Effect of potassium ferrate on disintegration of waste activated sludge (WAS)

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ABSTRACT

The activated sludge process of wastewater treatment results in the generation of a considerable amount of excess activated sludge. Increased attention has been given to minimization of waste activated sludge recently. This paper investigated the effect of potassium ferrate oxidation pretreatment on the disintegration of the waste activated sludge at various dosages of potassium ferrate. The results show that potassium ferrate pretreatment disintegrated the sludge particle, resulting in the reduction of total solid content by 31%. The solubility (SCOD/TCOD) of the sludge increased with the increase of potassium ferrate dosage. Under 0.81 g/g SS dosage of potassium ferrate, SCOD/TCOD reached 0.32. Total nitrogen (TN) and total phosphorous (TP) concentrations in the solution all increased significantly after potassium ferrate pretreatment. The sludge particles reduced from 116 to 87 μm. The settleability of the sludge (SVI) was enhanced by 17%, which was due to the re-flocculation by the by-product, Fe(III), during potassium ferrate oxidation and the decrease of the viscosity. From the result of the present investigations, it can be concluded that potassium ferrate oxidation is a feasible method for disintegration of excess activated sludge.

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1. Introduction

Activated sludge process is a core technology in wastewater treatment plants, which produces large quantities of waste activated sludge (WAS). WAS produced in the process must be treated and disposed of properly and may account for up to 60% of total plant operating cost [1]. So far, the ultimate disposal of WAS has been and continues to be one of the most serious problems faced by wastewater utilities. The increase of WAS generation, together with the more stringent regulations, generates a real challenge in the field of environmental engineering technology. There is therefore considerable impetus to develop strategies for reducing the volume and mass of WAS produced.

WAS is known to be more difficult to dewater and digest than primary sludge, and hydrolysis is considered to be the limiting step during anaerobic digestion [2]. Indeed, after aerobic treatment in a wastewater treatment plant, the main part of the sludge's organic matter is enclosed in bacterial flocs which reduce its availability to anaerobic microorganisms. Therefore, WAS disintegration or solubilization by thermal [3,4], chemical [5,6],
thermochemical [7,8], mechanical [9–11], and biological [12,13] methods were investigated, with the purpose of improving its biodegradability. The investigations mentioned above all found that excess sludge containing a great portion of bacteria is the most suitable for disintegration. Disintegration of the sludge can be achieved by the destruction of the sludge floc or by the disruption of the microorganism cells in the sludge, which depends on the type of disintegration and the intensity of disintegration. The disintegration of the cells leads to a release of organic substances which are easily accessible to a subsequent biological degradation process. Minimization of the excess sludge production could be achieved by re-circulating the pretreated sludge into a bioreactor. Pre-treating the sludge by thermal hydrolysis before digestion reduced energy demand compared to the conventional digestion, but associated with odorous problem. Alkaline hydrolysis has traditionally studied for the sludge reduction and the enhancement of the sludge biodegradability. Ozone oxidation is one of powerful sludge destruction processes, but is expensive.

Potassium ferrate is a strong oxidant reagent that can oxidize organic compounds efficiently [14,15]. Potassium ferrate oxidation, which is one of the advanced oxidation processes (AOPs), may offer a promising technology for the disintegration of the excess sludge. It is an environmental-friendly oxidant, commonly applied in wastewater treatment [16,17], as it yields no noxious or polluting byproducts. By applying potassium ferrate, organic compounds, such as extracellular polymeric substances (EPS) in the

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excess activated sludge are destroyed, a part of the sludge is solubilized to biodegradable organics, and ferric coagulating species are generated simultaneously. It may lead to increased flocculation and dewaterability of the sludge, and also reduction of the sludge volume. It can be expected that potassium ferrate oxidation disintegrate biomass particulates and transform them into soluble compositions, destruct bacterial cell membranes. Several researchers found that Photo-Fenton process and H_2O_2 oxidation disintegrated the activated sludge efficiently [18,19]. However, to the best of our knowledge, the effect of potassium ferrate oxidation on the disintegration of the sludge has not been studied.

The aim of this study is thus to test the suitability of applying potassium ferrate oxidation to disintegrate and/or solubilize WAS, in order to make them more easily biodegradable and reduce the sludge production. The characteristics of the sludge, such as total solid content, solubility, particle size, viscosity, and settleability were examined after applying potassium ferrate oxidation to asses the sludge disintegration degree.

2. Materials and methods

2.1. Activated sludge samples

The sludge samples were taken from the aerated basin of Ningbo municipal wastewater treatment plants (WWTP) in Ningbo, China. Table 1 shows the characteristics of the sludge sample. Ningbo WWTP treats 10,000 m^3/d of wastewater (80% domestic and 20% leachate) using conventional activated sludge process. The sludge was stored in some plastic containers placed in an ice cooler during the transportation from the plant site to the laboratory. The sludge was settled for 1 h and the supernatant was decanted to obtain the concentrated sludge sample. The excess activated sludge was not conditioned with any conditioner. Sample tests started immediately and were completed within 20 h, while being kept refrigerated at 4 °C.

2.2. Potassium ferrate preparation

Potassium ferrate (K_2FeO_4) was prepared using the method by Li et al. [20] and modified slightly. 50 mL HCl (37%) was slowly reacted with 20 g of MnO_2 to produce chlorine. The chlorine was subsequently added to pre-chilled KOH solution (30 g KOH in 50 mL of water) with stirring for over 2 h. A quantity of 45 g KOH was then added into this solution and the resulting suspension was cooled. The precipitate of KCl was removed from the suspension by filtration using a glass filter (P-0), leaving a concentrated and strongly alkaline solution of potassium hypochlorite. This yellow solution of KClO was then stirred rapidly while 18.75 g Fe(NO$_3$)$_3$·9H$_2$O was added slowly for over 1 h under cooling conditions (<5 °C). In these conditions, the Fe(III) ion was readily oxidized to Fe(VI) and the solution became dark purple in color. A quantity of 35 g KOH was then added in small portions to the Fe(VI) solution and the mixture was stirred for 15 min. The resulting solution was allowed to stand for a further 40 min. The resulting dark purple slurry was filtered with a glass filter (P-0), after which the filtrate was discarded, and the precipitate was washed six times with a 5 mL of cold 3 M aqueous KOH solution every time. The filtrate from the washings was collected and added to a flask containing 42.5 mL of a chilled saturated KOH solution. The solution was mixed, allowed to stand for 10 min, and then filtered initially with a glass filter (P-3), followed by double filtering with GF/A filter papers (Whatman Ø70 mm). The precipitate was flushed with benzene (four times × 5 mL), isopropyl alcohol (three times × 5 mL) and ether (three times × 5 mL). The final product, solid potassium ferrate was collected and stored in a vacuum desiccator prior to further use.

2.3. Sludge oxidation pretreatment with potassium ferrate

The sludge samples (350 mL) in a 500 mL beaker were rapidly mixed with different dosage of potassium ferrate at a speed of 400 rpm for 3 min to start the pretreatment process, and then slowly stirred for 7 min. Samples were collected and analyzed after 2 h reaction. Experiments were carried out at an ambient temperature (25 °C). Each test was performed in triplicate.

2.4. Sludge particle size

The particle size was determined by a Malvern Mastersizer/E instrument (Malvern Instruments Ltd., Worcestershine, UK) with a 300 mm lens which enables the measurement of particles in the range of 0.9–546 μm. The sludge suspension was then continuously recycled through the sample cell of the Malvern with a peristaltic pump to be exposed to a 2 mW He–Ne laser (wavelength 633 nm). Each sample was measured three times with a relative standard deviation 0.1–4.5%. The scattered light is detected by means of a detector that converts the signal to a size based on volume. The average size of the flocs was given as the mean based on the volume equivalent diameter.

2.5. Analytical methods

The concentration of potassium ferrate in an aqueous solution was determined by UV–vis spectroscopy. K_2FeO_4 dissolved as FeO_4^{2-}, has a distinctive UV–vis spectrum with a maximum absorbance at 510 nm.

The physicochemical characteristics of the excess activated sludge, including pH, total suspended solid (TSS), volatile suspended solid (VSS), soluble chemical oxygen demand (SCOD), total chemical oxygen demand (TCOD), total nitrogen (TN), and total phosphorous (TP) were measured by the Standard Methods [21]. The COD of filtrate after 0.45 μm filters was referred as SCOD. The ratio of SCOD/TCOD was applied as an indicator to evaluate the solubilization degree of the sludge. Viscosity of the sludge mixture was determined by a rotational viscometer Brookfield DV + PRO (Brookfield Engineering Laboratories, Inc.). Settleability of the sludge was evaluated by carrying out a sludge volume index (SVI) test according to the Standard Method [21]. All samples were made in triplicate.

3. Results and discussion

3.1. Effect of potassium ferrate on solubilization of WAS

The prepared potassium ferrate crystal is black purple in color and remains stable for more than a year if moisture is excluded. The XRD and infrared spectroscopy results all indicated that the prepared sample was potassium ferrate (Fig. 1). The purity of the potassium ferrate sample measured by UV–vis spectroscopy method was 99%.

Potassium ferrate can solubilize the particulate component of the excess sludge into soluble form, resulting in reduction of the sludge volume. Organic matter is released and solubilized during the disruption of the sludge floc and cell after potassium ferrate oxidation. TSS contents of the excess sludge were measured to confirm the effect of potassium ferrate oxidation on sludge solid reduction. As shown in Fig. 2, TSS content of excess sludge decreased from 13.05 to 8.98 g/L (31% removal of TSS) with an increase of potassium ferrate dosage from 0 to 0.81 g/g SS. However, the TSS reduction was not obvious after further increase of potassium ferrate. The results were comparative to the other findings. Kim et al. [19] found that TSS content reduced 33% with 1.6 M hydrogen peroxide oxidation treatment.

Table 1
Characteristics of excess sludge collected from municipal WWTP of Ningbo.

	TSS (g/L)	VSS (g/L)	pH	Conductivity (mS/cm)	COD (mg/L)	SCOD (mg/L)
Sludge sample	13.05 ± 1.22	8.86 ± 0.89	6.95	7.96	7893 ± 621	211 ± 17
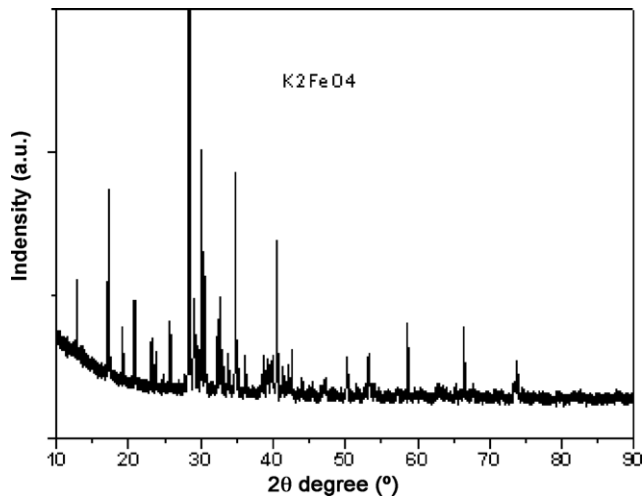
Fig. 1. XRD spectra of K_2FeO_4 samples.

Fig. 3. Effect of potassium ferrate oxidation on the sludge disintegration degree.

Many of suspended solid ingredients in WAS are organic matters of the living thing origin, so the effect of sludge disintegration was determined in terms of changes in the dissolved COD. Fig. 3 shows the effect of potassium ferrate oxidation on the sludge solubilization. For the original sludge, most COD is associated with the solid phase rather than in a soluble form. The SCOD/TCOD of the original sludge was 2.76%, and it increased to 32.18% at potassium ferrate dosage of 0.81 g K_2FeO_4/g SS. The COD in supernatant thereby increased by 12 times after potassium ferrate pre-treatment. The SCOD/TOD increased with the increase of potassium ferrate, but the increase degree was not significant when the dosage of potassium ferrate was beyond 0.81 g/g SS. The SCOD/TOD ratio was 35.87% at potassium ferrate dosage of 1.58 g K_2FeO_4/g SS. This may be due to two reasons: (1) the further added potassium ferrate oxidized SCOD of the sludge suspension, not the sludge solid; (2) a part of potassium ferrate decomposed itself due to its poor stability. Therefore, the embedded potassium ferrate for controlled release is being prepared and investigated its oxidation and stability in our laboratory. If the cost of potassium ferrate was taken into account, the dosage of ferrate should not be higher than 0.8 g/g SS.
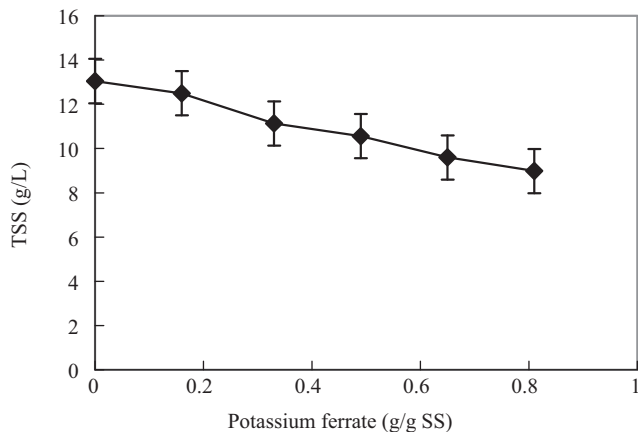
The change of the dissolved COD is similar to the results of Egemen et al. [22], Vlyssides and Karlis [23] and Deleris et al. [24]. In their studies, SCOD increased as ozonation or thermal/alkaline pretreatment continued, and reached a maximum finally. It suggested that mineralization of the dissolved COD occurred during the latter reaction phase.

The ratio of SCOD/TCOD is an indication of the extent of hydrolysis or disintegration or solubilization. Such an observation closely corresponds to the other reports. SCOD/TCOD increased from 0.06 (original sludge) to 0.15 using a microwave treatment to 96 °C, and to 0.27 using a conventional thermal treatment at 96 °C [4]. Chu et al. [25] reported that an ultrasound treatment achieved SCOD/TCOD ratio of 0.2 after 120 min of a sonication (20 kHz, 110 W) for WAS. A combination of chemical treatment with thermal method could provide a better disintegration of the sludge. SCOD/TCOD ratio of 0.8 was obtained at 170 °C (autoclave) at pH 12 with KOH [26]. The solubilization efficiencies (SCOD/TCOD) were 54.7% and 27.6% by hydrogen peroxide and alkaline treatment, respectively [19]. When hydrogen peroxide was applied to oxidize the sludge after the acid or alkaline treatment, the solubilization efficiencies were enhanced. Bougrier et al. [27] compared the COD solubilization of the sludge by sonication, ozone oxidation, and thermal treatment. They achieved SCOD/TCOD ratio of 0.15 for sonication (6250–9350 kJ/kg TS), 0.20–0.25 for ozone oxidation (0.10–0.16 g O_3/kg TS), and 0.40-0.45 for thermal treatment (170–190°C). Using the sludge cultured by artificial sewage and a semi-batch operational mode, Saktaywin et al. [28] obtained a sludge solubilization ratio of 0.3 with a specific ozone consumption of 30–40 mg O_3/g SS. The combination of alkaline pH 12.5 and microwave pretreatments led to an improved COD solubilization. SCOD/TCOD increased from 0.005 (control) to 0.37 [29].

Valuable components like nitrogen and phosphorous are abundant in the sludge, so various technologies for the recovery of nitrogen and phosphorous in the sludge have been developed. The recycling of the sludge components can only be done with the dissolved fraction, but most of the organic fraction is located in the solids. The high concentrations of the dissolved organic components in the sludge are important for a cost efficient recycling of these valuable components. The TN and TP concentrations in the supernatant of the sludge after potassium ferrate oxidation were depicted in Fig. 4. Nitrogen and phosphorus in the sludge

Fig. 2. Effect of potassium ferrate oxidation on reduction of the excess sludge solid.

Fig. 4. Effect of potassium ferrate oxidation on TN and TP release of the sludge.
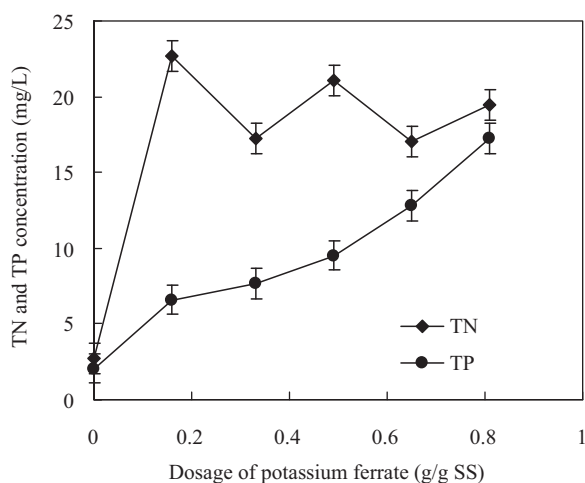
solid were found to be solubilized into the bulk solution. The TN and TP concentrations all increased with the increase of potassium ferrate dosage. With a specific potassium ferrate consumption of 0.81 g/g SS, TN and TP concentrations in solution were increased by 6 times and 7 times, respectively. However, the release of nitrogen and phosphorus was not effective with low ozone dose. The fractions of nitrogen and phosphorus in the supernatant increased to 12% and 5%, respectively. A large part of the released substances from the sludge were in the form of micro-solids [30]. The results again implied that potassium ferrate had a significant effect on the solubilization of the excess sludge. However, until now there has been little report about the effect of disintegration technology on the solubilization of nitrogen and phosphorous in the sludge. Additionally, due to limited literature on potassium ferrate oxidation of the sludge, it is quite difficult to compare the results obtained from this study with the previous relevant studies. The detailed information on recovery of the valuable components should be investigated thoroughly in the future.

3.2. Effect of potassium ferrate on physicochemical characteristics of WAS

By applying potassium ferrate oxidation the break-up of the sludge flocs occurred immediately. As shown in Fig. 5, initially, the flocs size decreased with the increase of potassium ferrate. Compared with the untreated sludge, the sludge particle size reduced by 25% when potassium ferrate of 0.65 g/g SS was added. Similar changes in particle size also were observed by Kim et al. [19]
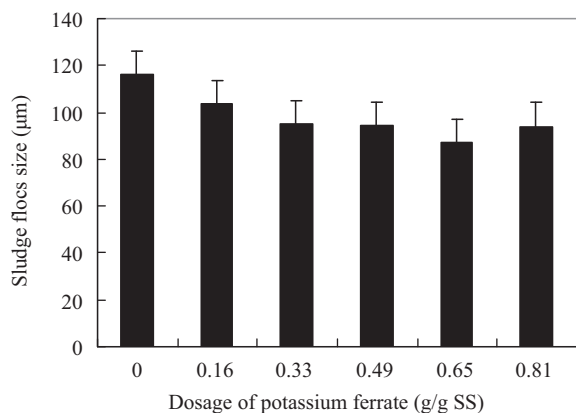

Fig. 5. Effect of potassium ferrate oxidation on the sludge flocs size.
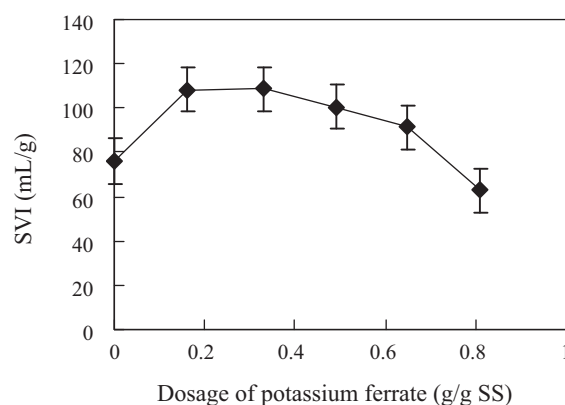

Fig. 6. Effect of potassium ferrate oxidation on the sludge settleability.

with hydrogen peroxide. They found that the median sludge size decreased 61% with hydrogen peroxide treatment. Ozonation only slightly decreased the median diameter of the particle size from 36 to 33 μm [27]. The difference in reduction degree of the sludge size may be due to the fact that the oxidant, concentration of oxidant, and size of the original sludge were all different from each other. In their study, the size of the original sludge was 34.5 μm, but it was 116 μm in our present study. According to Jorand et al. [31] the formation model of the sludge floc consists of primary particles (~2.5 μm), microflocs (~13 μm) and macroflocs (~100 μm). The polymers link the microorganisms inside the primary and microflocs, whereas microflocs and isolated primary particles also link together by the polymers. Therefore the larger macroflocs contain much more polymers than smaller microflocs. Thus, much stronger forces were needed to disintegrate these macroflocs than smaller sludge particles. Potassium ferrate disintegrated the sludge macrofloc into microfloc, resulting in the reduction of the sludge flocs. However, sludge size increased slightly when the dosage of potassium ferrate was 0.81 g/g SS. This increase of the particle size was probably attributed to the re-flocculation by the production of the coagulant, Fe(III), during the oxidation reaction of potassium ferrate.

Initially, the SVI values increased with the increase of the potassium ferrate (Fig. 6). SVI increased from 76 mL/g for the untreated sludge to 109 mL/g for the sludge treated by potassium ferrate of 0.33 g/g SS. It was suggested that the settleability of the sludge was affected by potassium ferrate oxidation pretreatment. However, SVI reduced gradually when the dosage of potassium ferrate was greater than 0.49 g/g SS. After potassium ferrate of 0.81 g/g SS was applied to oxidize the sludge, the SVI value was 63 mL/g, which was even lower than that of the original sludge. The settleability was improved by 17%. Hydrogen peroxide oxidation enhanced the sludge settleability [19]. Sponza [32] found that very small and big flocs show high SVI values indicating poor settling properties. From these results, it could be inferred that the sludge settleability reduction initially was due to the particle size reduction or the increase of viscosity (Fig. 7), and the sludge settleability enhancement subsequently was due to the re-flocculation produced by the coagulant, Fe(III), and the viscosity reduction by potassium ferrate oxidation which are described in Figs. 5 and 7, respectively. The settleability has a significant impact on the dewaterability of the sludge flocs. Therefore, improvement of the sludge settleability can also reduce the sludge volume.

Using other disintegration methods, additional costs are caused by the increased amount of flocculants needed because of the high content of the small particles. However, there is no such problem due to the production of coagulant, Fe(III), resulting in a quite cost effective implementation of this technique.
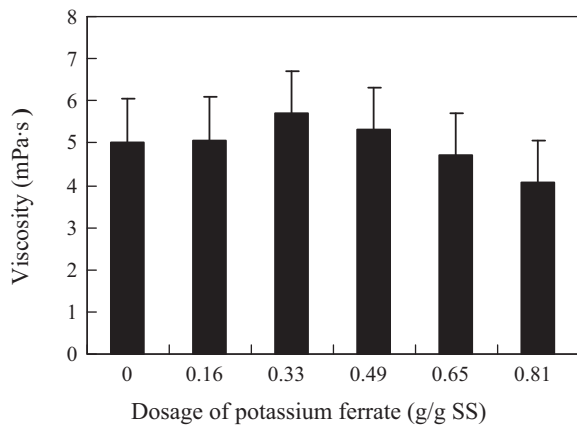
Fig. 7. Effect of potassium ferrate oxidation on the viscosity of the sludge.

Economic-efficiency calculation is important tool for performing the cost-benefit analysis of a disintegration process. Actual investigations show that sludge ozonation is only economic for larger plants with high sludge-disposal costs or of interest if operating problems such as foaming and bulking sludge that could lead to process overload can be reduced. Sludge disintegration could then obviate a plant extension [33]. The application of sludge disintegration must consequently be carefully evaluated. The economic-efficiency calculation comprised investment, operation and maintenance costs as well as the additional advantage due to biogas increase and a cost reduction for sludge disposal. Hence, the cost calculation will be evaluated during our pilot-scale investigation.

The viscosity of the original sludge was 5.034 mPa s. Potassium ferrate oxidation of the sludge increased its viscosity under lower dosage (Fig. 7), which was attributable to disintegration of the sludge aggregates, and the release of intercellular and extracellular polymers. The subsequent part oxidation of these polymers resulted in the reduction of viscosity, and the improvement of the settleability.

4. Conclusions

A novel disintegration approach was applied to disintegrate WAS. The excess activated sludges were pretreated with different dosages of potassium ferrate. Potassium ferrate oxidation led to the destruction of the sludge particle, resulting in TSS reduction, and increase of SCOD, TN and TP in solution. Under 0.81 g/g SS dosage of potassium ferrate, 31% TSS decreased and SCOD/TCOD increased to be 0.32. The sludge particles and viscosity were reduced, but the setteability of the sludge was enhanced by 17% because the voluminous flocs were resized into compact flocs due to the production of coagulant, Fe(III).

Acknowledgements

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