradation of organic matter in the subsequent aerobic digestion. For WAS samples with combined pretreatment, the released COD levels were higher than those with ultrasonic or alkaline pretreatment alone. When combined with the ultrasonic treatment, NaOH treatment was more efficient than $Ca(OH)_2$ for WAS solubilization. The COD levels released in various sequential options of combined NaOH and ultrasonic treatments were in the the following descending order: simultaneous treatment > NaOH treatment followed by ultrasonic treatment > ultrasonic treatment followed by NaOH treatment. For simultaneous treatment, low NaOH dosage (100 g/kg dry solid), short duration (30 min) of NaOH treatment, and low ultrasonic specific energy (7500 kJ/kg dry solid) were suitable for sludge disintegration. Using combined NaOH and ultrasonic pretreatment with optimal parameters, the degradation efficiency of organic matter was increased from 38.0% to 50.7%, which is much higher than that with ultrasonic (42.5%) or with NaOH pretreatment (43.5%) in the subsequent aerobic digestion at the same retention time.

Key words: waste activated sludge (WAS); ultrasonic treatment; alkaline treatment; aerobic digestion

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Introduction

Waste activated sludge (WAS) is the main by-product of wastewater treatment processes, and its mass is about 0.5%–1% of total influent water. In China, 20%–50% of operation costs of wastewater treatment plants are spent on WAS treatment. To minimize the quantities of sludge, anaerobic digestion and aerobic digestion are commonly used in most municipal wastewater treatment plants. Generally, aerobic digestion is more suitable for small wastewater treatment plants due to its low capital cost and high energy consumption (Barbusinski and Kosc, 1997), and also can be used in medium-sized treatment plants. However, aerobic digestion requires large digestion tanks due to the long detention time (15–30 d) for digesters.

Since the hydrolysis of proteins and carbohydrates in sludge is the rate-limiting step, sludge disintegration pretreatment can be considered as a simple candidate for faster sludge degradation or higher degradation degree in a fixed duration (Kim *et al*., 2002). Sludge disintegration methods include mechanical (Muller *et al*., 1998; Kampas *et al*., 2007), thermal (Stuckey and McCarty, 1984; Camacho *et al*., 2002), chemical (Rajan *et al*., 1989; Lin *et al*., 1997) and biological treatments. They can disrupt sludge flocs and bacteria cells, release cellular components and accelerate subsequent aerobic digestion.

Compared with other methods, alkaline treatment has advantages of simple device, convenient operation, and high efficiency (Weemaes and Verstraete, 1998; Navia *et al*., 2002; Kim *et al*., 2003; Neyens *et al*., 2004; Cassini *et al*., 2006). Alkaline treatment destroys floc structures and cell walls by hydroxy anions. Extremely high pH causes natural shape losing of proteins, saponification of lipid, and hydrolysis of RNA. Chemical degradation and ionization of the hydroxyl groups ($-OH \longrightarrow -O^-$) lead to extensive swelling and subsequent solubilization of gels in sludge (Neyens *et al*., 2004). After the destruction of extracellular polymer substances (EPS), the cell walls, being exposed to a high pH, cannot withstand the appropriate turgor pressure, that results in the disruption of cells and release of intracellular substances (Erdincler and Vesilind, 2000).

Ultrasonic treatment is another common sludge disintegration method, which has also been studied extensively owing to its effectiveness and simple operation (Tiehm *et al*., 1997; Wang *et al*., 1999; Chu *et al*., 2001; Tiehm *et al*., 2001; Gonze *et al*., 2003; Hogan *et al*., 2004; Bougrier *et al*., 2005; Gronroos *et al*., 2005; Dewil *et al*., 2006; Zhang *et al*., 2007). As ultrasound pressure waves propagate through water, gas and vapor bubbles are formed. They

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grow and collapse violently at high velocity, which leads to high shear forces and micro-region with an estimated temperature as high as 5000 K and a pressure up to 1×10^8 Pa. This phenomenon is called acoustic cavitation. Water undergoes thermolysis in the bubbles and releases radical species (Riesz *et al*., 1985). During ultrasonic sludge treatment, high shear forces predominantly affect the sludge disintegration (Wang *et al*., 2005). Since ultrasonic and alkaline treatment are based on different mechanisms of sludge dissolution, the combination of these two methods takes advantages of both methods and can achieve a better treatment efficiency. Chiu *et al*. (1997) reported that the ratio of soluble chemical oxygen demand (SCOD) and total chemical oxygen demand (TCOD) increased from 36.3% to 89.3% in case that ultrasonic treatment was followed by alkaline treatment. The ratio could reach 77.9%, if simultaneous alkaline and ultrasonic treatments were performed.

In most reports, alkaline treatment and ultrasonic treatment were studied separately as the pretreatment methods of anaerobic digestion. Wang *et al*., (2005) investigated the effect of pH on ultrasonic treatment, in which the alkali only played a supplemental role with a low-level dosage. The purpose of current work was to provide more insights into the changes of sludge characteristics during combined alkaline and ultrasonic pretreatment, and to explore the impact of the pretreatment on subsequent aerobic digestion. Both NaOH and $Ca(OH)_2$ in different dosage were used. The ultrasonic power density was 0.1–0.4 W/mL. The degree of sludge disintegration was measured by the changes of SCOD. Based on the analytical results, an appropriate treatment sequence alkaline and ultrasonic parameters were selected. The pretreatment scheme was verified by subsequent aerobic digestion.

1 Materials and methods

1.1 Waste activated sludge characterization

In this study, sludge samples were taken from outlets of aerobic tanks and a thickening tank in a local full-scale municipal wastewater treatment plant, where the anaerobic-anoxic-aerobic process was performed. Samples were stored at 4°C before use. The characteristics of sludge samples are shown in Table 1.

1.2 Sludge disintegration

Alkaline sludge disintegration was performed in a 2.0-L batch mixed reactor, which was placed in a water bath (THZ95, SBL Ltd., China) to adjust the reaction temperature to (20 ± 2)°C. The NaOH dosage ranged from 0.04 to 0.5 mol/L (100–1250 g/kg dry solid (ds)), and $Ca(OH)_2$ dosage varied from 0.02 to 0.3 mol/L (92.5–1387.5 g/kg ds).

Table 1 Characteristics of sludge samples

Parameter	Value
Moisture content (%)	98.1
pH	6.9
Soluble chemical oxygen demand (SCOD) (mg/L)	275
Total solid (TS) (mg/L)	16277
Suspended solid (SS) (mg/L)	16084
Volatile suspended solid (VSS) (mg/L)	12726
Organic content (%)	79.1

The ultrasonic irradiation of sludge was performed in a probe system (JY90-II, Xinzhi Inc., China) that emits 25 kHz ultrasound through a tip with a diameter of 6 mm. For each sonication test, 100 mL sludge was filled in a stainless steel beaker and the ultrasonic probe was dipped 1 cm into the sludge. The beaker was placed in a water bath (THZ95, SBL Ltd., China) to maintain a temperature at (20 ± 2)°C. The specific supplied energy (E_s) was defined as a function of ultrasonic power (P), ultrasonic time (t), sample volume (V), and initial total solid concentration (T_S^0):

$$E_s = \frac{P \times t}{V \times T_S^0} \quad (1)$$

Three sequential options of combined ultrasonic and alkaline treatment were tested: ultrasonic treatment followed by alkaline treatment, alkaline treatment followed by ultrasonic treatment, and simultaneous treatment. During the simultaneous treatment, aliquots of alkali were introduced into sludge samples, after mixing, the samples were treated in the ultrasonic reactor immediately.

1.3 Aerobic digestion

Aerobic digestion expriments were carried out in two plexiglass cylinders with an effective volume of 7 L each (Fig. 1). The sludge retention time was fixed at 10 d and the effect of pretreatment was evaluated with the degradation of volatile suspended solid (VSS). Everyday, 700 mL digested sludge was discharged from each reactor, and then 700 mL fresh sludge or pretreated sludge was added into each of the two reactors. Circular water system was used to maintain the temperature at 30°C. Air flux in the digesters was 40 L/h and the dissolved oxygen (DO) was about 5 mg/L. The suspended solid (SS) and

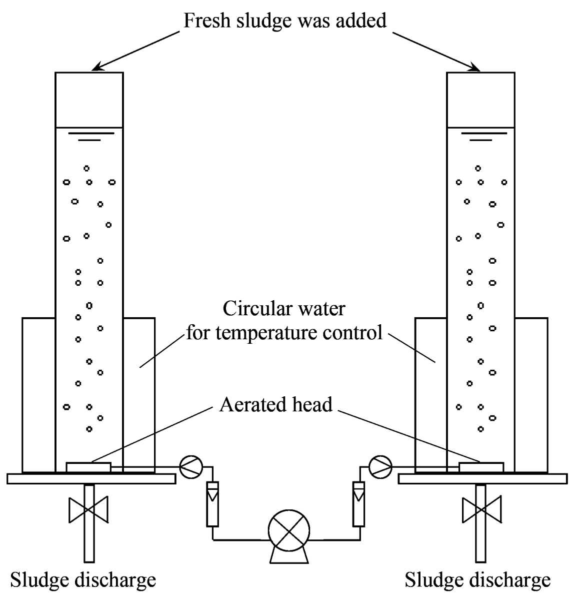

Fig. 1 Experimental device for aerobic digestion.

VSS of digested sludge were monitored during the aerobic digestion process. The settlement performance of digested sludge, as well as SCOD, pH, total nitrogen (TN), total phosphate (TP) variation in the supernatant phase, were also examined.

1.4 Analytical procedures

The sludge samples were used directly for the measurement of their water content, TS, SS, VSS, pH and TCOD according to the standard methods (APHA, 1995). The pH value was measured using a Cyberscan510 pH meter (Eutech, Singapore). The samples were centrifuged at 5000 $\times g$ for 10 min and then filtered through a 0.45-μm membrane. The filtrate was used for the measurements of SCOD, TN and TP. TN was determined with alkaline potassium persulphate digestion-UV spectrophotometric method, and TP was determined with ammonium molybdate spectrophotometric method.

The degree of sludge disintegration (DD_{COD}) was calculated as the ratio of the SCOD increment by alkaline or ultrasonic treatment to the maximum possible SCOD increment:

$$DD_{COD} = \frac{SCOD - SCOD_0}{TCOD - SCOD_0} \quad (2)$$

To measure the sludge dewaterability, the settling and centrifugal methods were utilized. The settling ability was represented by the settling volume of 100 mL sludge in a measuring cylinder in 30 min (SV_{30}) or 120 min (SV_{120}). The centrifugal ability was represented by the water content of centrifugal cake (5000 $\times g$, 10 min) and the turbidity of centrifugal supernatant. The turbidity of sludge supernatant was measured with a HACH 2100P nephelometer. Sludge particle size was examined by a Mastersizer laser beam diffraction granulometer (Malvern, U.K.).

2 Results and discussion

2.1 Effect of treatment sequence on sludge disintegration

The effect of alkaline sludge treatment is presented in Fig. 2. NaOH was more efficient than $Ca(OH)_2$ for the solubilization of organic substances. The SCOD value decreased as the $Ca(OH)_2$ dosage exceeded 0.02 mol/L ds. Because bivalent cations, such as Ca^{2+} and Mg^{2+}, are key substances connecting cells with extracellular polymer substances (EPS) (Urbain *et al*., 1993), the dissolved organic polymers were re-flocculated (Neyens *et al*., 2003) in the presence of calcium cations that led to a decrease in SCOD. For the same reason, the NaOH treatment can release more COD than the $Ca(OH)_2$ treatment when combined with ultrasonic treatment (Fig. 3). The released COD was approximately equal to the sum of the released COD by ultrasonic and by NaOH treatment alone. For combined NaOH (0.04 mol/L, 30 min) and ultrasonic (3750 kJ/kg ds) treatment, the increase of SCOD approached the level during a 24-h NaOH (0.5 mol/L) treatment. Hence, the addition of ultrasonic treatment can reduce the required alkaline dosage and shorten the treatment duration time for the same disintegration efficiency.

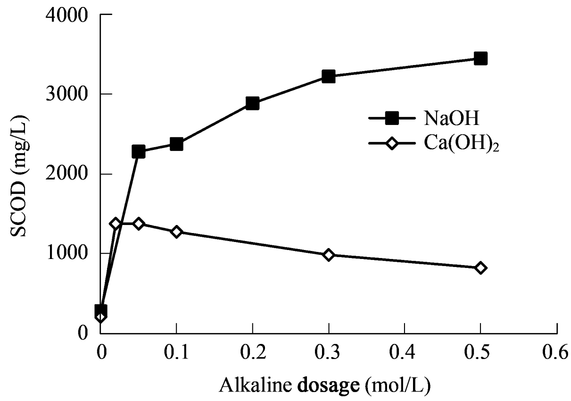

Fig. 2 Sludge solubilization with various alkaline treatments (treatment duration time 30 min).

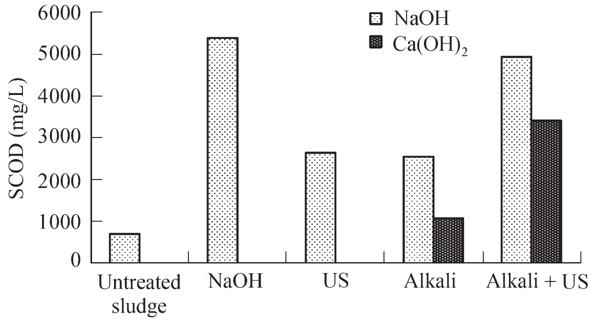

Fig. 3 Effect of combined alkaline and ultrasonic (US) treatment on sludge solubilization. Condition: NaOH: 0.5 mol/L 24 h; US: 3750 kJ/kg ds; alkali: NaOH or CaOH 0.04 mol/L, 30 min; alkali+US: NaOH or CaOH 0.04 mol/L, US 3750 kJ/kg ds.

The effect of treatment on sludge disintegration is shown in Table 2. The amounts of released COD for different treatment processes were in the following order: simultaneous treatment > NaOH treatment followed by ultrasonic treatment >ultrasonic treatment followed by NaOH treatment. The results were in agreement with the report by Chiu *et al*. (1997). This was due to the fact that some dissolved organic polymers enhanced the re-flocculation of disrupted floc fragments. Although this enhancement effect by organic polymers was not as significant as that with the aid of Ca^{2+}. Sludge particle size distributions were monitored to verify the reflocculation effect. The results

Table 2 Effects of treatment sequence on sludge solubilization and dewaterability

Treatment	SCOD (mg/L)	SV_{30} (%)	Water content (%)	Turbidity (NTU)
Untreated sludge	275	100	92.5	65.6
NaOH treatment	5429	80	92.3	81.3
US followed by NaOH treatment	5976	43	91.9	395
NaOH treatment followed by US	6408	100	87.3	994
Simultaneous treatment	6797	100	88.9	701

US: ultrasonic treatments 3750 kJ/kg ds; NaOH treatments: 0.5 mol/L, 30 min; water content of centrifugal sludge cake (5000 $\times g$, 10 min); turbidity of centrifugal supernatant (5000 $\times g$, 10 min).

showed that the accumulative size distribution d_{90} of sludge particles increased from 115.69 to 135.51 μm (data not shown) after NaOH (0.5 mol/L) treatment. Ultrasonic treatment can disintegrate sludge flocs, nevertheless the fragments can be re-flocculated and formed compact flocs (initial sludge flocs were porous and loose) during the subsequent NaOH treatment. This was not desirable for sludge disintegration. When ultrasonic treatment was applied simultaneously with alkaline treatment, the re-flocculation process could be retarded by ultrasonic treatment. Moreover, the simultaneous treatment can utilize the interaction of chemical and mechanical effects adequately. For the ultrasonic treatment, the protection of EPS and gels limited its ability for sludge disintegration, while alkaline treatment promote the EPS hydrolysis and gels solubilization. Therefore, simultaneous ultrasonic and alkaline treatment was the most option for sludge disintegration, and was chosen as a optimal treatment method in the following experiments.

Sludge dewaterability was also examined to verify the change of sludge flocs during alkaline and ultrasonic treatments (Table 2). Ultrasonic treatment followed by Alkaline treatment was the best option for sludge settlement. As stated above, ultrasonic treatment resulted in smaller particle size, while subsequent high-dosage alkaline treatment will make these fragments re-flocculated. Moreover, the density of the re-flocculated particles increased due to solubilization of organic substances. Its compact structure and low organic content improved sludge settling performance obviously. The supernatant turbidity also increased because some fine particles were produced during the ultrasonic treatment. A study of the sludge particle size distribution showed that the accumulative size distribution d_{10} of sludge particles decreased from 15.77 to 11.87 μm (data not shown) after NaOH (0.5 mol/L) treatment. If alkaline treatment followed with ultrasonic treatment or it was emerged into simultaneously combined treatment, the re-flocculation process was inhabited by the ultrasonic treatment, which resulted in obvious deterioration of sludge settling ability and significant increase of supernatant turbidity.

2.2 Appropriate ultrasonic and alkaline parameters

For simultaneous ultrasonic and alkaline treatment, the degree of sludge disintegration was influenced by the NaOH treatment duration time. During the NaOH treatment, an initial period (30 min) of rapid increase of SCOD and a subsequent periods of slow SCOD increase were observed (Fig. 4). Similar duration time of the first period was also reported previously (Riesz *et al*., 1985; Wang *et al*., 2005). The amount of solubilized organic substances in the first 30 min was 60%–71% of the total amount in 24 h. The solubilization ratio was consistent with the EPS content of WAS, which is generally in the range of 50%–90% of total sludge organic substances (Frolund *et al*., 1996; Nielsen *et al*., 1997; John and Nielsen, 1998; Dignac *et al*., 1998). Since the cells were surrounded by EPS (Chu *et al*., 2001), it could be concluded that during the first period only the EPS were solubilized. As

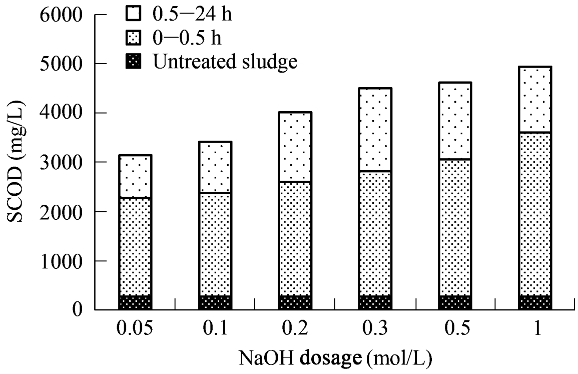

Fig. 4 Variation of SCOD during 24 h NaOH treatment.

the highly porous flocs were disrupted, the extracellular organic substances contained in the flocs were released. During the second period, cell walls were disrupted and intracellular substances were released gradually. Since sludge flocs were difficult to be disintegrated by ultrasonic treatment alone owing to the protection of EPS and gels, a combination of 30 min NaOH treatment with ultrasonic treatment was appropriate.

For simultaneous ultrasonic and alkaline treatment, the degree of sludge disintegration degree increased with both the NaOH dosage and ultrasonic specific energy (Fig. 5). As the NaOH dosage exceeded 0.04 mol/L, no further increase of SCOD was observed. After a 30-min NaOH treatment, the pH value of the sludge remained higher than 12. This implies that NaOH was excessive even at a low dosage of 0.04 mol/L. Similar result was reported by Xiao and Liu (2006). Hence, a dosage of 0.04 mol/L was sufficient in a 30-min NaOH treatment.

When NaOH dosage was chosen to be 0.04 mol/L and treatment duration time to be 30 min, the degree of sludge disintegration was about 30%. The addition of ultrasonic treatment can improve sludge disintegration (Fig. 6). For the ultrasonic sludge treatment, the reaction fits well with a first-order equation in short sonication time (Wang *et al*., 2005; Zhang *et al*., 2007). The change of DD_{COD} can be expressed as:

$$DD_{COD} = 2.27 \times 10^{-5} \times E_s + 0.3 \qquad R^2 = 0.9322 \qquad (3)$$

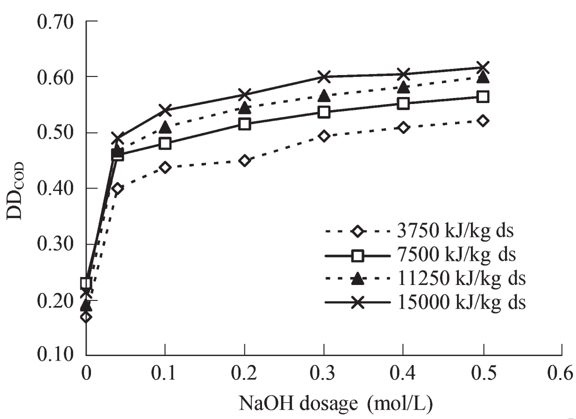

Fig. 5 Variation of the degree of sludge disintegration with the ultrasonic specific energy and NaOH dosage (NaOH treatment duration: 30 min).

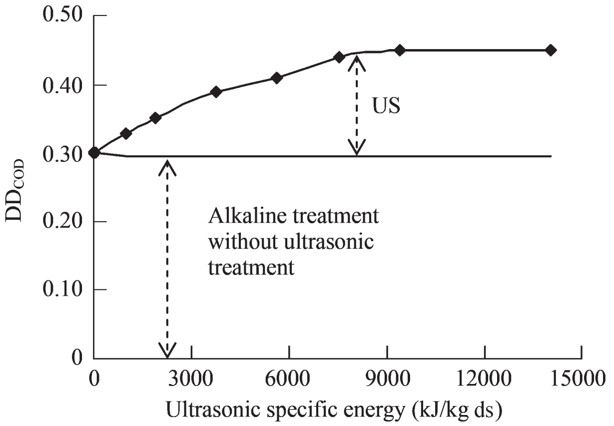

Fig. 6 Variation of the degree of sludge disintegration with the sonication time (ultrasonic power density: 0.2 W/mL; NaOH treatment: 0.04 mol/L, 30 min).

where, 2.27×10^{-5} is the coefficient for 0.2 W/mL ultrasonic treatment within 10 min; 0.3 is the degree of sludge disintegration with NaOH treatment (0.04 mol/L, 30 min). The degree of sludge disintegration could be controlled through the adjustment of ultrasonic specific energy and NaOH dosage (0–0.04 mol/L) for subsequent aerobic digestion.

2.3 Effect of pretreatment on aerobic digestion

Base on the above findings, simultaneous ultrasonic and NaOH treatment was used as the pretreatment method for aerobic digestion. Samples were collected after the aerobic digestion reached a steady state. From day 1 to day 5, the reactors run normally without any pretreatment. From day 6 to day 13, only ultrasonic pretreatment (7500 kJ/kg ds) was introduced. From day 14 to day 22, only NaOH pretreatment (0.04 mol/L, 30 min) was employed and the pH of pretreated sludge was adjusted to 7.0. From day 23 to day 40, the combined ultrasonic and NaOH pretreatment was performed (Fig. 7). It was found that all these pretreatments enhanced the removal of sludge suspended solid (VSS degradation rate increased from 38.0% to 42.5%, 43.5% and 50.7% with ultrasonic, alkaline, and combining pretreatments, respectively), where, the combined ultrasonic and NaOH pretreatment gave the best results. Moreover, the combined pretreatment could lead to a shorter sludge retention time for the same VSS degradation rate.

Other characteristics of the digested sludge were also measured (Table 3). SV_{120} of the digested sludge from the ultrasonic and alkaline assisted digestion reactor (UAAD) was lower than that from the conventional aerobic digestion reactor (CAD). However, the ratios of SV−120/SS in the two reactors were similar. This revealed a good settlement performance of the digested sludge from UAAD. The pH value of the digested sludge from UAAD was slightly higher than that from CAD. When disintegrated sludge was added into the UAAD reactor, the organic substrate level increased. This can result in both a decrease in nitrification efficiency (Genc *et al*., 2003) and an increase in pH, which was beneficial for aerobic digestion. The concentrations of SCOD, TN, and TP in the supernatant of the digested sludge increased slightly. Therefore, the pretreatment of combined ultrasonic and alkaline pretreatment could improve aerobic digestion without obvious deterioration of the digested sludge characteristics.

Table 3 Effect of ultrasonic and alkaline pretreatment on digested sludge characteristics

Digested sludge parameters	CAD	UAAD		
		Period 1	Period 2	Period 3
SV_{120} (%)	94	90	79	75
pH	4.6	4.7	4.7	4.9
SCOD (mg/L)	317	301	410	441
TN (mg/L)	73	79	85	83
TP (mg/L)	21	30	35	29

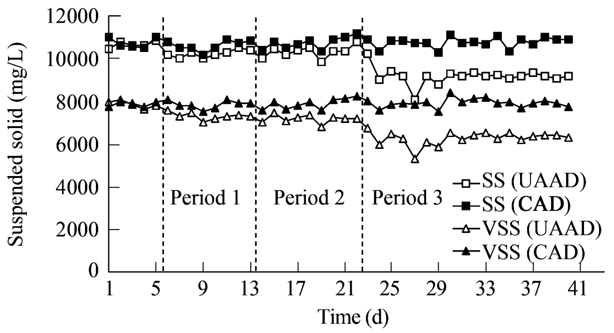

Fig. 7 Effect of ultrasonic and NaOH pretreatment on sludge aerobic digestion. UAAD: ultrasonic and alkaline assisted digestion reactor; CAD: conventional aerobic digestion reactor. Period 1: from day 6 to day 13, only ultrasonic pretreatment (7500 kJ/kg ds) was introduced; Period 2: from day 14 to day 22, only NaOH pretreatment (0.04 mol/L, 30 min) was employed; Period 3: from day 23 to day 40, combined ultrasonic and NaOH pretreatment was performed.

Although both NaOH and ultrasonic pretreatment could cause extra costa, the subsequent treatment cost can be largely reduced because of sludge decrement. In this study, CAD can only reduce about 38% of the sludge, while UAAD can reduce over 50% of the sludge. It means that the cost in subsequent sludge dewatering, drying, incineration or landfilling could be reduced. Furthermore, faster degradation means smaller digestors or shorter treatment duration can be employed. Thus, the combined pretreatment could also save capital and aeration costs.

3 Conclusions

The combination of alkaline and ultrasonic pretreatments could increase the degree of sludge disintegration. In case of alkaline treatment, NaOH was more effective than $Ca(OH)_2$. On one hand, the ultrasonic treatment disrupted flocs and cells quickly, and promoted NaOH treatment; on the other hand, the NaOH treatment led to the hydrolysis of EPS and the solubilization of gels, and enhanced the efficiency of ultrasonic treatment. Consequently, the combination was beneficial for both pretreatment methods.

The degrees of sludge disintegration in various sequential options of combined pretreatment was foun to be in the following order: simultaneous treatment > alkaline treatment followed by ultrasonic treatment > ultrasonic treatment followed by alkaline treatment.

The digested sludge with combined pretreatment had good settling ability, and the concentrations of supernatant organic pollutants increased slightly, as compared with those from aerobic digestion without any pretreatment.

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