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Enhanced anaerobic digestion and sludge dewaterability by alkaline pretreatment and its mechanism

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Abstract

To investigate the influences of alkaline pretreatment on anaerobic digestion (AD) and sludge dewaterability after AD, waste activated sludge was adjusted to different pH values (8, 9, 10, 11, 12) and placed at ambient temperature for 24 hr. The samples were then adjusted to the initial pH and subjected to 25 days of AD. The results showed that, when compared with the control (pH 6.8), total suspended solids (TSS) and volatile suspended solids (VSS) reduction following pretreatment at pH 9–11 increased by 10.7%–13.1% and 6.5%–12.8%, respectively, while biogas production improved by 7.2%–15.4%. Additionally, significant enhancement of sludge dewaterability after AD occurred when pretreatment at pH 8–9 was conducted. The proteins and carbohydrates transferred from the pellet and tightly bound extracellular polymeric substances (TB-EPS) fractions to the slime and loosely bound EPS (LB-EPS) fractions after pretreatment and during the AD process, and the concentrations of proteins and carbohydrates in the slime fraction had a good linear relationship with the normalized capillary suction time (CST). During the AD process, the normalized CST was positively correlated with the organic materials in the loosely bound fraction of the sludge matrix ($R^2 \geqslant 0.700$, $p < 0.01$), while it was negatively correlated with the organic materials in the tightly bound fraction ($R^2 \geqslant 0.702$, $p < 0.01$). These results suggest that alkaline pretreatment could break the EPS matrix and release inner organic materials, thus influencing the efficiency of the AD process and dewaterability after AD.

Key words: waste activated sludge; alkaline pretreatment; digestibility; dewaterability; extracellular polymeric substances

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Introduction

Waste activated sludge (WAS) is the inevitable byproduct of sewage treatment processes in municipal wastewater treatment plants (WWTP). Improper disposal of WAS can cause serious harm to the environment, due to the high concentrations of pathogens and organic materials present in WAS (Li et al., 2008). Anaerobic digestion (AD) has been widely employed in recent years because of its obvious advantages of low energy requirement, stable digestive products and biogas production in the form of methane (Lin et al., 1997). However, the low hydrolysis rate of granular organics in the sludge matrix limits the performance of AD. Recently, increased attention has been given to pretreatment methods to improve the hydrolysis rate and digestion efficiency, including ultrasonic, acidic, alkaline and thermal pretreatments (Neyens et al., 2003a, 2003b; Shao et al., 2010; Navia et al., 2002).

Alkaline pretreatment has become a preferred method of sludge pretreatment due to its simple procedure, ease of operation and high efficiency (Weemaes and Verstraete, 1998). Alkaline pretreatment can increase the levels of solubilization by splitting complex polymers into smaller molecules, thereby improving the performance of subsequent AD (Lin et al., 1997, 1999; Vlyssides and Karlis, 2004; Cassini et al., 2006). Recently it has been revealed that hydrolysis of sludge at pH 10.0 (everyday control) could inhibit methanogens and produce more VFA from activated sludge than other pH values and they showed that these soluble carbon compounds of hydrolysis and acidification can be used as carbon sources for biological nutrient removal (Chen et al., 2007; Yuan et al., 2006). Meanwhile, by applying a novel fractionation approach to the use of sludge flocs, Yu et al. (2008, 2010) found that pH 10.0 could improve the VFA production by breaking the sludge matrix and creating effective contact between extracellular organic materials and enzymes.

Most studies conducted to date have investigated the degree of solubilization in response to alkaline pretreatment and the digestibility of alkaline pretreated sludge, or the VFA production of sludge under alkaline conditions. However, few studies have been conducted to investigate the dewaterability of alkaline pretreated sludge after AD, or the mechanism of the effects of alkaline pretreatment on sludge dewaterability after AD. Sludge dewatering is very important to reducing sludge bulk and improving its handling properties. Therefore, dewatering followed by AD is essential in most sludge disposal plants (Appels

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et al., 2008). In this study, sludge flocs were initially pretreated by different pH alkaline pretreatments. During pretreatment, the pH values of sludge were adjusted once, after which they were adjusted to the initial pH values and the sludge was anaerobically digested for 25 days. The variations of organic materials and sludge dewaterability during alkaline pretreatment and AD were all investigated, and the enhancement mechanisms of AD performance and sludge dewaterability after AD were explored.

Extracellular polymeric substances (EPS) in the sludge matrix are produced by secretion, cell lysis, shedding of cell surface material and sorption from the environment (Liu and Fang, 2003). It is generally accepted that AD performance and sludge dewaterability are closely related to the concentrations of proteins and carbohydrates in EPS (Liu and Herbert, 2003; Raszka et al., 2006; Cetin and Erdincler, 2004). To investigate the mechanism by which AD performance is enhanced and sludge dewaterability occurs, an EPS fractionation approach was adopted to investigate the variations of proteins and carbohydrates in each fraction during anaerobic digestion. The sludge flocs were fractioned into slime, loosely bound EPS (LB-EPS), tightly bound EPS (TB-EPS) and pellets (Yu et al., 2007).

1 Materials and methods

1.1 Materials

WAS used in this study was obtained from the aerated tank of a WWTP in Shanghai, China. The plant used the anaerobic-anoxic-oxic process to treat 75,000 m^3/day wastewater (93% domestic sewage and 7% industrial sewage). Sludge was initially concentrated by settling for 2 hr, after which it was screened through a 2-mm sieve. Inoculated granular sludge (IS) for anaerobic digestion was obtained from a mesophilic anaerobic digestion reactor in the laboratory. The characteristics of WAS and IS are shown in Table 1.

1.2 Experimental methods

WAS was first divided into six portions, one for a control and five that were adjusted to pH 8, 9, 10, 11, and 12 by the addition of 4 mol/L NaOH. Alkaline pretreatment was carried out in 1.0 L batch reactors at ambient temperature for 24 hr. Next, 4 mol/L HCl was applied to adjust the pH value to the initial pH value (pH 6.8).

Batch anaerobic digestion tests were conducted using conical flasks with a working volume of 1.5 L. The tested sludge, with or without alkaline pretreatment, was mixed with inoculated sludge at a volume ratio of 10:1 and then put into conical flasks. Oxygen in the conical flasks was removed by nitrogen gas sparging for 60 sec, after which the flasks were capped with rubber stoppers and placed in an incubator (SPX-25013-D, China) at (37.0±0.1)°C. Gas sampling bags were used to collect the gas produced in each reactor. During the AD process, conical flasks were shaken five times per day manually for 10 min each to prevent the sludge from settling. Sampling for chemical analysis was then conducted at different digestion times.

1.3 Analytical methods

The fractionation of sludge flocs was conducted using the ultrasonication-centrifugation method (Yu et al., 2007). Briefly, the sludge samples were centrifuged at 2000 ×*g* for 15 min. The bulk solution was collected as the slime and the part that could be removed by soft centrifugation. The sediments were then re-suspended to the original volumes using buffer solution (pH 7) consisting of Na_3PO_4, NaH_2PO_4, NaCl and KCl with a molar ratio of 2:4:9:1. The conductivities of the buffer were adjusted with deionized water to match the initial sludge samples. The suspensions were centrifuged at 5000 ×*g* for 15 min and the bulk solution was collected as the loosely bound extracellular polymeric substances (LB-EPS). The collected sediments were again re-suspended in buffer solution to the original volumes, after which the samples were subjected to ultrasound at 20 kHz and 480 W for 10 min. Following ultrasound, the suspensions were centrifuged at 20,000×*g* for 20 min. The bulk solution was then collected as the tightly bound EPS (TB-EPS). The residues re-suspended again to the original volumes using buffer solution were called the pellet.

pH was measured using a pH meter (ZD-2, China). Sludge dewaterability was obtained using a capillary suction time (CST) instrument (Model 319, Trition, UK). To measure the dewaterability potential of sludge flocs, the CST values were normalized by dividing them by the TSS concentration. Biogas production was analyzed by the drainage method. The potential biogas production was obtained by dividing the cumulative biogas production by the initial VSS. Soluble COD (SCOD) was analyzed by a COD meter (Hach DRP2000, USA). Carbohydrate was measured by the anthrone colorimetry method, using glucose as the standard (Gaudy, 1962). Soluble protein was determined by the Lowry-Folin method (Frøund et al., 1995). Other parameters, including the volatile suspended solids (VSS) and total suspended solids (TSS), were measured according to the Standard Methods (APHA et al., 1998). All tests were conducted in triplicate.

Table 1 Characteristics of waste activated sludge (WAS) and inoculated sludge (IS)

Sludge	TSS (g/L)	VSS (g/L)	SCOD (mg/L)	CST (sec·L/g-TSS)	pH	Ammonia nitrogen (mg/L)	Conductivity (μS/cm)
WAS	10.6 ± 0.1	7.7 ± 0.3	73 ± 10	9.0 ± 0.3	6.80 ± 0.01	72.3 ± 0.1	1085 ± 10
IS	7.6 ± 0.5	4.8 ± 0.5	2084 ± 15	–	–	–	–

TSS: total suspended solids; VSS: volatile suspended solids; SCOD: soluble COD; CST: capillary suction time.

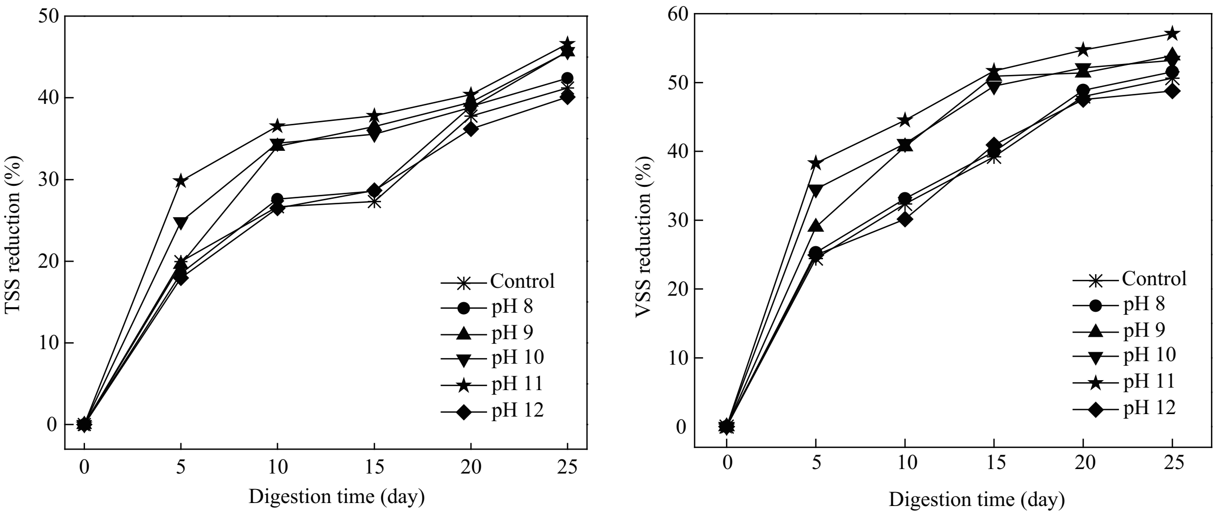

Fig. 1 Variations of TSS and VSS reduction during anaerobic digestion.

2 Results

2.1 Effects of alkaline pretreatment on anaerobic digestion performance

Figure 1 shows the effects of alkaline pretreatment on TSS and VSS reduction during the AD process. After anaerobic digestion, the TSS and VSS reduction for the control were 41.2% and 50.6%, respectively. The digestion efficiency improved when the NaOH dosage increased, with the TSS and VSS reduction reaching 46.6% and 57.1%, respectively, at pH 11. However, when compared with the control, digestion efficiency was remarkably lower at pH 12, indicating that extreme pH was detrimental to the subsequent AD.

The digestive rate was found to be much higher in the first 5 days. As shown in Fig. 1, the TSS and VSS reduction of the first 5 days accounted for 64.0% and 67.0% of the total reduction at pH 11. At other pH values, these numbers were all above 40%.

2.2 Effect of alkaline pretreatment on biogas production

Biogas production was also affected by pretreatment pH at 8.0–12.0 (Fig. 2). As shown in Fig. 2, biogas production increased as the alkaline dose increased, reaching 476.0 mL/g-VSS at pH 10 under ambient temperature and pressure. This represents a 15.4% improvement when compared with the control; however, biogas production decreased with further increasing alkalinity. Specifically, the biogas production at pH 12 decreased by 18.1% when compared with the control. Taken together, these finding indicate that the optimal pH for biogas production was 10.

2.3 Effect of alkaline pretreatment on organic matter during AD

It has been reported that proteins and carbohydrates are the main components of EPS of sludge (Dignac et al., 1998); accordingly, changes in their concentrations and distributions will influence sludge dewaterability and many other properties. As shown in Fig. 3, the organic matters were mainly distributed in the TB and pellet fractions in each group. Specifically, 97.9% of proteins and 97.2% of carbohydrates were distributed in the inner fractions (TB-EPS and pellet) for the control group, while only 73.3% of proteins and 91.6% of carbohydrates were distributed in these fractions for the pH 12 group. In addition, 81.0%–97.8% of proteins and 94.5%–97.1% of carbohydrates were distributed in the TB-EPS and pellet fractions for other groups. The different distribution patterns of organic matters in different groups indicated that alkaline pretreatment could make organic matters release from inner fractions (TB-EPS and pellet) to outer fractions (slime and LB-EPS), thus increasing the hydrolysis rate of granular organics and improving digestion efficiency. The concentrations of protein and carbohydrate in EPS in the six groups decreased 29.9%–37.8% and 48.3%–58.6%, respectively, after AD. Yu et al. (2010) observed similar results in an investigation of anaerobic fermentation of excess sludge at pH 5.5 and pH 10. A slower decrease of protein and carbohydrate concentrations was observed at pH 12, which might have been due to poor biodegradability with extreme pH pretreatment. As shown in Fig. 2, the biogas production at pH 12 decreased.

Protein and carbohydrate concentrations increased in

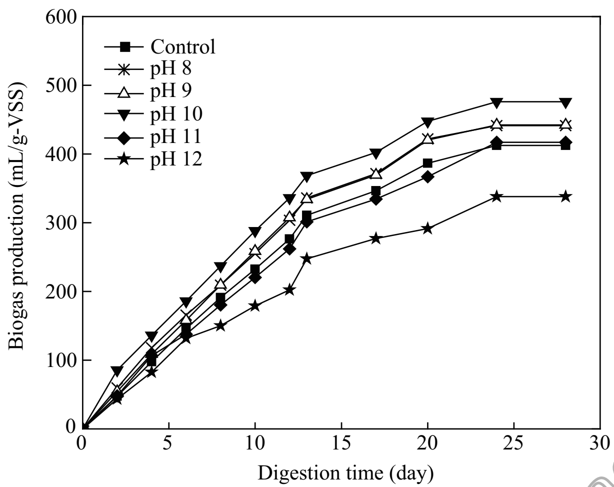

Fig. 2 Variations of biogas production during anaerobic digestion.

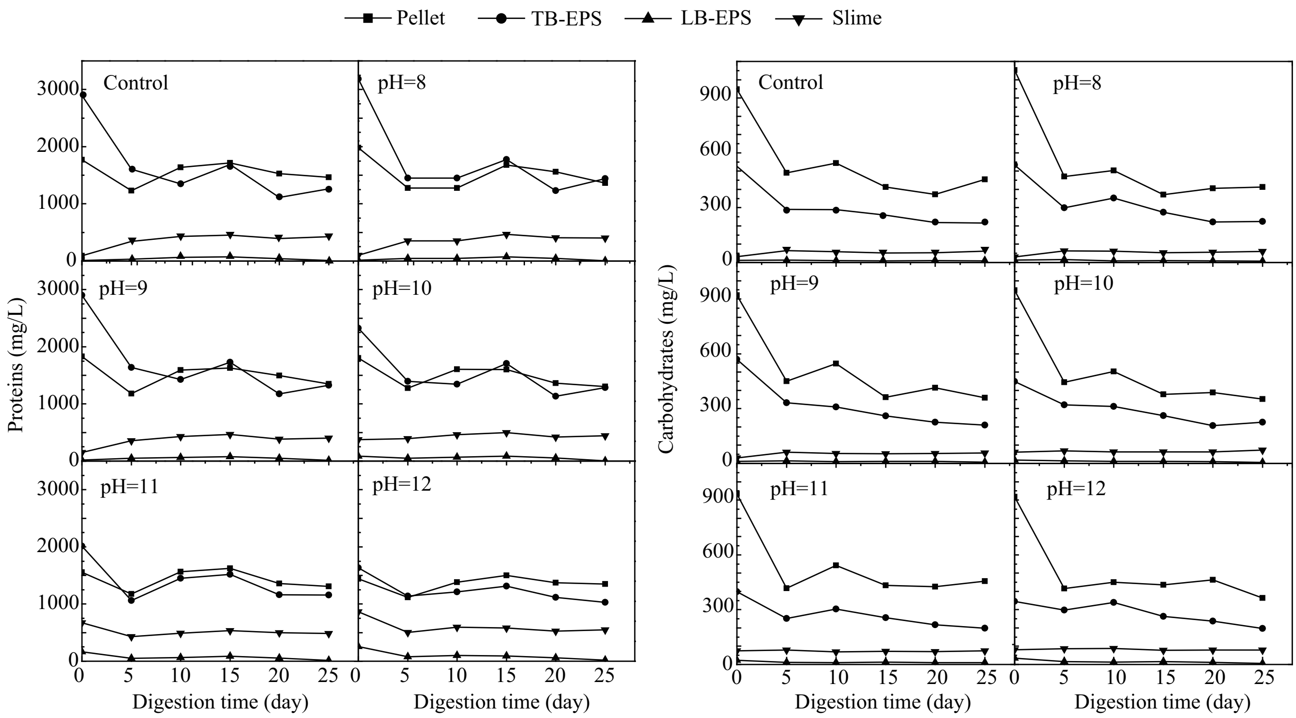

Fig. 3 Variations of proteins, and carbohydrates during anaerobic digestion. TB-EPS: tightly bound extracellular polymeric substances (EPS); LB-EPS: loosely bound EPS.

slime fractions, while they decreased in pellet and TB-EPS fractions when pretreated with low pH levels (pH 8, pH 9 and the control). These findings indicate that proteins and carbohydrates were transferred from tightly bound fractions to outer fractions in the AD process. Decreased protein and carbohydrate concentrations were observed in all fractions at pH 11 and pH 12, which might be due to pretreatment with highly alkaline doses leading to releasing inner organic matters, and these groups had high initial protein and carbohydrate concentrations in slime and LB-EPS fractions. In the AD process, when the biodegradation rate in slime and LB-EPS fractions exceeded the release from inner fractions, the concentrations in slime and LB-EPS fractions decreased.

In addition, the protein and carbohydrate concentrations significantly decreased in the TB-EPS and pellet fractions during the first five days, which showed that the highest biodegradation rate was obtained during this period. As shown in Figs. 1 and 2, the TSS and VSS reduction and biogas production rate were high during this period.

2.4 Effects of alkaline pretreatment on normalized CST

Sludge dewaterability was indicated by the CST values. Variations of normalized CST during anaerobic digestion are shown in Fig. 4. As shown in Fig. 4, normalized CST showed few changes at pH $\leqslant$ 9 after alkaline pretreatment, but increased rapidly when pH increased, with a value of 163.5 (sec·L)/g-TSS being observed at pH 12 (17 times that of raw sludge). Normalized CST increased rapidly during the first 5 days and underwent small fluctuations when the AD process continued. At the end of the AD process, the normalized CST was smaller than that of the control (142.7 sec·L/g-TSS) at pH $\leqslant$ 9, but higher than the control at pH > 9. These findings indicate that

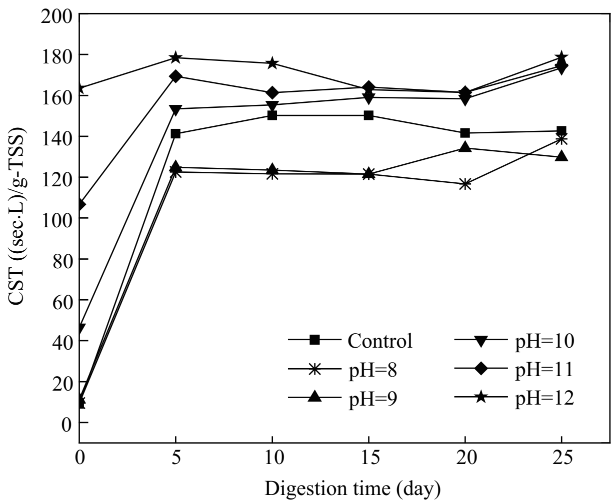

Fig. 4 Variations of normalized capillary suction time (CST) during anaerobic digestion.

dewaterability can be improved by pH 8 and pH 9, but that it deteriorates when pH pretreatment is conducted above 9. In conclusion, sludge digestibility and dewaterability could both be improved at pH 9.

3 Discussion

3.1 Mechanism of improved sludge digestibility in response to alkaline pretreatment

The effects of alkaline pretreatment on the distribution of initial organic materials in the sludge matrix are shown in Fig. 5. Proteins and carbohydrates were mainly distributed in the pellet and TB-EPS fractions in the control group, and experienced similar variations when pH increased. Specifically, they increased in the slime and LB-EPS fractions

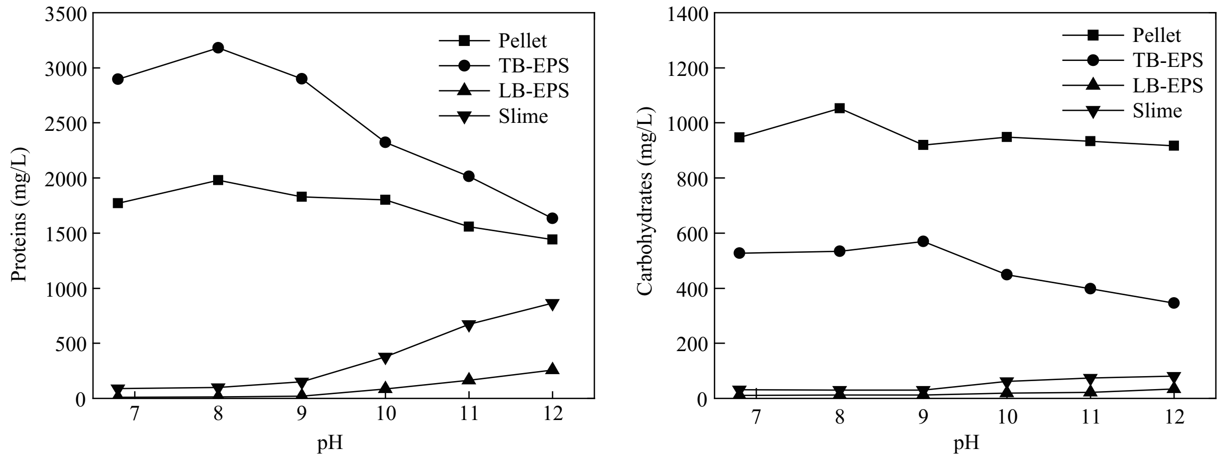

Fig. 5 Effect of alkaline pretreatment on the distribution of initial organic matters in the sludge matrix protein and carbohydrate.

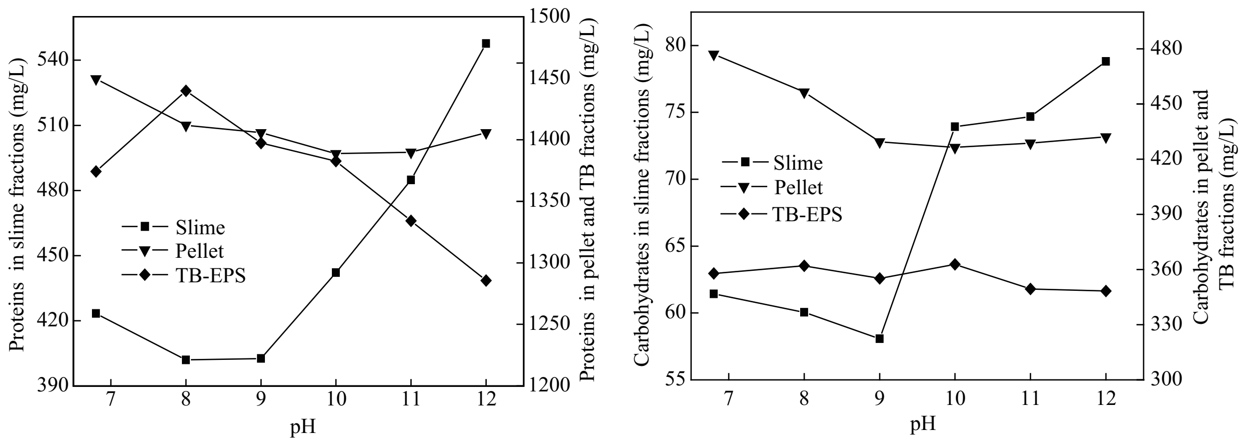

Fig. 6 Distribution of organic matters in the sludge matrix after AD.

and decreased in the TB-EPS and pellet fractions. The results demonstrated that organic materials were released from the inner fractions to the outer fractions, which led to an increase of dissolved organic materials. The results of this study also showed that, as the pH increased from 6.8 to 12.0, the SCOD increased in a parabolic pattern ($y_{SCOD} = 201\times pH^2 - 2891\times pH + 10568$, $R^2 = 0.9709$). Kim et al. (2010) investigated the effects of combined (alkaline + ultrasonic) pretreatment on sewage sludge disintegration and concluded that the SCOD linearly increased as pH increased. The above results revealed that alkaline pretreatment can disrupt flocs and cells of microorganisms in the sludge, release inner organic materials into soluble organic materials and increase the possibility of contact between microbes and organic materials, thereby improving subsequent anaerobic digestibility.

Dissolved organic materials increased after alkaline pretreatment and were then converted to biogas after acidification and methanization. As a result, cumulative biogas production increased when compared with the control. The improvement in biogas production supports the idea that proper alkaline pretreatment could improve biogas production. However, the gas production efficiency was not further improved when the pH increased. Indeed, as shown in Fig. 2, biogas production was inhibited at pH $\geqslant$ 11. The results also revealed that the TSS and VSS reduction were inhibited when sludge was pretreated at pH 12. Similar problems have been reported in other studies when investigating the influence of sodium hydroxide addition on solubilization and anaerobic biodegradability (Penaud et al., 1999) and thermochemical pretreatment of substrates at extreme pH values (Patel et al., 1993). It is believed that these results occur because there are some intermolecular reactions between simple compounds pretreated under extreme conditions, which leads to the formation of complex substances with low degradability, thereby leading to decreased biogas production and digestion performance. Penaud et al. (1999) reported that the biodegradability of samples decreased when they were pretreated with excess alkaline dosages, and that this was not caused by the presence of excess sodium cations, but rather by the formation of refractory compounds due to the Maillard reaction. Maillard reactions are complex chemical reactions between amino groups and reducing sugars (Lan et al., 2010). Many Lignin-like macromolecular pigments and melanoidins form in such reactions, and these compounds are considered to be indigestible (Sniffen et al., 1992; Soest and Mason, 1991).

3.2 Factors influencing sludge dewaterability after alkaline pretreatment

It has been widely reported that the amount of protein and carbohydrate in EPS influence the sludge dewaterability. Many researchers have found that the effects of protein

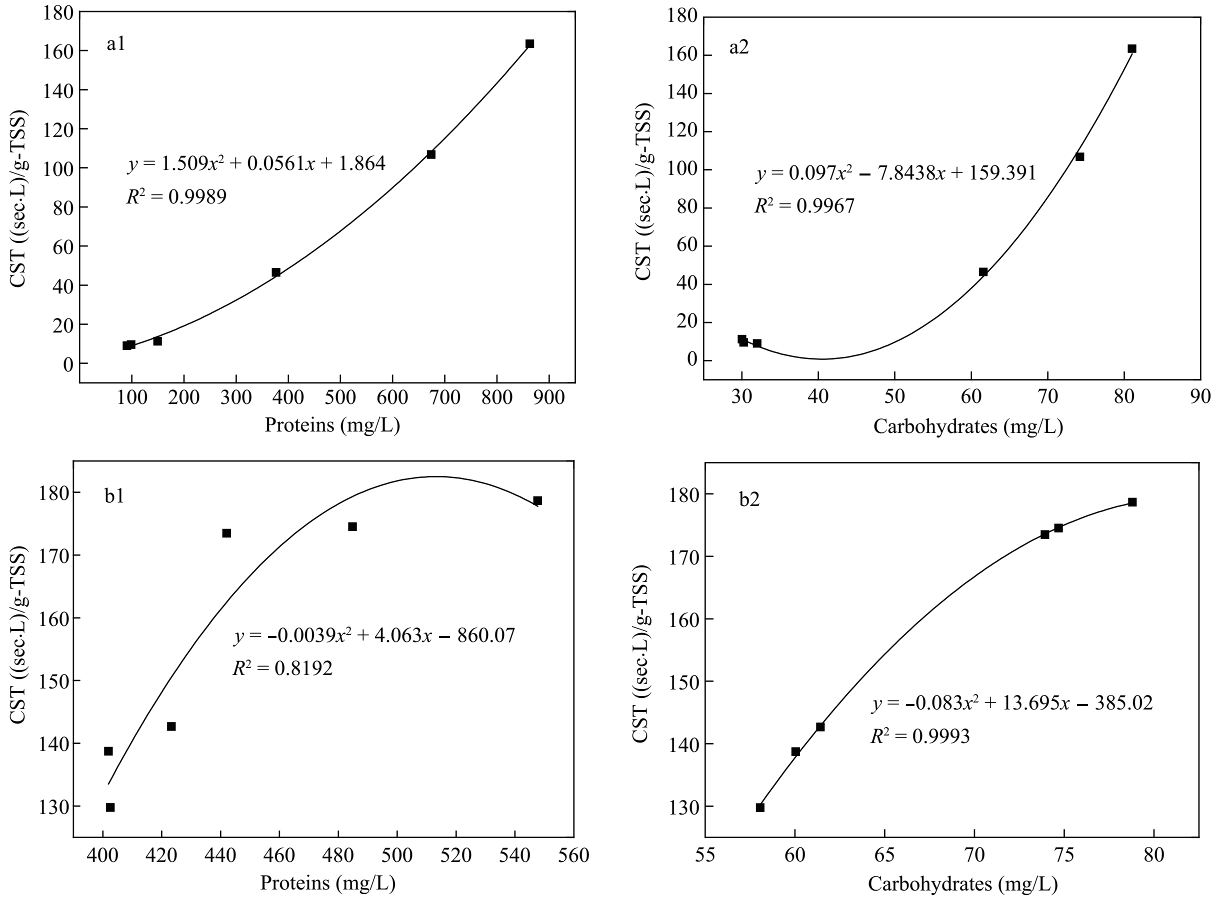

Fig. 7 Relationship between CST and organic matters in slime fractions after alkaline pretreatment (a) and after AD process (b).

were positive, other researchers found them negative. However, the effects of carbohydrate are always reported to be negative (Higgins and Novak, 1997; Jin et al., 2004; Murthy and Novak, 1999).

The distribution of organic materials changed when the AD process proceeded, as indicated in Fig. 3. As shown in Fig. 6, with the increase of pH, the concentrations of protein and carbohydrate after AD in slime fractions had a rising trend, while the concentrations of protein and carbohydrate in pellet fractions had a downward trend. The relationship between normalized CST and organic materials in slime fractions after alkaline pretreatment and after the AD process are shown in Fig. 7. The results revealed that the normalized CST increased when the concentrations of protein and carbohydrate in slime fractions increased.

The Pearson correlation between the normalized CST and proteins and carbohydrates in different sludge fractions during the AD process are shown in Table 2. The normalized CST was clearly positively correlated with protein and carbohydrate concentrations in slime fractions ($R^2 \geqslant 0.700$, $p < 0.01$), and negatively correlated in TB-EPS and pellet fractions ($R^2 \geqslant 0.702$, $p < 0.01$). Poxon and Darby (1997) studied the sludge dewaterability and found that the specific components of EPS were more important than the amounts of EPS with respect to their effects on sludge dewaterability, and that the soluble components of EPS could deteriorate sludge dewaterability. The dissolved organic materials in slime fractions increased markedly in response to alkaline solubilization after 24 hr of alkaline pretreatment and this effect increased as pH increased (Fig. 5), which led to the deteriorated sludge dewaterability in the beginning of the anaerobic digestion period. The dissolved organic matters degraded at pH 8 and pH 9 during the AD process. However, the formation of less biodegradable components at pH > 10 resulted in the accumulation of dissolved organic materials in the slime fractions. This accumulation in the slime fractions led to poor dewaterability at pH > 10 after the AD process.

Table 2 Pearson correlation between normalized CST and protein and carbohydrate in different sludge fractions during AD process (n = 36)

EPS	Normalized CST		
	Protein	Carbohydrate	Protein + Carbohydrate
Slime	0.700**	0.811**	0.722**
LB-EPS	0.180	–0.097	0.155
TB-EPS	–0.906**	–0.826**	–0.906**
Pellet	–0.702**	–0.725**	–0.820**

* Significance level is 0.05, ** significance level is 0.01.

In conclusion, variations in sludge dewaterability result from different distributions of proteins and carbohydrates in flocs after alkaline pretreatment. Sludge dewaterability can be improved by alkaline pretreatment at pH 8 and pH 9, and deteriorated by alkaline pretreatment at pH 10, pH 11 and pH 12. The deteriorated dewaterability is caused by the accumulation of organic matters in slime fractions. Alkaline pretreatment at pH 9 is preferable for sludge dewaterability. Based on the results of Pearson correlation, sludge dewaterability can be improved by reducing the accumulation of soluble organic materials in outer fractions or reducing the release of macromolecular materials to outer fractions.

4 Conclusions

Appropriate pH pretreatment can increase sludge reduction and biogas production by 10% and effectively improve sludge dewaterability. Organic materials are transferred from inner fractions to outer fractions after alkaline pretreatment, and this effect increases with increased pH. Alkaline pretreatment increases the possibility of contact between microbes and organic materials and improves subsequent AD efficiency. Normalized CST is positively correlated with protein and carbohydrate concentrations in slime fractions and negatively correlated in the TB-EPS and pellet fractions. Once the soluble proteins and carbohydrates in slime fractions increase, sludge dewaterability deteriorates.

Acknowledgments

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