Effect of potassium ferrate \((K_2FeO_4)\) on sludge dewaterability under different pH conditions

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**Highlights**

- The SRF values decreased with the decrease of sludge pH value pretreated by \(K_2FeO_4\).
- \(K_2FeO_4 = 1200\) mg/L at pH 3 is an optimal condition for improving sludge dewatering.
- EPSs content and sludge disintegration degree increased pretreated by \(K_2FeO_4\) at pH 3.
- EPSs content and sludge disintegration degree were positively correlated with SRF.

**Article Info**

Article history:
Received 20 May 2012
Received in revised form 27 August 2012
Accepted 10 September 2012
Available online 17 September 2012

**Keywords:**
Sludge dewaterability
Potassium ferrate
SRF
Extracellular polymeric substances (EPSs)

**Abstract**

The potential effects of potassium ferrate \((K_2FeO_4)\) on sludge dewatering under different pH values (between 3 and 8) and the mechanism of its reaction were investigated in this study. Specific resistance of filtration (SRF) was used to evaluate sludge dewaterability. Sludge water distribution was measured by the drying test. Sludge floc structure was observed by microscopic examination. Soluble chemical oxygen demand (SCOD), extracellular polymeric substances (EPSs) content, sludge disintegration degree and sludge particle size were measured to explain the observed changes in sludge dewaterability. The results indicated that the potassium ferrate pretreatment at pH 3 enhanced sludge dewaterability, while potassium ferrate pretreatment at pH values of 4–8 caused deterioration of sludge dewaterability. At pH 3, sludge dewaterability increased with the increase of potassium ferrate, then decreased slightly when the dosage of potassium ferrate was greater than 1200 mg/L. The results showed that a potassium ferrate dosage of 1200 mg/L at pH 3 was an ideal condition, yielding maximum sludge dewaterability characteristics by generating sludge with optimal disintegration, and EPSs concentration. However, particle size changed slightly after potassium ferrate pretreatment at pH 3.

1. Introduction

Large amounts of waste activated sludge (WAS) are produced from wastewater treatment plants (especially the municipal wastewater treatment plants). Dewatering is one of the fundamental steps in sludge processing as it reduces sludge volume and, consequently, the cost of sludge transportation and disposal. However, the high water content and biological gel structure properties of sludge lead to difficulties in dewatering. Because of more stringent disposal regulations, appropriate sludge conditioning processes should be chosen prior to dewatering.

Various methods have been investigated as potential pretreatment technologies to enhance sludge dewaterability, such as the addition of calcined aluminum salts [1], acids and surfactants [2], seawater and brine [3], Fenton’s reagent pretreatment [4,5], fungal treatment [6], ultrasonication [7], microwave irradiation [8], electrolysis [9,10], and explosive explosion shockwave pretreatment [11]. Most of these processes improved sludge dewaterability characteristics by disrupting extracellular polymeric substances (EPSs), one of the main components with a strong affinity for water [12,13]. Polysaccharides and proteins, which entrap the water and cause a high viscosity, are the main components of EPSs [14]. By binding cells and particulate matter together, EPSs also change the particle size distribution of the sludge, again influencing the dewatering properties.

For decades, advanced oxidation processes (AOPs) utilizing free radicals as a primary oxidant [5,12], have been given increasing amounts of attention for sludge dewatering [15,16], in view of the short treatment time and high dewatering efficiency. From previous researches, AOPs improved the sludge dewaterability by affecting of the EPSs in two ways: (1) AOPs have the potential to degrade EPSs and; (2) AOPs influence the multifunctional groups of EPSs and promote their participation in several interactions [12,17]. The responsible mechanism is not fully understood, but...
the pretreatment might be based on partially oxidation and rearrangement of the surface components of sludge flocs.

Potassium ferrate ($\text{K}_2\text{FeO}_4$) is a strong oxidizing agent based on the reduction potentials of reactions (1) and (2) in acidic and alkaline solutions, respectively [18].

$$\text{FeO}_2^+ + 8\text{H}^+ + 3\text{e}^- \rightarrow \text{Fe}^{3+} + 4\text{H}_2\text{O}, \quad E^0 = +2.20 \text{ V} \quad (1)$$

$$\text{FeO}_2^+ + 4\text{H}_2\text{O} + 3\text{e}^- \rightarrow \text{Fe}^{3+} + 5\text{OH}^-, \quad E^0 = +0.72 \text{ V} \quad (2)$$

Under acidic conditions, the oxidizing power of ferrate(VI) is higher than those of the common oxidants such as permanganate, ozone, hydrogen peroxide, and hypochlorite [18]. Potassium ferrate can oxidize organic compounds efficiently [19–21]. By applying pre-treatment with potassium ferrate, the organic compounds, such as EPSs, in activated sludge are destroyed. Ferrate(VI) ions will be reduced to Fe(III) ions or ferric hydroxide, which not form any harmful by-product in the treatment processes, simultaneously generating a coagulant in the mixing unit process. Ye et al. [22] reported that potassium ferrate had a negative effect on the filterability, but improved the settleability and dewaterability extent of the sludge.

This study thus explored the possibility of adjusting pH and applying potassium ferrate pretreatment to increase the filterability and dewatering efficiency of the waste activated sludge. Specific resistance of filtration (SRF) and moisture in dewatered sludge cake were selected as indexes in the investigation of the characteristics of sludge filterability and dewaterability. Sludge disintegration degree, particle size, and EPSs content were selected as the parameters to explain the mechanism involved in the filterability and dewaterability of the sludge. The mechanism behind the changes observed in sludge dewaterability is also discussed.

2. Materials and methods

2.1. Activated sludge

WAS samples were collected from Lijiao Municipal Wastewater Treatment Plant (WWTP) in Guangzhou, China. This wastewater treatment plant utilizes an anaerobic–anoxic–oxic treatment process at a flow rate of 200,000 tons daily. The sludge was thickened by gravity to the required solid concentration and stored in a plastic box for less than 48 h at 4 °C prior to use. The characteristics of raw sludge are presented in Table 1.

2.2. Chemicals

Potassium ferrate ($\text{K}_2\text{FeO}_4$, 99%) was obtained from Galaxy Chemical Company (Wuhan, China). Other chemicals were of (or beyond) analytical grade and used as received without further purification. All solutions were prepared freshly using deionized water before experiments. Sulfuric acid ($\text{H}_2\text{SO}_4$, 8 M) and sodium hydroxide ($\text{NaOH}$, 8 M) were used to adjust the pH.

2.3. Experimental procedure

The sludge samples (500 mL) in a 1000 mL beaker were firstly adjusted to the required pH by $\text{H}_2\text{SO}_4$ and $\text{NaOH}$. The required dosages of potassium ferrate were thereafter added while the mixture was stirred using a six league mixing device ($\text{ZBR-4-G}$, Zhongrun, China) at a speed of 300 rpm for 5 min to start the pretreatment processes, and the sludge was then stirred slowly for 25 min. The pH value of the sludge was measured with a pHS–3C digital pH-meter (Shanghai, China). Samples taken prior to any treatment will be referred to in the text, tables and figures as ‘blank’ samples.

Each experiment was performed in triplicate, and average values were then obtained. Statistical correlations were analyzed using Sigmaplot software (version 10).

2.4. Analytical methods

The SRF were obtained using the method described by Feng et al. [7]. The moisture in dewatered sludge cake was measured using gravimetric method [23].

For measuring the free and bound water in sludge, a thermogravimetric method was introduced as described by Kopp and Dichtl [24]. The sludge sample was thickened in a centrifuge at 1000 rpm for 10 min and then was dried with a constant airflow of 300 mL/min at a temperature of 105 °C. The water distribution could then be derived from the plot of drying time vs. water content ([mass$_{\text{water}}$]/[mass$_{\text{solids}}$]) of the sample.

Microscopic examination of sludge floc structure was conducted immediately after $\text{K}_2\text{FeO}_4$ pretreatment in order to ensure that any observed changes were the product of $\text{K}_2\text{FeO}_4$ pretreatment. By using pipette, 0.05 mL sludge sample was spread evenly onto a microscope slide. And then, the floc structure was observed and photographed using a Nikon Eclipse 150 at 100 × magnification.

Chemical oxygen demand (COD) was measured in according with standard methods [23]. Sludge particle size distributions were examined with a laser particle size analyzer (LS-pop III, OMEC, China) that could detect particle size in the range of 0.2–200 μm. Particle size results were expressed in terms of its dp50, which is defined as the cutoff diameter at which 50% of the particles (by volume) have diameters equal to or less than the value of dp50. Sludge disintegration degree (DD$_{\text{SCOD}}$) was defined by Li et al. [25], comparing the potassium ferrate oxidation and the maximum soluble COD (SCOD$_{\text{NaOH}}$).

$$\text{DD}_{\text{SCOD}}(\%) = \frac{\text{SCOD}_3 - \text{SCOD}_{50}}{\text{SCOD}_{\text{NaOH}} - \text{SCOD}_{50}} \times 100 \quad (3)$$

SCOD$_3$ and SCOD$_{50}$ values are for treated and untreated sludge samples, respectively. SCOD$_{\text{NaOH}}$ was obtained via an alkaline hydrolysis procedure in which the initial sludge sample was mixed with 0.5 M NaOH at room temperature for 24 h [26].

The extracellular polymeric substance (EPSs) content of sludge supernatant were determined spectrophotometrically using a T6 UV/visible spectrophotometer (PGeneral, China). Proteins were determined by the Coomassie Brilliant Blue G-250 method [27], using casein as the standard. Polysaccharides were measured by the anthrone method [28], using glucose as the standard. To elucidate the relationship among sludge disintegration degree, EPSs concentration, and dewaterability, we analyzed the observed changes of the above parameters treated with potassium ferrate at pH 3.

3. Results and discussion

3.1. Dewatering characteristics

3.1.1. Dewatering characteristics of sludge treated with potassium ferrate under different pH conditions

The SRF value and the moisture of sludge cake were monitored to assess the dewaterability/filterability of treated sludge. The SRF value and the moisture of sludge cake were initially $7.08 \times 10^4$ m/kg

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>6.68</td>
</tr>
<tr>
<td>Water content (%)</td>
<td>98.58 ± 0.01</td>
</tr>
<tr>
<td>Soluble chemical oxygen demand (mg/L)</td>
<td>63.83 ± 2.4</td>
</tr>
<tr>
<td>Proteins (mg/L)</td>
<td>3.99 ± 0.15</td>
</tr>
<tr>
<td>Polysaccharides (mg/L)</td>
<td>6.15 ± 0.26</td>
</tr>
<tr>
<td>Specific resistance of filtration (m/kg)</td>
<td>$7.08 \times 10^5 \pm 2.71 \times 10^7$</td>
</tr>
</tbody>
</table>
and 86.13%, and decreased to $4.92 \times 10^3$ m/kg and 75.35% treated with potassium ferrate (1200 mg/L sludge) at pH 3. When the pH was beyond 4, the SRF values and moisture of the sludge cake increased rapidly with the increase of pH, even exceeding the SRF values and moisture of untreated sludge (Table 2). The SRF value and moisture in the sludge cake rose to $9.25 \times 10^3$ m/kg and 90.81%, respectively, when the sludge was treated only with potassium ferrate (1200 mg/L sludge), indicating that potassium ferrate had a negative effect on the dewaterability of sludge under neutral conditions. The SRF of the raw sample was approximately 1.5 times the SRF of the treated sludge at pH 3, indicating that treating with potassium ferrate at pH 3 was an appropriate means to release water.

The changes may be attributed to breakage of sludge flocs, which are repositories for water. Sludge flocs are broken into smaller fragments by potassium ferrate, a strong oxidizing agent over a broad pH range. The small sludge particle may clog the filter paper, resulting in reduction of the filterability. In general, acidification reduces the SRF and the moisture of sludge cake because acidification causes the zeta potential of sludge particles to approach neutrality, resulting in superior sludge dewatering [22,29]. For sludge flocs, the isoelectric point is in the pH range of 2.6–3.6 [30]. The smaller fragments can therefore be re-flocculated to larger particles at pH 3, improving the dewaterability. The variation in the EPSs concentration may also explain the observed changes of sludge dewaterability [22]. Zhang et al. [31] found that slight reductions in flocs size and slight increases in EPSs concentration improved sludge dewaterability after lower ultrasound dosages. While sludge dewaterability deteriorated significantly after higher ultrasound energy dosages due to the disruption of flocs structure and integrity, and to the release of intracellular and extracellular materials.

### Table 2
Sludge dewatering characteristics treated with potassium ferrate (K$_2$FeO$_4$, 1200 mg/L) under different pH conditions (mean ± S.E.).

<table>
<thead>
<tr>
<th>Condition</th>
<th>SRF (m/kg)</th>
<th>Moisture of the sludge cake (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blank test</td>
<td>$7.08 \times 10^3 \pm 2.71 \times 10^3$</td>
<td>86.13 ± 0.38</td>
</tr>
<tr>
<td>pH 3 + K$_2$FeO$_4$</td>
<td>$4.92 \times 10^3 \pm 1.46 \times 10^3$</td>
<td>75.35 ± 0.42</td>
</tr>
<tr>
<td>pH 4 + K$_2$FeO$_4$</td>
<td>$7.84 \times 10^3 \pm 2.23 \times 10^3$</td>
<td>84.51 ± 0.13</td>
</tr>
<tr>
<td>pH 5 + K$_2$FeO$_4$</td>
<td>$8.49 \times 10^3 \pm 1.32 \times 10^3$</td>
<td>89.25 ± 0.51</td>
</tr>
<tr>
<td>pH 6.68 (raw sludge) + K$_2$FeO$_4$</td>
<td>$9.25 \times 10^3 \pm 3.39 \times 10^3$</td>
<td>90.81 ± 0.26</td>
</tr>
<tr>
<td>pH 8 + K$_2$FeO$_4$</td>
<td>$1.63 \times 10^4 \pm 9.88 \times 10^3$</td>
<td>93.61 ± 0.35</td>
</tr>
</tbody>
</table>

3.1.2. Dewatering characteristics of sludge treated with different dosages of potassium ferrate at pH 3

As shown in Fig. 1, the SRF values and moisture of the sludge cake were smaller than those of raw sludge when the sludge was treated with different dosages of potassium ferrate (200–2000 mg/L sludge) at pH 3. When the dosage of potassium ferrate increased to 1200 mg/L sludge, the SRF value and moisture of the sludge cake decreased by 18.9% and 6.8%, respectively, compared to the SRF value and moisture of the sludge treated only with acid (pH 3). When the potassium ferrate dosage was beyond 1200 mg/L, sludge dewaterability deteriorated slightly. This deterioration may be explained by the kinetics of Fe(VI) reactions in aqueous solution. The reactions of Fe(VI) in aqueous solution can be categorized into two pathways, that is, reaction with water and reactions with solutes. The reaction of the ferrate ion with water is reported to be of second order with respect to Fe(VI) concentration [32]:

$$-\frac{d[Fe(VI)]}{dt} = k_1[Fe(VI)][Fe(VI)]$$  \hspace{1cm} (4)

Nearly all reactions of Fe(VI) with a variety of compounds were reported to be first order with respect to both reactants [33]:

$$-\frac{d[Fe(VI)]}{dt} = k_2[Fe(VI)][P]$$  \hspace{1cm} (5)

where [P] is the concentration of the compound that reacts with ferrate. The reaction of ferrate with a compound in aqueous solution is predominant compared to the reaction with water when $k_2[P] > k_1$, i.e., $[Fe(VI)] < (k_2/k_1) [P]$. When the concentration of Fe(VI) is higher than $(k_2/k_1) [P]$, the reaction with water prevails. Mechanical dewatering is significantly improved after hydrogen peroxide treatment [5]. SRF and capillary suction time (CST) values all decreased after Fenton’s reagent conditioning [34].

According to the data from SRF and the water content of sludge cake, we conclude that the optimal dose of potassium ferrate at pH 3 is 1200 mg/L sludge.

3.2. Drying test

The drying processes of the sludge treated with different dosages of potassium ferrate at pH 3 are illustrated in Fig. 2, the curve of which is composed of three stages as described by Kopp and Dichtl [35]. The three stages clearly represent the drying process of free, interstitial, and bound water, respectively. These curves demonstrate that all the water in the sludge samples was almost completely dried within 4 h. With increasing K$_2$FeO$_4$ dosage, the total

![Fig. 1](image_url) Sludge dewatering characteristics treated with different dosages of potassium ferrate (K$_2$FeO$_4$, 0, 200, 400, 800, 1200, 1600, 2000 mg/L) at pH 3.
drying times of treated sludge samples first declined and then increased slightly, but are shorter than the total drying time of untreated sludge. The shorter time implies the easier evaporation of water from sludge, and that the water is more easily removed from sludge by mechanical separation methods [7]. The free water and the bound water critical points were 3.35 g/g and 1.12 g/g, respectively, for the original sludge. When sludge was treated with a 1200 mg/L dosage of potassium ferrate under pH 3, as expected, the free water and the bound water critical points declined to 2.99 g/g and 0.95 g/g, respectively. A possible explanation for this decline is that the sludge flocs were disrupted and rearranged by acid hydrolysis and potassium ferrate oxidation, resulting in the release of bound water.

3.3. Microscopic observations

Photomicrographs of floc structure can provide good visual evidence for changes induced by potassium ferrate treatment at pH 3. Fig. 3 presents the photomicrographs of flocs at a magnification of 100× following potassium ferrate treatment at pH 3. These images revealed a series of changes: the floc structure was at first broken up and floc clusters were aggregated together treated with acid only; then the floc structure was slightly broken up and floc clusters were dispersed and made somewhat looser, which was due to the potassium ferrate oxidation. The change in floc structure is likely to affect the floc density [36], the particle size, and consequently, the filterability [37]. The images indicated that the sludge floc was broken up and rearranged treated with potassium ferrate and acid.

3.4. Changes in sludge particle size following potassium ferrate pretreatment at pH 3

The particle size distribution was measured in terms of cutoff diameters dp50 in sludge that was subjected to different dosages of potassium ferrate at pH 3 (Fig. 4). The dp50 of untreated sludge was 28.52 μm. The maximum dp50 was 30.03 μm when the sludge was treated with acid (pH 3) only. A similar result has been reported by Neyens et al. [12], who observed that acidic treatment caused the small flocs to aggregate. Initially, dp50 decreased with the increase of potassium ferrate. The particle size reduced by about 7% (compared to the maximum dp50) when potassium ferrate at a concentration of 1200 mg/L sludge was added, while the particle size increased slightly only when the dosage of potassium ferrate was greater than 1200 mg/L. In this case, potassium ferrate disintegrated the sludge flocs into smaller flocs, resulting in the slight reduction of dp50. Potassium ferrate oxidation at pH 3 did not have a significant effect on particle size. Similar changes in particle size were obtained by Bougrier et al. [38], who observed that ozonation only slightly decreased the median diameter of the particle size from 36 to 33 μm. Kim et al. [39] reported that the median sludge size decreased 61% with hydrogen peroxide treatment. The difference in degree of reduction of particle size may be due to the fact that the oxidation, concentration of oxidant, and size of the original sludge particles were all different from each other.

3.5. Effect of sludge disintegration on sludge dewaterability

3.5.1. Changes in sludge disintegration following potassium ferrate pretreatment at pH 3

Fig. 4 shows the sludge disintegration degree conditioned with potassium ferrate at pH 3. The sludge disintegration degree increased dramatically when the pH was adjusted to 3, indicating that acidification played an important role in the disruption of sludge flocs. The sludge disintegration increased to almost 2% when potassium ferrate at a concentration of 1200 mg/L was added, while the sludge disintegration degree declined slightly when the dosage of potassium ferrate was greater than 1200 mg/L due to the disruption of floc structure and release of extracellular and intracellular biopolymers [22]. The slight decrease of disintegration degree may be explained by the kinetics of Fe(VI) reactions in aqueous solution (described in Section 3.1.2).

3.5.2. Effect of disintegration degree on SRF and moisture of the sludge cake

Potassium ferrate oxidation and acidification (pH 3) can disrupt flocs and destroy microbial cells [2,22]. Increasing dosages of potassium ferrate (<1200 mg/L) increased both the SCOD of the supernatant as well as the DSCOD that describes the degree of sludge disintegration. Fig. 5 shows the relationship between the sludge disintegration degree and SRF/moisture of the sludge cake. The SRF and moisture of the sludge cake gradually decreased with the increasing DSCOD. These results are in agreement with those of Yu et al. [8] who observed that a proper sludge disintegration degree was vital for sludge dewaterability based on SRF. They found
that approximately 1.5–2% of $D_{\text{SCOD}}$ was the optimum value during microwave irradiation to improve the dewaterability of sludge.

During the potassium ferrate oxidation, the diffuse sludge flocs were broken into smaller fragments and bound water was released, improving the sludge dewaterability when these particles flocculated. The effect of disintegration degree on sludge dewaterability was also reflected by the correlation between $D_{\text{SCOD}}$ and SRF/moisture of the sludge cake ($R = 0.9711$, $R = 0.9983$, respectively).

3.6. Effect of EPSs on sludge dewaterability

3.6.1. Changes in EPSs concentration of the sludge supernatant

Proteins and polysaccharides in the raw sludge were estimated to have concentrations of 3.99 mg/L and 6.15 mg/L, respectively. After conditioning with acid and potassium ferrate, the EPSs and cellular substances were released into the aqueous phase, leading to an increase in protein and polysaccharide levels (Fig. 6). Proteins
and polysaccharides concentrations at first increased gradually with increasing dosage of potassium ferrate, then decreased slightly. At the dosage of 1200 mg/L sludge, proteins and polysaccharides concentration increased by 181% and 1163%, respectively, compared to the original sludge. At above 1200 mg/L dosage, the slight decrease in EPSs concentration may be explained by the kinetics of Fe(VI) reactions in aqueous solution (described in Section 3.1.2).

In the sludge flocs, proteins were distributed mainly in the pellet, less in the TB-EPSs (tightly bound-EPS), and virtually not at all in the LB-EPSs (loosely bound-EPS) and slime [40]. However, the distribution of polysaccharides was relatively uniform. According to Shao et al. [40], in our study, the EPSs were corresponded to LB-EPSs and slime in their study.

At the dosage of 1200 mg/L, proteins concentration was 11.25 mg/L, increased by 181%, compared to the raw sludge (3.99 mg/L). Based on the data of proteins, potassium ferrate did not exhibit adequate power in oxidizing TB-EPS in the present study, which possibly due to the reaction with inorganics and too short a contact time before potassium ferrate oxidized TB-EPS [18,22]. The effect of potassium ferrate dosage on the EPSs concentration was also reflected by the correlation between potassium ferrate dosage and protein/polysaccharide concentration ($R = 0.9799$, and $R = 0.9313$, respectively).

3.6.2. Effect of EPSs on SRF and moisture of the sludge cake
EPS, presumed to be the predominant polysaccharide and protein, is regarded as one of the most important factors influencing the dewaterability characteristics of waste sludge [41,42].

When sludge is treated with potassium ferrate at pH 3, large quantities of EPSs are released into the aqueous phase (Fig. 6). The high EPSs concentration not only increases the sludge viscosity but also decreases sludge dewaterability [2,22,43]. The relationship between EPSs concentration and sludge dewaterability, however, is not linear. In the present work, both SRF and water content of the sludge cake were observed to decrease with an increasing EPSs concentration less than 90 mg/L (Fig. 7). The increase in EPSs content indicated that the sludge were disrupted and rearranged...
because of acid hydrolysis and potassium ferrate oxidation, resulting in the improvement of sludge dewaterability. In this study, the correlations between the EPSs concentration and the SRF/moisture of the sludge cake were 0.9716 and 0.8945, respectively.

4. Conclusions

The effect of potassium ferrate pretreatment on sludge dewaterability was subjected to potassium ferrate dosages and sludge pH. Potassium ferrate pretreatment enhanced the sludge dewaterability at pH 3, while sludge dewaterability deteriorated at pH between 4 and 8. In this study, the optimal condition based on the SRF and the moisture of the sludge cake results were determined to be 1200 mg/L of potassium ferrate at pH 3. Sludge disintegration and EPSs concentration were found to play a vital role in the observed changes in sludge dewaterability. The optimal condition produced sludge disintegration, and the resultant EPSs concentration facilitated sludge dewaterability. Potassium ferrate pretreatment at pH 3 is a useful method for enhancing dewaterability.

The results obtained from our experiment provided an experimental and theoretical basis which is beneficial for the sludge dewaterability. Challenges have existed, however, to the implementation of ferrate(VI) technology in the full-scale treatment sewage sludge owing to either instability property of a ferrate(VI) solution or high preparation cost of a solid ferrate(VI) product. Therefore, further work should be carried out aiming to prepare the ferrate(VI) with high stability but a low manufacturing cost.

Acknowledgements

This research was supported by Guangzhou Science and Technology Plan Projects (No. 11C72050788). We also thank the staff of Lijiao Municipal Wastewater Treatment Plant for experimental assistance.

References


