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Effects of particle size of zero-valent iron (ZVI) on peroxydisulfate-ZVI enhanced sludge dewaterability

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Abstract−The advanced oxidization process has proven to be an effective conditioning technique for the improvement of sludge dewaterability. Zero-valent iron (ZVI) is often used as the catalyst of the oxidization process. This study applied ZVI with different particle sizes to the ZVI- peroxydisulfate reactions, and investigated their effects on the improvement of sludge dewaterability. It was found that ZVI particles with smaller sizes (100 and 400 meshes) led to slightly higher enhancement of sludge dewaterability (69.1%-72%) than the larger size particles (20-40 meshes) with the reduction rate of CST by 64%. However, after the treatment, the recycle rate of larger size ZVI particles was obviously higher than the small sizes ZVI particles: 98.3% vs. 87.6-89.7%. Different surface areas of the ZVI particles with different sizes might contribute to the phenomenon. For the small ZVI particles with the sizes of 100 and 400 meshes, no obvious differences of oxidization effects and the improvements of sludge dewaterability were found between them, which might be because an oxide layer could have been formed on the surface of fine ZVI particles and led to agglomeration. According to the economical analysis, the small particles (100 and 400 meshes) of ZVI were more economically favorable for the oxidative conditioning process with ZVI-peroxydisulfate than large ZVI particles (20-40 meshes).

Keywords: Waste Sludge, Dewaterability, Peroxydisulfate, Zero-valent Iron, Particle Sizes

INTRODUCTION

Although activated sludge processes have been widely applied to treat wastewater, the production of huge volume of waste activated sludge becomes its main drawback [1-3]. To reduce the volume of the waste activated sludge and facilitate its final disposal, the sludge has to be dewatered mechanically [4]. The bound water contained in activated sludge, which is combined with sludge flocs by capillary forces or chemical bonds, is the main factor hindering the dewatering process [5,6]. Therefore, a conditioning process was developed to transform the bound water into free form and improve the sludge dewaterability.

Recently, advanced oxidization processes have proven to be promising conditioning methods to improve sludge dewaterability. Classical Fenton reactions are the most traditional advanced oxidization processes. In the reaction process, ferrous salt is used as the catalyst, which could activate hydrogen peroxide and form oxidizing hydroxyl radicals in the acid condition [7]. But both ferrous salt and hydrogen peroxide are chemically unstable and the process is difficult to conduct due to its harsh operation condition. Therefore, more stable chemicals, such as the combination of zero-valent iron and peroxydisulfate (PDS) salt have been used to replace the traditional Fenton reagent [8]. The reactions between zero-valent iron and peroxydisulfate ion are shown as below (Eqs. (1)-(3)) [9]:

$$Fe+S_2O_8^{2-}\rightarrow Fe(II)+SO_4^{2-} \quad (1)$$

$$Fe(II)+S_2O_8^{2-}\rightarrow Fe(III)+SO_4^{2-}+\bullet SO_4^{-} \quad (2)$$

$$Fe(II)+\bullet SO_4^{-}\rightarrow Fe(III)+SO_4^{2-} \quad (3)$$

As shown in Eqs. (1)-(2), zero-valent iron is oxidized to Fe(II) by peroxydisulfate ion. After that, the Fe(II) activates the peroxydisulfate ion and produces sulfate radicals. The reactions can be initiated in the neutral condition. Furthermore, the sulfate radicals have higher oxidization potential than hydroxyl radicals, which could further facilitate the oxidization and decomposition of sludge structure, and then enhance the sludge dewaterability.

The oxidizing process with combination of ZVI and peroxydisulfate has been applied to remove the refractory organics from wastewater; promising treatment effect has been achieved as well [10]. In our previous studies, we also found that the sludge dewaterability could be effectively improved with the ZVI- peroxydisulfate system [11,12]. The capillary suction time, which was the main indicator of sludge dewaterability, could be reduced by 50%-90% after the conditioning process, depending on different types of sludge. The economical analysis also demonstrated that the ZVI-peroxydisulfate system was a feasible method for sludge conditioning. It

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was reported that the particle size of ZVI was an important characteristic, which could significantly influence the ZVI related reactions [13,14]. However, the effects of ZVI particle sizes on the sludge conditioning process with ZVI-peroxydisulfate system have never been investigated before. As we hypothesized, different sizes of ZVI particles might greatly affect the treatment efficiency of sludge conditioning process. Therefore, different sizes of ZVI particles were used to investigate their effects on sludge dewaterability for the combined conditioning process with ZVI and peroxydisulfate. CST and dissolved iron concentration of sludge were measured before and after treatment.

METHODS

1. Sludge Sources and Chemicals

The full scale waste activated sludge used in this study was collected from a local wastewater treatment plant (Shenzhen, China). Before the tests, the sludge was settled for 48 h by gravity to increase its concentration. The total suspended solids (TSS) and volatile suspended solids (VSS) of the WAS were 26.4±0.6 g/L and 12.8±0.3 g/L, respectively. The SCOD was 608.8 mg/L, the CST was 86.3±0.9 s, and the pH was 6.6.

Analytical grade of reduced iron powder (Macklin Co.) with the size of 100 meshes, 400 meshes and reduced iron particles (Xingyi Co.) with the size of 20-40 meshes were used as the iron source for the experiments.

Analytical grade of potassium peroxydisulfate (Macklin Co.) was used as the source of peroxydisulfate ion.

2. Batch Tests

The batch tests were conducted with three kinds of ZVI particles with the sizes of 100, 400 and 20-40 meshes, respectively.

To investigate the effects of different ZVI particle sizes on the improvement of sludge dewaterability by combined conditioning process with ZVI and PDS, two groups of batch tests were designed for each size of ZVI particles (Table 1), respectively. Test group (1) was to study the effect of ZVI concentration (0-4 g/g TSS) during the conditioning process while the concentration of PDS was kept at 0.5 g/g TSS. Test group (2) was to investigate the effect of PDS concentration (0-1 g/g TSS) on sludge dewaterability while the concentration of ZVI was kept at 0.5 g/g TSS. The same batch tests were performed with all three different sizes of ZVI particles; the results were compared and analyzed. The concentration ranges of ZVI and PDS were selected based on our previous studies [11,12]; all the batch tests were conducted in duplicate.

Table 1. Design of the batch tests

Group[a]	No.	ZVI concentration (g/g TSS)	PDS concentration (g/g TSS)
I, Effect of ZVI concentrations	1	0	0.5
	2	0.25	0.5
	3	0.5	0.5
	4	1	0.5
	5	2	0.5
	6	4	0.5
II, Effect of PDS concentrations	7	0.5	0
	8	0.5	0.1
	9	0.5	0.25
	10	0.5	0.5
	11	0.5	0.75
	12	0.5	1

For each batch test, 100 ml of WAS was added into a 250 ml flask, ZVI and potassium peroxydisulfate were then transferred into the flasks as well. The samples within flasks were then placed on an orbital shaker at 150 rpm for 30 min. The treated sludge was sampled for measuring CST as described in section 4. The rest of the sludge was placed on the operating magnetic stirrers with the addition of magnetic stirrer bar. After 3 min treatment, the undissolved iron could be attached on the stirrer bar magnetically and then was separated from the sludge. The dissolved iron in sludge was measured by the approaches described in section 4.

3. Analytical Methods

Capillary suction timer (Trition-WPRL, Type 304) was used to measure the CST value. The measurement process was described in our previous studies [11]. CST was used as the indicator of sludge dewaterability, which stood for the filtration time needed for the sludge.

The coupled plasma-optical emission spectroscopy instrument (ICP-OES, optimal 8000) was applied to measure the concentrations of dissolved iron in sludge. Before the measurement process, the sludge sample was digested by nitric acid with microwave oven (MARS Xpress) for 20 min. The method was previously desctibed by Zhou et al. [11].

The TSS, VSS and SCOD concentrations of the original sludge were measured according to APHA standard approaches.

4. Data Analysis

The enhancement of sludge dewaterability was expressed by reduction percentage R (%) of CST, as calculated below:

$$\mathrm{CST}\ (\%) = \frac{\mathrm{CST}_0 - \mathrm{CST}_e}{\mathrm{CST}_0} \times 100\% \tag{4}$$

where CST_0 stood for the CST of the original sludge (s); CST_e represented the CST after treatment (s).

The dissolved iron from ZVI was expressed as follows:

$$\text{Dissolved iron from ZVI} = \mathrm{Iron}_e - \mathrm{Iron}_o \tag{5}$$

where $Iron_0$ was the iron concentration of original sludge (mg/L); $Iron_e$ was the residual iron concentration in sludge after the recycle of undissolved ZVI (mg/L).

The dissolved rate of ZVI represented the dissolved percentage of ZVI after treatment, which was evaluated as below:

$$\text{ZVI dissolved rate}\ (\%) = \frac{\mathrm{Iron}_e - \mathrm{Iron}_0}{\mathrm{ZVI}_0} \times 100\% \tag{6}$$

ZVI_0 was the addition of ZVI in sludge (mg/L).

RESULTS AND DISCUSSION

1. Effect of ZVI Particles with Small Sizes (100 and 400 meshes) on Sludge Dewaterability

Fig. 1(a) shows the effect of ZVI (100 meshes) concentrations on

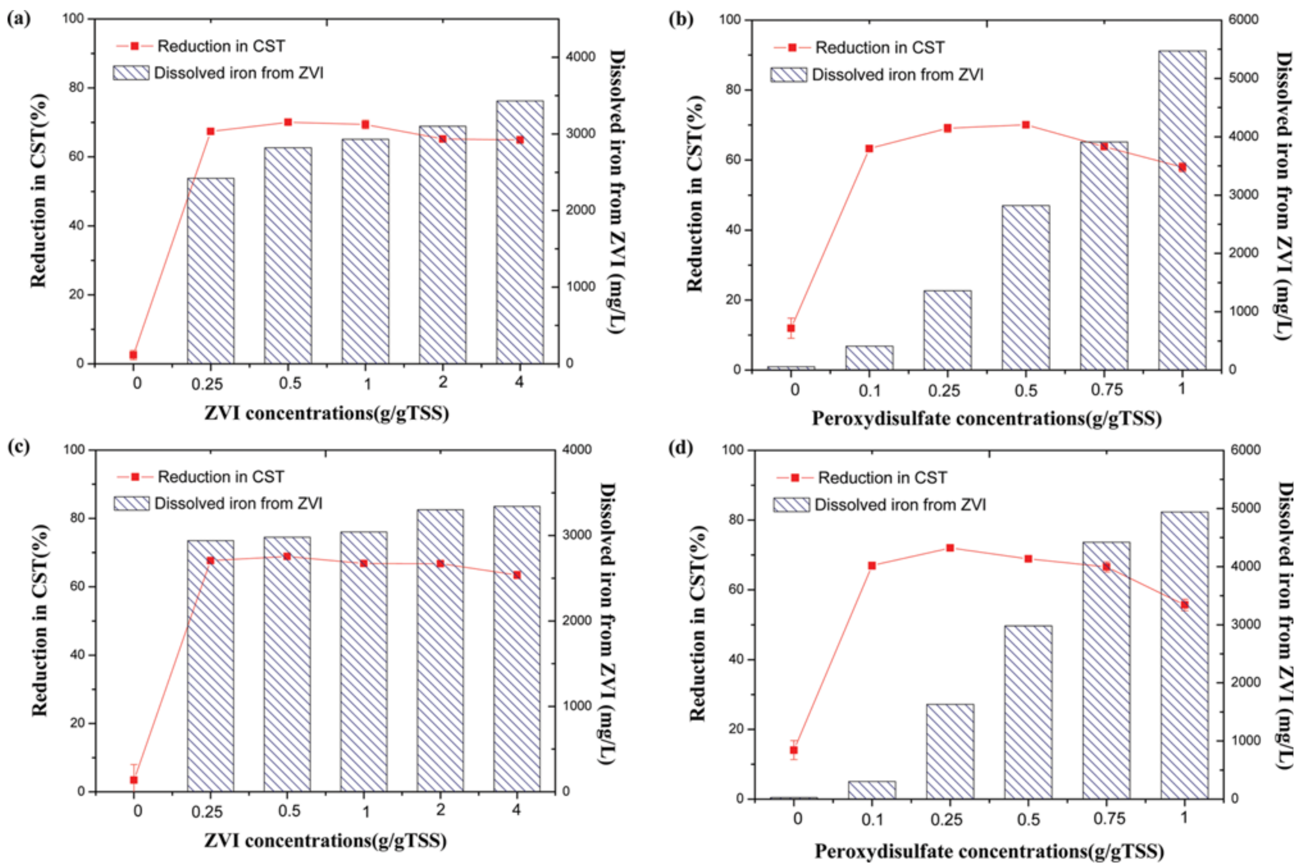

Fig. 1. (a) Effect of ZVI particles (100 meshes) concentrations on WAS dewaterability, along with the dissolved iron from ZVI. The PDS concentration was 0.5 g/gTSS. (b) Effect of PDS concentrations on WAS dewaterability, along with the dissolved iron from ZVI. The ZVI (100 meshes) concentration was 0.5 g/gTSS. (c) Effect of ZVI particles (400 meshes) concentrations on WAS dewaterability, along with the dissolved iron from ZVI. The PDS concentration was 0.5 g/gTSS. (d) Effect of PDS concentrations on WAS dewaterability, along with the dissolved iron from ZVI. The ZVI (400 meshes) concentration was 0.5 g/gTSS.

the enhancement of sludge dewaterability while the addition of peroxydisulfate (PDS) was kept at 0.5 g/gTSS. According to the results, significant enhancement of sludge dewaterability was achieved by the combination of ZVI and PDS. The CST reduction precentage was attained by 70.1%, while the addition of ZVI was 0.5 g/gTSS. Meanwhile, the dissolved iron from ZVI was 2821 mg/L. However, a further increase in ZVI addition from 0.5 g/gTSS to 4 g/gTSS could not facilitate the improvement of sludge dewaterability; the CST reduction rate decreased to 65.0%. At the same time, the dissolved iron from ZVI increased by 3,431 mg/L.

Based on Eq. (1), excess addition of ZVI could lead to higher production of Fe(II) while the peroxydisulfate ion was kept at a certain concentration. However, excess amount of Fe(II) could be the radical scavenger for the sulfate radicals as shown in Eq. (3). As a result, fewer sulfate radicals were available for oxidizing the sludge flocs. Therefore, excess addition of ZVI could negatively influence the sludge dewaterability.

According to results shown in Fig. 1(a), ZVI concentration of 0.5 g/gTSS was sufficient for enhancing sludge dewaterability. Thus, the effect of PDS concentrations on sludge dewaterability was studied with 0.5 g/gTSS ZVI. As shown in Fig. 1(b), the CST reduction rate increased from 11.9% to 70.1%, while the concentration of PDS increased from 0 g/gTSS to 0.5 g/gTSS. However, the CST reduction rate decreased to 57.9%, while a further increase of PDS concentration from 0.5 g/gTSS to 1 g/gTSS. Meanwhile, the dissolved iron from ZVI increased from 57 mg/L to 5,471 mg/L gradually.

The highest CST reduction was attained by 70.1%, while the concentration of PDS was 0.5 g/gTSS. However, the CST reduction rate was 69.1%, while the addition of PDS was 0.25 g/gTSS, and the P value of the two groups was over 0.05. Therefore, no significant difference existed between the PDS concentration at 0.25 g/gTSS and 0.5 g/gTSS, and the optimum PDS concentration was approximately 0.25 g/gTSS.

ZVI with the size of 400 meshes posed quite similar effect on sludge dewaterability. As shown in Fig. 1(c), the highest CST reduction rate was attained by 68.9% at 0.5 g/gTSS ZVI, while the PDS concentration was kept at 0.5 g/gTSS. After that, the CST reduction rate decreased to 63.4%, while the addition of ZVI increased to 4 g/gTSS. Meanwhile, the dissolved iron from ZVI increased to 3,341 mg/L. When the addition of ZVI was kept at 0.5 g/gTSS, the optimum CST reduction rate was achieved by 72%, while the concentration of PDS was 0.25 g/gTSS (Fig. 1(d)). A further increase of PDS concentration to 1 g/gTSS led to decrease of CST reduction

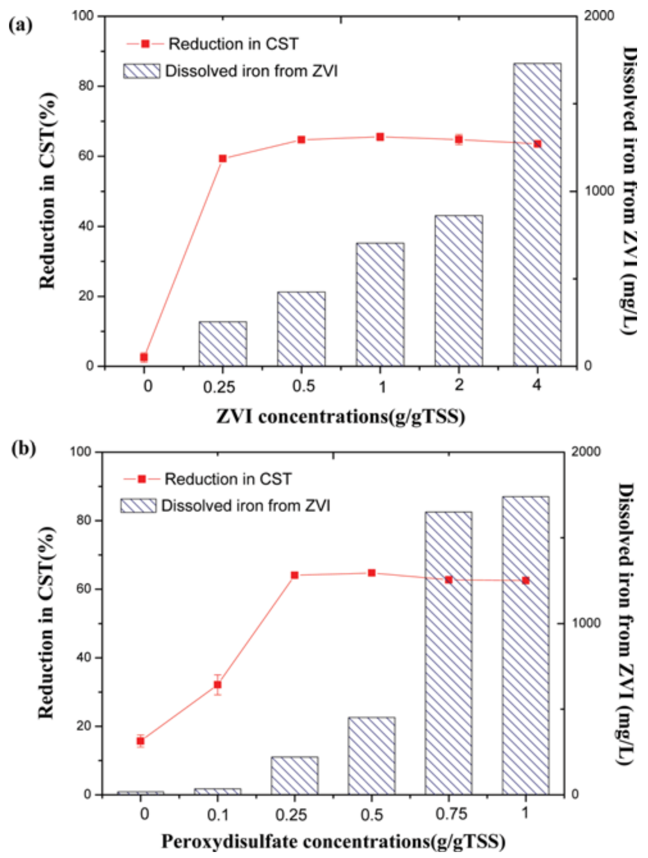

Fig. 2. (a) Effect of ZVI particles (20-40 meshes) concentration on WAS dewaterability, along with the dissolved iron from ZVI. The PDS concentration was 0.5 g/g TSS. (b) Effect of PDS concentration on WAS dewaterability, along with the dissolved iron from ZVI. The ZVI (20-40 meshes) concentration was 0.5 g/g TSS.

rate by 55.7%, while the dissolved iron from ZVI was 4,941 mg/L.

2. Effect of ZVI Particles with Large Sizes (20-40 meshes) on Sludge Dewaterability

According to the results shown in Fig. 2(a), significant enhancement of sludge dewaterability was attained after the addition of ZVI particles (20-40 meshes) in the presence of PDS at 0.5 g/g TSS. The CST reduction rate was achieved by 64.7%, while the concentration of ZVI increased to 0.5 g/g TSS. Although the CST reduction rate slightly increased to 65.6% while ZVI addition increased from 0.5 g/g TSS to 1 g/g TSS, the p value of the two groups was over 0.05. Therefore, no significant differences could be found between the CST reduction rate with 0.5 g/g TSS and 1 g/g TSS ZVI, and 0.5 g/g TSS could be sufficient for the dewaterability enhancement. When the concentration of ZVI increased from 1 g/g TSS to 4 g/g TSS, the CST reduction rate decreased to 63.5%. Meanwhile, the dissolved iron from ZVI gradually increased from 0 to 1,731 mg/L when the ZVI addition increased from 0 to 4 g/g TSS.

Fig. 2(b) shows the effect of PDS concentrations on the sludge dewaterability, while the addition of ZVI was kept at 0.5 g/g TSS. When the concentration of PDS increased from 0 g/g TSS to 0.5 g/g TSS, the CST reduction rate increased from 15.7% to 64.7%. However, the P value between the CST data with the addition of 0.25 g/g TSS and 0.5 g/g TSS PDS was over 0.05. Thus the optimum of addition of PDS was 0.25 g/g TSS, while the addition of ZVI was 0.5 g/g TSS. CST reduction percentage decreased to 62.5%, while the addition of PDS further increased from 0.5 g/gTSS to 1 g/gTSS. At the same time, the dissolved iron from ZVI increased from 17 mg/L to 1,741 mg/L. The could be explained by the mechanisms discussed in section 3.1.

3. Effect of ZVI and PDS on the Change of Sludge Dewaterability

It was found that the CST reduction rate reached 11.9% while only 0.5 g/gTSS ZVI was added in the absence of PDS (Fig. 1(b)). The phenomenon was also reported by our previous studies; it was likely due to the fact that ZVI was highly reductive, so it could decompose the sludge flocs and change its dewaterability alone.

Furthermore, after the addition of excess PDS over 0.5 g/gTSS, the CST reduction rate decreased obviously, while the addition of ZVI kept at 0.5 g/gTSS. As shown in Eq. (1)-(3), excess addition of PDS could transform more ZVI into Fe(II); the superabundant produced Fe(II) could then transform the oxidizing sulfate radicals produced in Eq. (2) into sulfate ion, and finally exhaust the sulfate radicals. Therefore, excess addition of PDS could negatively affect the sludge dewaterability as well.

The sludge properties were also changed obviously after the treatment by ZVI and PDS. When the addition of ZVI and PDS was 0.5 g/gTSS and 0.25 g/gTSS, the SCOD of the WAS increased from 608.8 mg/L to 979.3 mg/L, that implied the decomposition of WAS flocs after oxidization process. No significant change was found on the particle sizes of the WAS flocs after treatment; the results agreed with our previous studies.

4. Effect of Particle Sizes of ZVI on Oxidizing Capacity

The surface area of ZVI particles varied due to the sizes of ZVI. Normally, the surface area increased while the sizes of ZVI were smaller. With larger surface area, the ZVI particles could more easily take part in the chemical reactions. Therefore, the particle sizes of ZVI played an important role in the chemical reactions. We found that the oxidizing conditioning process with small sizes of ZVI particles (100 meshes and 400 meshes) had higher CST reduction rate than the large sizes of ZVI particles (20-40 meshes), i.e., 69.1% and 72% vs. 64%. The fact was likely due to that the small sizes ZVI particles could provide larger surface area, which could react with peroxydisulfate ion and produce more sulfate radicals; thus the sludge flocs could be easier to be decomposed and the sludge dewaterability was finally enhanced. The dissolved rates of ZVI also agreed with the speculation, as shown in Figs. 1-2. More dissolved iron could be detected by the small sizes of ZVI particles rather than the large ones, which implied that small sizes of ZVI particles took part in the reactions to a greater extent.

However, for the two kinds of small size ZVI with the meshes of 100 and 400, i.e., 149 μm and 37 μm, respectively, the CST reduction rates and dissolved iron rates were quite similar. For example, the optimum values of CST reduction rates and dissolved iron rates were 69.1%/10.3% (100 meshes) and 72%/12.4% (400 meshes), respectively. According to Dong et al. [13], it is easier to form oxide layer on the surface of fine ZVI particles, which could further facilitate the agglomeration of ZVI particles and hinder the reactions. Therefore, although very fine ZVI particles, for instance, with the size of 37 μm, had larger surface area, it did not necessarily enhance the oxidizing effect more obviously than the larger ones with the size of 149 μm.

5. Recovery of ZVI and Economical Analysis

According to Figs. 1-2, for all three kinds of ZVI particles with different sizes, the optimum addition of ZVI and PDS was the same, 0.5 g/gTSS and 0.25 g/gTSS, respectively. However, for the two kinds of ZVI particles with smaller sizes, the dissolved iron concentrations were much higher than the ZVI particles with large sizes. For example, when the addition of ZVI particles with small sizes was kept at 0.5 g/gTSS, the dissolved rate of ZVI ranged from 0.4% to 41.5% and 0.2% to 37.4%, while the PDS concentration increased from 0 g/gTSS to 1 g/gTSS (see Fig. S1-S2), respectively. Nevertheless, the dissolved rate of ZVI particles with large size only increased from 0.1% to 13.2% under the same conditions (see Fig. S3). Furthermore, when the addition of ZVI and PDS was kept at the optimal concentration, the dissolved rate for these different kinds of ZVI was 10.3% (100 meshes), 12.4% (400 meshes) and 1.7% (20-40 meshes). Therefore, the recycle rate of ZVI particles with large sizes was much higher than the ZVI particles with small size.

As mentioned, the improvement rate of sludge dewaterability with the application of ZVI particles with smaller sizes (100 and 400 meshes) was quite similar. Therefore, the smaller ZVI particles with the size of 100 meshes were used to compare to the larger ZVI particles with the size of 20-40 meshes. A desktop scaling-up study was conducted to compare the ZVI+PDS conditioning processes with the ZVI particles sizes of 100 meshes and 20-40 meshes for the improvement of sludge dewaterability in an assumed WWTP with a population equivalent of 100,000.

In the economic analysis, it was assumed that two types of conditioning processes had the same efficiency on the enhancement of sludge dewaterability (i.e., 64% reduction in CST), while the addition amount of PDS and ZVI (100 meshes) was 0.5 g/g TSS and 0.10 g/g TSS and PDS and ZVI (20-40 meshes) was 0.5 g/g TSS and 0.25 g/g TSS, respectively. According to the results (see Table 2), the ZVI+PDS conditioning process with the ZVI particle size of 100 meshes could save up to $53045 per year compared with the ZVI particle size of 20-40 meshes.

CONCLUSIONS

The effect of zero-valent iron with different particle sizes on sludge dewaterability with the combination of peroxydisulfate was investigated via a series of batch tests. The reduction rate of CST was applied as the indicator of the enhancement of sludge dewaterability. The results showed that optimum CST reduction rates could be achieved by 69.1%-72% with small sizes of ZVI particles (100 meshes and 400 meshes), while the optimum CST reduction rate with the large size of ZVI particles (20-40 meshes) was 64%. However, after the treatment, the recycle rate of ZVI particles with large sizes was much higher (98.3%) than the smaller size ZVI powder (87.6-89.7%). According to the economical analysis, the small particles of ZVI were more economically favorable for the oxidative conditioning process with ZVI-peroxydisulfate than large ZVI particles.

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SUPPORTING INFORMATION

Additional information as noted in the text. This information is available via the Internet at http://www.springer.com/chemistry/journal/11814.

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Table 2. Economic analyses of the ZVI+PDS conditioning processes with the ZVI particles sizes of 100 meshes and 20-40 meshes for the improvement of sludge dewaterability in an assumed WWTP with a population equivalent of 100,000[a]

Parameters	ZVI (100 meshes)+PDS conditioning[b]	ZVI (20-40 meshes)+PDS conditioning[b]
Amount of WAS before and after conditioning (dry tonne/year)	750	750
ZVI powder ($/year)	4640	2560
PDS ($/year)	36750	91875
Total cost ($/year)	41390	94435
Total saving ($/year)	Not applicable	53045

[a]In the economic analysis, it was assumed that two types of conditioning processes had the same efficiency on the improvement of sludge dewaterability (64% reduction in CST)

[b]For the ZVI+PDS conditioning method with the ZVI particle size of 100 meshes, the ZVI and PDS concentrations were set as 0.5 g/g TSS (only the consumption of unrecovered ZVI was considered) and 0.10 g/g TSS. For the ZVI+PDS conditioning method with the ZVI particle size of 20-40 meshes, the ZVI and PDS concentrations were set as 0.5 g/g TSS (only the consumption of unrecovered ZVI was considered) and 0.25 g/g TSS. The prices of ZVI, Potassium persulfate are $400 and $700/tonne, respectively

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Supporting Information

Effects of particle size of zero-valent iron (ZVI) on peroxydisulfate-ZVI enhanced sludge dewaterability

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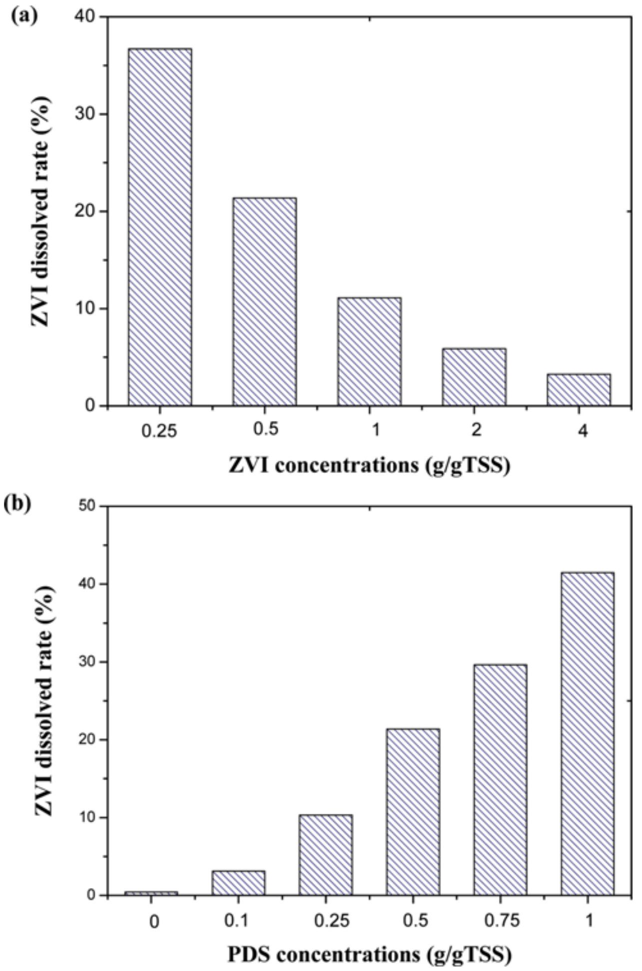

Fig. S1. ZVI (100 meshes) dissolved rate at different ZVI (a) and PDS (b) concentrations, in (a), the addition of ZVI was 0.5 g/g TSS in all tests. In (b), the addition of PDS was 0.5 g/g TSS in all tests.

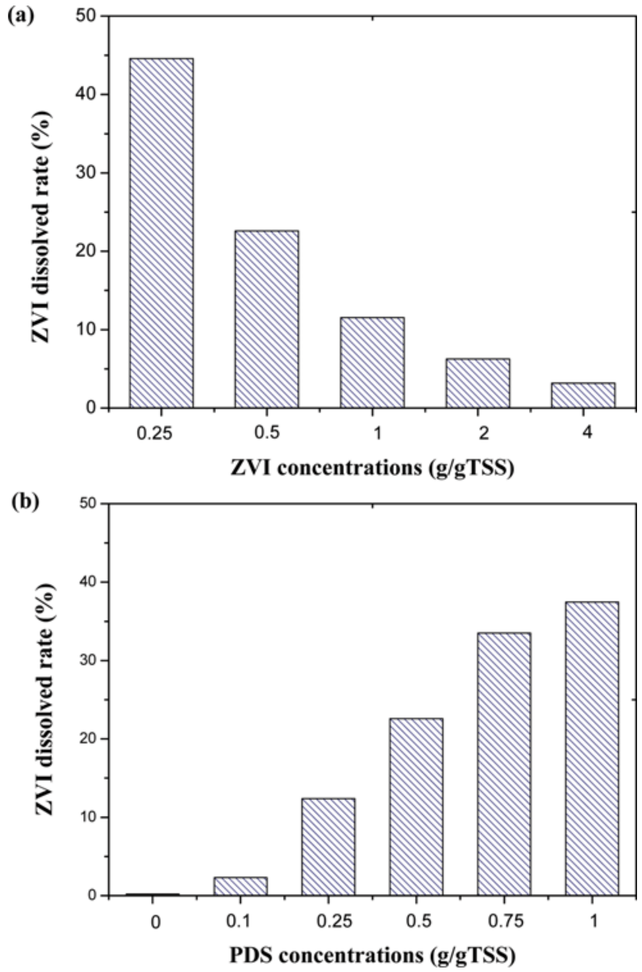

Fig. S2. ZVI (400 meshes) dissolved rate at different ZVI (a) and PDS (b) concentrations, in (a), the addition of ZVI was 0.5 g/g TSS in all tests. In (b), the addition of PDS was 0.5 g/g TSS in all tests.

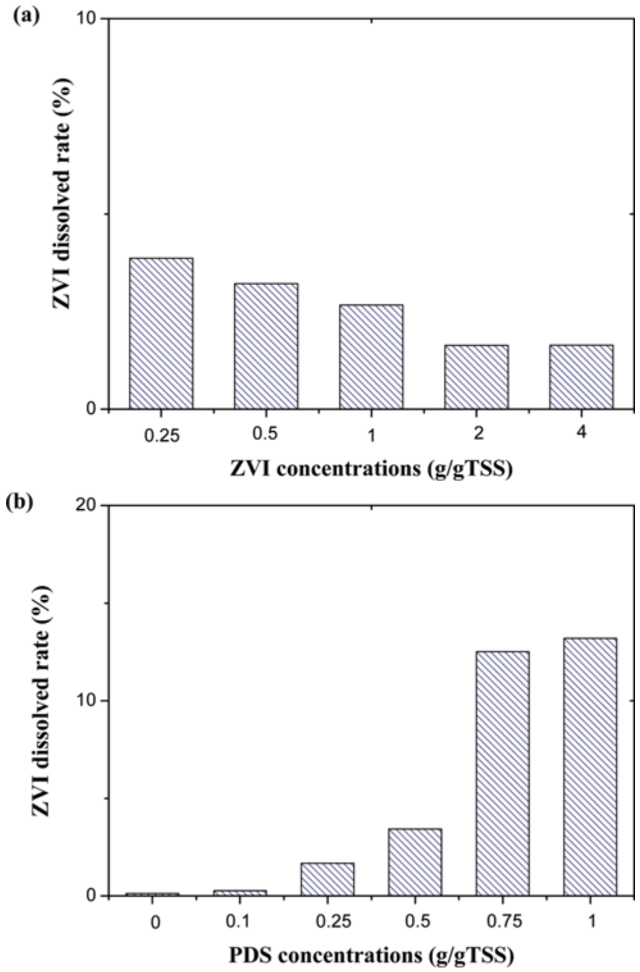

Fig. S3. ZVI (20-40 meshes) dissolved rate at different ZVI (a) and PDS (b) concentrations, in (a), the addition of ZVI was 0.5 g/g TSS in all tests. In (b), the addition of PDS was 0.5 g/g TSS in all tests.