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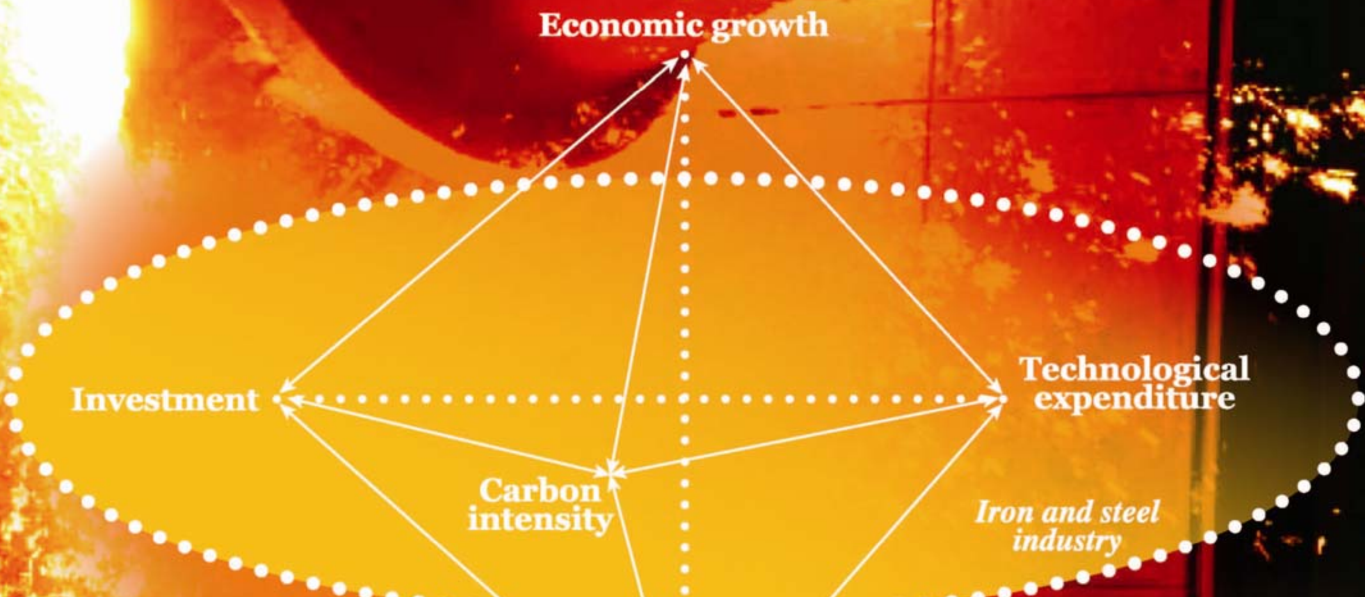

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Feasibility of bioleaching combined with Fenton oxidation to improve sewage sludge dewaterability

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ABSTRACT

A novel joint method of bioleaching with Fenton oxidation was applied to condition sewage sludge. The specific resistance to filtration (SRF) and moisture of sludge cake (MSC) were adopted to evaluate the improvement of sludge dewaterability. After 2-day bioleaching, the sludge pH dropped to about 2.5 which satisfied the acidic condition for Fenton oxidation. Meanwhile, the SRF declined from 6.45×10^{10} to 2.07×10^{10} s^2/g, and MSC decreased from 91.42% to 87.66%. The bioleached sludge was further conditioned with Fenton oxidation. From an economical point of view, the optimal dosages of H_2O_2 and Fe^{2+} were 0.12 and 0.036 mol/L, respectively, and the optimal reaction time was 60 min. Under optimal conditions, SRF, volatile solids reduction, and MSC were 3.43×10^8 s^2/g, 36.93%, and 79.58%, respectively. The stability and settleability of sewage sludge were both improved significantly. Besides, the results indicated that bioleaching-Fenton oxidation was more efficient in dewatering the sewage sludge than traditional Fenton oxidation. The sludge conditioning mechanisms by bioleaching-Fenton oxidation might mainly include the flocculation effects and the releases of extracellular polymeric substances–bound water and intercellular water.

Introduction

Activated sludge process plays an important role in worldwide wastewater treatment (Chang et al., 2001), but it has a serious drawback of producing huge amounts of excess sludge (Feng et al., 2009). The water content of excess sludge is generally over 98%, which leads to the difficult dewatering (Vaxelaire and Cezac, 2004). Therefore, the treatment of excess sludge has already become a serious environmental problem in wastewater treatment plants. It has been reported that the performance of sludge dewatering significantly depends on sludge properties, such as particle size, extracellular polymeric substances (EPS), water content, etc. (Karr and Keinath, 1978; Mikkelsen and Keiding, 2002; Neyens and Baeyens, 2003; Novak et al., 1998). Sludge dewatering has been pointed out as the most expensive and the least understood process (Bruus et al., 1992), and the cost of sludge treatment and disposal nearly accounts for as high as 50%–60% of the entire operating cost of wastewater treatment plants (Egemen et al., 2001). With the development of stringent environmental regulations, more efficient sludge treatment technologies are demanded.

Advanced oxidation processes for sludge conditioning have gained the worldwide attention in recent years (Tony et al., 2009). Fenton oxidation as one of the advanced oxidation processes has

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been proved to be a promising technology for conditioning sludge. During the last decade, many efforts have been made to explore the possibility of sludge dewatering by Fenton oxidation, and the experimental results indicated that Fenton oxidation had a positive effect on sludge dewatering (Buyukkamaci, 2004; Debowski et al., 2008; Erden and Filibeli, 2010; Kaynak and Filibeli, 2008; Lu et al., 2003). In addition, it was reported that Fenton pretreatment played a positive role in sludge minimization (Kaynak and Filibeli, 2008). Through Fenton pretreatment, the anaerobic biodegradability of biological sludge was also improved significantly (Erden and Filibeli, 2010; Kaynak and Filibeli, 2008). As a result, the higher volatile solid (VS) reduction and higher biogas production were achieved (Kaynak and Filibeli, 2008).

Traditionally, inorganic acid is always needed in Fenton reaction to reduce sludge pH to achieve the desired efficiency. In that case, large amounts of inorganic acid for sludge conditioning and further alkali for neutralizing are required, which leads to high operation cost of Fenton treatment. Bioleaching as one of the microbial technologies for sludge treatment may serve as a substitution method of conventional chemical acidification, because the sludge pH can decline to the optimal pH range for Fenton reaction through bioleaching. In addition, it is widely accepted that bioleaching is superior in leaching heavy metals (Benmoussa et al., 1997; Couillard and Mercier, 1991; Kim et al., 2005), destroying and destructing pathogens (Benmoussa et al., 1997; Couillard and Mercier, 1991), controlling odor (Filali-Meknassi et al., 2000), reducing volume and improving stability (Benmoussa et al., 1997). Therefore, bioleaching has gained increasing attention to sludge conditioning in recent years.

In our previous studies, bioleaching combined with Fenton-like oxidation was proved to be efficient in removing heavy metals from sewage sludge (Zhu et al., 2013). In this study, we continuously investigated the possibility of bioleaching combined with Fenton oxidation to improve sludge dewaterability. The specific resistance to filtration (SRF), moisture of sludge cake (MSC), supernatant volume, and VS reduction were adopted to characterize the treated sludge. Bioleaching provides a suitable reaction condition for Fenton oxidation, which has been scarcely reported. The main objective of this study was to evaluate the feasibility and efficiency of the combined process for sludge conditioning and dewatering.

1. Materials and methods

1.1. Sewage sludge

Sewage sludge was collected from sludge thickener of a full-scale wastewater treatment plant in Changsha, China. After gravity settling for 12 hr, the supernatant was removed, and then the sludge as experimental sample was stored at 4°C in a refrigerator. Before conditioning experiments, the sludge sample was kept in a water bath at 20°C for 30 min. Some properties of raw sludge are given in Table 1.

All chemicals used in this study were of analytic grade, and purchased from Sinopharm Chemical Reagent Co. Ltd.

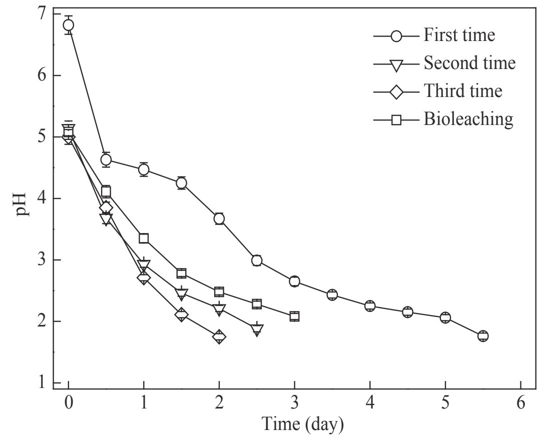

Fig. 1 – pH change during inocula preparation and sludge bioleaching process. Experimental condition: sulfur powder dosage = 0.31 mol/L for inocula preparation, sulfur powder dosage = 0.09 mol/L for sludge bioleaching.

1.2. Inocula preparation

It has been reported that pure *Thiobacillus* for bioleaching could be isolated from sewage sludge or acid wastewater (Wong et al., 2004). Thus, fresh sewage sludge as the seed sludge was applied to enrich and culture the indigenous acidophilic *Thiobacillus*, which was collected from the sludge thickener in the same wastewater treatment plant. All experiments were performed at ambient temperature of 28°C. Inocula preparation was described in detail as follows. Firstly, sulfur powders of 0.31 mol/L as the energy substance were added into a 250 mL Erlenmeyer flask (Zhengzhou Zhongtian Chemical Instrument Co., Ltd., Zhengzhou, China) filled with feed sludge of 100 mL. Then the flask was agitated in an orbital shaker (ZHWY-1102, Shanghai Zhicheng Analytical Instrument Co., Ltd., Shanghai, China) at a shaking speed of 180 r/min until the pH of seed sludge dropped to below 2.0. Subsequently, the acidified sludge of 10 mL was transferred into a 250 mL Erlenmeyer flask filled with 90 mL feed sludge, under the same conditions the *Thiobacillus* were enriched and cultured twice again. After being cultured and enriched

Table 1 – Properties of raw sludge and bioleached sludge.

Sludge	pH	TS (mg/L)	VS (mg/L)	Supernatant volume (mL)	MSC (%)	SRF (s^2/g)
Sludge sample	6.83	15461	10248	2.0	91.42	6.45×10^{10}
Bioleached sludge	2.23	14,503	9449	3.0	87.66	2.07×10^{10}

TS, VS, MSC, and SRF denote total solids, volatile solids, moisture of sludge cake, and specific resistance to filtration, respectively.

for three times, the acidified sludge could serve as inocula for sludge bioleaching, of which the *Thiobacillus* activity was strongly strengthened (Zhang et al., 2009). The whole period of inocula preparation was about 10 days as shown in Fig. 1. During this process, the loss of evaporated water was compensated by adding distilled water.

1.3. Sludge bioleaching and Fenton oxidation

The inocula of 15 mL were added into a 250 mL Erlenmeyer flask filled with 135 mL raw sludge, and the flask was agitated in an orbital shaker at a shaking speed of 180 r/min for bioleaching. During bioleaching process, the loss of evaporated water was compensated by adding distilled water. After finishing bioleaching process, different dosages of H_2O_2 and Fe^{2+} were added into 150 mL bioleached sludge. Subsequently, the sludge was rapidly mixed at 200 r/min for 5 min, followed by slowly stirring at 50 r/min for a certain time. The pH of the traditional Fenton oxidation was adjusted by sulfuric acid.

1.4. Analytical procedures

The SRF was determined using Buchner funnel method. 100 mL sludge was poured into a Buchner funnel (Zhengzhou Zhongtian Chemical Instrument Co., Ltd., Zhengzhou, China) to filter at a vacuum pressure of 0.03 MPa. The SRF is calculated in accordance with Eq. (1) (Buyukkamaci, 2004; Lu et al., 2003):

$$\mathrm{SRF} = \frac{2bA^2P}{\mu C} \tag{1}$$

where, SRF (s^2/g) is the specific resistance to filtration, P (g/cm) is the filtration pressure, A (cm^2) is the filter area, μ (g/(cm·s)) is the viscosity of filtrate, b (s/cm) is the slope of filtrate discharge curve, and C (g/cm) is the weight of cake solids per unit volume of filtrate.

Sludge cakes produced by centrifugal process at 6000 r/min for 5 min in a batch laboratory centrifuge (LDZ4, Changzhou Wanhe Instrument Manufacture Co., Ltd., Changzhou, China) were dried in an oven at 105°C to determine the moisture. Sludge settleability was determined by recording the supernatant volume of 100 mL sludge after gravity settling for 30 min. The viscosity of filtrate was determined by a rotary viscometer (NDJ-1, Shanghai Pingxuan Scientific Instrument Co., Ltd., Shanghai, China).

2. Results and discussion

2.1. Sludge bioleaching

Sulfur powder always has a low utilization rate during bioleaching process (Ravishankar et al., 1994) and the rest sulfur would cause the "later acidification" effect (Chen et al., 2003). Thus, a relatively low dosage of 0.09 mol/L was adopted as energy substance in this study, which was reported to be enough for sludge bioleaching (Chen et al., 2004). As shown in Fig. 1, it took 2 days to drop the pH below 2.5. Some properties of bioleached sludge are given in Table 1. Compared with raw sludge, the dewaterabilty was improved by the bioleaching. The SRF declined from 6.45×10^{10} to 2.07×10^{10} s^2/g with a reduction of 70%, and MSC decreased by 3.76%. Besides, the VS reduced by 7.8% after sludge bioleaching, which may be contributed from the metabolism of acidophilic *Thiobacillus* and the acidic circumstance developed in the sludge (Pathak et al., 2009).

2.2. Fenton oxidation

It has been reported that sewage sludge treated by bioleaching with a pH below 2 is more difficult to be dewatered than that with a higher pH (Xiao et al., 2010). This problem could be overcome by keeping the sludge pH between 2 and 3 (Pathak et al., 2009). In addition, it was reported that the optimal pH of Fenton reaction for sewage sludge condition was around 2.5 (Lu et al., 2001; Neyens and Baeyens, 2003). Therefore, it was considered that the bioleaching process was finished in this study when the sludge pH dropped to about 2.5.

2.2.1. Effects of H_2O_2 and Fe^{2+} dosages

The Fenton process causes the formation of highly reactive hydroxyl radicals (OH·) that attack and destroy organic matters (Neyens et al., 2004), and the amount of H_2O_2 directly influences the production of OH. which plays an important role in sludge dewatering (Lu et al., 2003). As shown in Fig. **2**, the lower SRF and MSC indicated the higher sludge dewaterability. At the beginning, the SRF decreased obviously with the increase in H_2O_2 dosage, but later there was no significant change. The minimum SRF and MSC were both achieved at the H_2O_2 dosage of 0.24 mol/L, with a value of 2.97×10^8 s^2/g and 78.58%, respectively. In China, sewage sludge with a SRF lower than 4.0×10^8 s^2/g is recommended as easy-dewatering sludge by Ministry of Environmental Protection of China (2006). Thus, the sludge became easy to be dewatered at the H_2O_2 dosage of 0.12 mol/L. The SRF and moisture of sludge cake were 3.56×10^8 s^2/g and 79.68%, respectively. Although the sludge dewaterability was improved by increasing the amount of H_2O_2, from an economical point of view, the suitable H_2O_2 dosage was 0.12 mol/L.

With a H_2O_2/Fe^{2+} (mol/mol) ratio of 3.3 obtained in Fig. 2, the effect of different dosages of H_2O_2 and Fe^{2+} on sludge

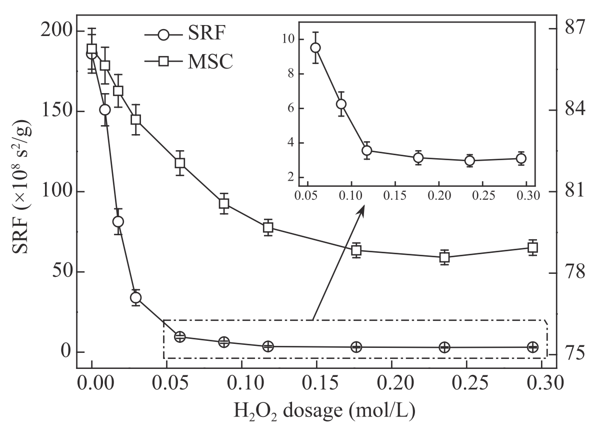

Fig. 2 – Effect of H_2O_2 dosage on sludge dewatering. Experimental condition: pH = 2.20 ± 0.1, reaction time = 60 min, Fe^{2+} dosage = 0.036 mol/L.

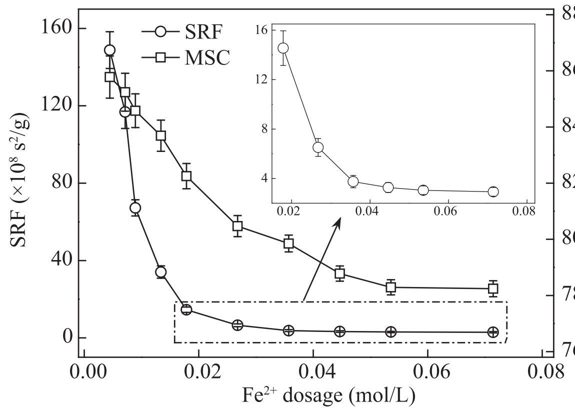

Fig. 3 – Effect of H_2O_2 and Fe^{2+} dosages on sludge dewatering with a H_2O_2/Fe^{2+} (mol/mol) ratio of 3.3. Experimental condition: pH = 2.25 ± 0.1, reaction time = 60 min.

dewaterability was investigated. As illustrated in Fig. 3, the SRF and MSC both decreased with the increase in the dosage of Fe^{2+}. Thus, sludge dewaterability was improved by increasing the dosages of H_2O_2 and Fe^{2+} with a H_2O_2/Fe^{2+} (mol/mol) ratio of 3.3. The larger the H_2O_2 and Fe^{2+} dosage, the higher the sludge dewaterability. But comparing the SRF from an economical point of view, the appropriate dosages of H_2O_2 and Fe^{2+} were 0.12 and 0.036 mol/L, respectively. Under this condition, sludge became easy to be dewatered.

2.2.2. Effects of reaction time

The influence of reaction time on the dewaterability of bioleached sludge is shown in Fig. 4. The results indicated that the sludge dewaterability was greatly improved at the beginning of the reaction, and later there were no significant change of the SRF and MSC. At 60 min, the SRF and MSC were 3.43×10^8 s^2/g and 79.58%, respectively, which indicated that sludge was easy to be dewatered. Thus, the optimal reaction time was selected to be 60 min.

2.3. Sludge settleability

In this study, supernatant volume after gravity settling for 30 min was selected to characterize the sludge settleability

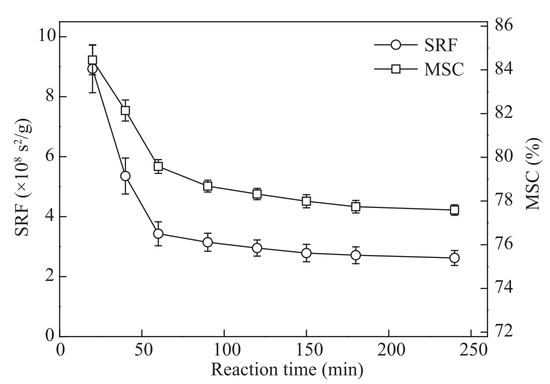

Fig. 4 – Effect of reaction time on sludge dewatering. Experimental condition: pH = 2.30 ± 0.1, Fe^{2+} dosage = 0.036 mol/L, H_2O_2 dosage = 0.12 mol/L.

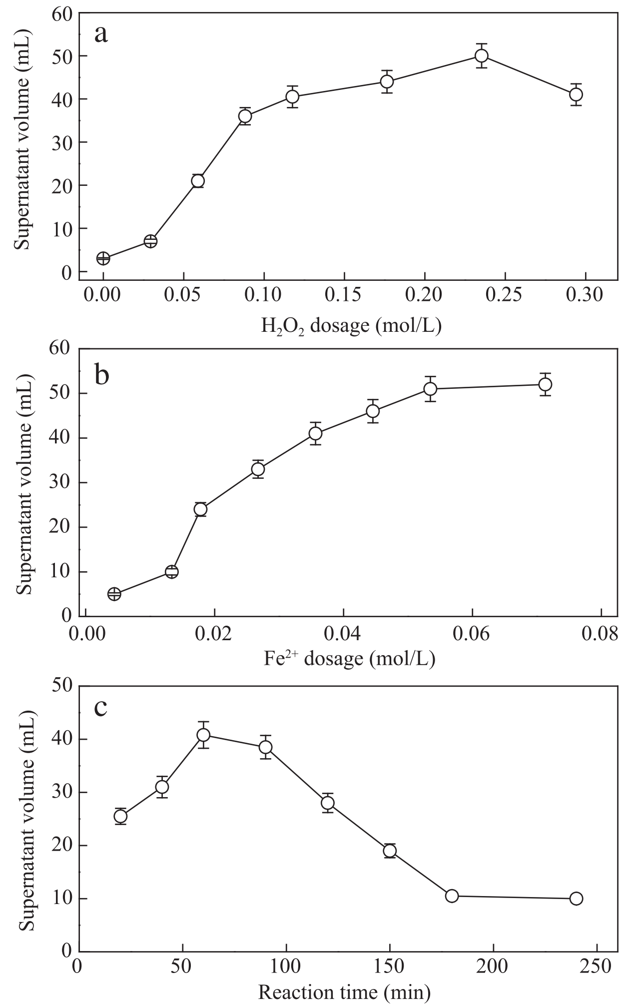

Fig. 5 – Supernatant volume change of sludge treated by bioleaching-Fenton oxidation. Experimental conditions of a, b, and c are the same as Figs. 2, 3, and 4, respectively.

after treatment. Fig. 5 shows the settleability of sludge treated by bioleaching-Fenton oxidation. The sludge settleability was enhanced with the increase of H_2O_2 dosage shown in Fig. 5a. With a H_2O_2/Fe^{2+} (mol/mol) ratio of 3.3, the settleability was improved by increasing the dosages of H_2O_2 and Fe^{2+} depicted in Fig. 5b. As illustrated in Fig. 5c, the settleability of sludge was improved rapidly at the beginning of the Fenton reaction, and the supernatant volume reached the maximum volume at 60 min, and later the supernatant volume appeared in a decreasing trend, which may be due to the particle size reduction along with the reaction time. Through the bioleaching-Fenton oxidation, sludge settleability was significantly improved.

2.4. Comparison for Fenton and bioleaching-Fenton oxidation

The effect of Fenton oxidation and bioleaching-Fenton oxidation on sludge dewatering was investigated. Some properties of experimental sludge treated respectively by Fenton (pH adjusted by sulfuric acid) and bioleaching-Fenton oxidation are given in Table 2. The dewaterability of sludge treated by

Table 2 – Properties of experimental sludge treated by Fenton oxidation and bioleaching-Fenton oxidation.

Treatment method	pH	VS reduction (%)	Supernatant volume (mL)	MSC (%)	SRF (s^2/g)
Fenton oxidation	2.24	18.68	15.0	83.24	4.26×10^9
Bioleaching-Fenton oxidation	2.18	36.93	41.0	79.85	3.75×10^8

Reaction time = 60 min, Fe^{2+} dosage = 0.036 mol/L, H_2O_2 dosage = 0.12 mol/L; the sludge pH was adjusted by sulfuric acid for Fenton oxidation.

bioleaching-Fenton oxidation (SRF of 3.75×10^8 s^2/g) was much higher than that of sludge treated by Fenton oxidation (SRF of 4.26×10^9 s^2/g). It was reported that VS reduction of sewage sludge was an indication of the stabilization (Pathak et al., 2009), the higher the VS reduction, the better the sludge stability. Thus, the sludge treated by bioleaching-Fenton oxidation had a better stability with a VS reduction almost twice as much as that of sludge treated by Fenton oxidation. Besides, the settleability of sludge treated by bioleaching-Fenton oxidation was much better. Therefore, the bioleaching-Fenton oxidation was more efficient than the Fenton oxidation for sludge dewatering, which might result from the bioleaching pretreatment assimilating the sludge and providing a better reaction condition for Fenton oxidation.

2.5. *Possible mechanisms of dewaterability*

The VS is a sum parameter for the content of organic matters in sludge (Mundhenke et al., 2001). The relationship between the dewaterability and VS reduction is shown in Fig. 6. The SRF and MSC decreased with the increase in VS reduction, the higher the VS reduction, the better the sludge dewaterability. Neyens and Baeyens (2003) considered that the difficulty in sludge dewatering mainly resulted from the presence of EPS, which accounts for up to 80% of the total sludge mass (Frolund et al., 1996). The VS reduction was up to 36.93% under the optimal conditions of bioleaching-Fenton oxidation, which meant that the EPS might be effectively degraded. The EPS degradation reduces its water retention property thereby releasing the EPS-bound water and improving sludge dewatering (Neyens et al., 2004). Additionally, the microbial cells would be more easily destructed through the bioleaching-Fenton oxidation due to microbial cells losing protection provided by the EPS (Houghton et al., 2001), and the destruction of microbial cells could make the intercellular water release and improve the sludge dewatering (Lu et al., 2003).

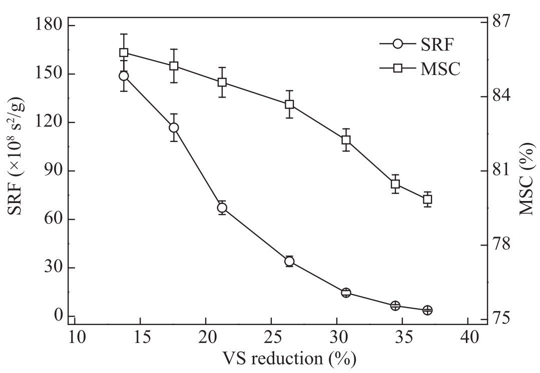

Fig. 6 – Relationship between sludge dewaterability and VS reduction during bioleaching-Fenton oxidation process. Experimental condition: pH = 2.25 ± 0.1, reaction time = 60 min, H_2O_2/Fe^{2+} (mol/mol) ratio = 3.3.

Under the low pH condition created by sludge bioleaching, the flocculation of sludge would be improved, because the electrostatic repulsive interactions are minimized at low pH so that the dissociation constants of sludge flocs are minimum (Neyens et al., 2004). The similar reports about sludge dewatering by bioleaching were represented in the previous studies (Pathak et al., 2009; Xiao et al., 2010). In addition, the ferric/ferrous ions were effective flocculants in sludge conditioning (Oikonomidis et al., 2010). As shown in Fig. 3, the SRF declined by increasing the Fe^{2+} dosage. The results were in good agreement with that obtained by Lu et al. (2003).

Therefore, the main mechanisms of sludge conditioning by bioleaching-Fenton oxidation might include the release of EPS-bound water and intercellular water resulting from the degradation of the organic matters and destructions of microbial cells, and the flocculation effect contributed from the low pH circumstance and ferrous/ferric ions. However, the more detailed mechanisms need to be further explored.

3. Conclusions

The dewaterability of sewage sludge treated by bioleaching-Fenton oxidation was investigated in this study. The results showed that the sludge bioleaching created a suitable condition for Fenton reaction, and the dewaterability of sludge was significantly improved by bioleaching-Fenton reagent. From an economical point of view, the optimal dosages of H_2O_2 and Fe^{2+} were respectively accepted as 0.12 and 0.036 mol/L, and the optimum reaction time was 60 min. Under optimal conditions, SRF, VS reduction, and MSC were 3.43×10^8 s^2/g, 36.93%, and 79.58%, respectively. Additionally, the bioleaching-Fenton oxidation was superior in sludge dewatering than Fenton oxidation. The bioleaching-Fenton oxidation not only significantly improved the sludge dewaterabilty, but also efficiently enhanced the sludge stability. The improvement of sludge dewaterability might mainly result from the release of EPS–bound water and intercellular water, and the flocculation effect.

Acknowledgments

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