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Enhancement of municipal sludge dewaterability by electrochemical pretreatment

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ABSTRACT

Electrolysis is a promising technology to improve sludge dewaterability efficiently with negligible environmental impact. To intensify the electrolytic efficiency, the effect of electrolytes (NaCl, Na_2SO_4, $NaNO_3$, and $NaClO_4$) on electrolysis pretreatment of municipal sludge and its mechanisms was investigated using Ti/PbO_2 electrodes. The electrolytes, which enhanced the production of oxidative radicals, showed a significant synergetic effect in reducing the capillary suction time (CST) of sludge. NaCl was distinguished from the other electrolytes since it formed a large amount of active chlorine species, which oxidized the sludge cells to improve the sludge dewaterability. The surface morphologies as well as the soluble proteins and polysaccharides were analyzed to unravel the underlying mechanisms of sludge dewaterability. Additionally, an economic assessment showed that NaCl addition in the electrolysis pretreatment can be a suitable technique for enhancing municipal sludge dewaterability.

Introduction

Large amounts of municipal sludge generated from biological wastewater treatment processes have caused environmental pollution. Excess municipal sludge treatment requires large energy inputs, and 25%–60% of the total operating cost in wastewater treatment plants is used for sludge treatment (Zhang et al., 2009). Sludge dewatering, which can potentially reduce the sludge volume and disposal cost, has been a promising strategy for decades (Dogruel and Ozgen, 2017). Numerous chemical and physical reduction technologies targeted on the disintegration of bacterial cells to improve sludge dewaterability have been widely studied in recent years, including Fenton pretreatment (Mo et al., 2015), hydrothermal treatment (Wang et al., 2017), electrical-alkali pretreatment (Zhen et al., 2014), and solar photocatalytic treatment (Liu et al., 2013). Among the above methods, electrolysis is one of the most promising technologies, as electrolysis can disintegrate and improve sludge biodegradability efficiently with negligible environmental impact.

In the past decades, some researchers have devoted a great deal of effort toward improving sludge dewaterability using electrolysis (Barrios et al., 2017). The effects of the electrolysis parameters on sludge dewaterability, such as electrolysis

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voltage and time, have commonly been investigated (Ye et al., 2016); the sludge dewaterability and energy consumption of electrolysis pretreatment were compared with those of other pretreatment methods (Xu et al., 2014). It was found that electrolysis was more suitable for sludge pretreatment than thermal, thermal-alkaline, or alkaline methods (Xu et al., 2014). Optimal CST reduction efficiency was found to occur at electrolysis voltage of 6–21 V and electrolysis time of 10–30 min to give 18.8%–22.15% CST reduction efficiency (Ye et al., 2016; Yuan et al., 2010). Most importantly, the energy consumption of electrolysis pretreatment for enhancing sludge anaerobic digestion was lower than that of microwave, ultrasound, thermal, Fenton or ozone while achieving the same level of methane production (Ye et al., 2016). Previous literature reports have mainly focused on the optimization of operating parameters and changes in the physicochemical properties of municipal sludge during electrolysis pretreatment, while little is known about the electrolysis mechanism and the ways to intensify the electrolytic process when sludge is pretreated by electrolysis. In particular, the use of various electrolytes has been widely reported for electrochemical applications (Clematis et al., 2017; Fajardo et al., 2017). Nonetheless, the effects of electrolytes have not been evaluated in the pretreatment of sludge by electrolysis.

In the electrolysis process, the pretreatment of municipal sludge mainly depends on the properties of electrodes. Consequently, a wide variety of electrodes such as Ti/RuO_2 (Hu et al., 2011; Yuan et al., 2010), Ti/RuO_2-IrO_2 (Li et al., 2016), Ti/IrO_2 (Chen et al., 2011), and diamond-based materials (Barrios et al., 2017) have been studied and found to be effective in sludge pretreatment. Ti/PbO_2 electrodes have become popular due to their low adsorption properties, excellent chemical stability, high oxygen evolution potential, and low cost compared to noble metals (García-Gómez et al., 2016). Hence, there is great interest in the application of Ti/PbO_2 electrodes for the electrolytic pretreatment of municipal sludge.

In this study, the effect of different supporting electrolytes on sludge dewaterability was investigated using Ti/PbO_2 electrodes. The primary concerns of this study were to verify the following questions: (i) Which electrolyte can effectively improve sludge dewaterability in the electrolytic process? (ii) What is the optimal addition amount for the selected electrolyte? and (iii) What is the mechanism of electrolytic sludge pretreatment enhanced by electrolytes? To the best of our knowledge, this is the first time the effect of supporting electrolytes on sludge dewaterability has been investigated in detail.

1. Methods

1.1. *Sludge samples*

Samples of municipal sludge were collected from a secondary sedimentation tank in a wastewater treatment plant located in Shenzhen, China. The plant includes the following processes: degritting, screening, primary sedimentation, anaerobic–anoxic–oxic process, secondary sedimentation, clarification, and disinfection. The fundamental properties of the sludge samples are as following: pH 6.4–6.6, water content 98.9%–99.3%, total suspended solids (TSS) 12.1–12.9 g/L, volatile suspended solids (VSS) 7.3–7.7 g/L, CST 175.6–200.8 sec and total solids 32.5–34.3 g/L.

1.2. *Pretreatment experimental design*

A series of bench-scale experiments were carried out at room temperature (25 ± 5°C) in a plexiglass cell (Appendix A Fig. S1). The apparatus consisted of a highly stable direct-current power supply, a plexiglass cell with dimensions of 12 cm (L) × 4 cm (W) × 12 cm (H), and two metal mesh plate electrodes with dimensions of 10 cm × 10 cm. Both the anode and cathode were Ti/PbO_2 meshes. During this experiment, the sludge was stirred in the plexiglass cell with a magnetic agitator. The stirrer speed was low (100 r/min) in order to prevent floc breakage, but high enough to keep the sludge homogenous and avoid settling of solids.

A 400 mL sludge sample was blended well with various electrolytes, then electrolytically treated to improve sludge dewaterability. A control experiment was conducted under the same experimental conditions with no electrolyte. The oxidative free radicals in the sludge electrolytic treatment were evaluated to investigate the mechanism of sludge dewaterability.

1.3. *Analytical methods*

The CST of sludge samples was evaluated by a CST meter (304B CST, Triton, UK). The CST reduction ratio (R_{CST}, %) was calculated by Eq. (1).

$$R_{CST} = (CST_0 – CST)/CST_0 \times 100\% \quad (1)$$

where CST_0 (sec) and CST (sec) are the CST of municipal sludge before and after the electrolysis pretreatment, respectively.

Electron Spin Resonance (ESR, Bruker A300-10/12, Germany) spectrometry coupled with the spin trapping technique was employed to identify the oxidative radicals generated during the sludge pretreatment (details in Appendix A). For mapping sludge cell morphology, atomic force microscopy (AFM, Bruker Multimode-8, Germany) was applied. Active chlorine was determined by the N,N-diethyl-p-phenylenediamine colorimetric method using a Shimadzu UV-2600 spectrophotometer (Rice, 2012). The concentration of proteins in the sludge supernatant was determined using the Coomassie Brilliant Blue method (Bradford, 1976). The concentration of polysaccharides was determined by the anthrone method (Dubois et al., 1956).

2. Results and discussion

CST, which is inversely proportional to sludge dewaterability, indicates how quickly sludge releases its water (Gharibi et al., 2013). Many studies have found that the efficiency of sludge pretreatment can be determined by the changes in CST (Ning et al., 2015). In this study, the R_{CST}, which is proportional to sludge dewaterability, was selected for study.

The influence of electrolysis voltage and electrode distance on CST was assessed (Appendix A Figs. S2 and S3). The results suggested that the optimum values of the electrolysis voltage and electrode distance were at 20 V and 2 cm, respectively, at which the following experiments were carried out.

2.1. Effect of supporting electrolyte

The influence of supporting electrolyte on the sludge dewaterability was examined through the changes of R_{CST} using four different electrolytes: NaCl, Na_2SO_4, $NaNO_3$, and $NaClO_4$ at the concentration of 6.7 mmol Na^+/L (Fig. 1a). The sludge dewaterability clearly depended on the supporting electrolyte. After 20 min of treatment, the value of R_{CST} increased to 80.6%, 53.3%, 72.8%, and 66.1% with the addition of NaCl, Na_2SO_4, $NaNO_3$, and $NaClO_4$ compared to 32.6% in the control experiment without electrolyte, respectively. The R_{CST} in sludge mixture decreased rapidly from 0% to −114.5%, −63.3%, −20.8% and −31.4% in the first 5 min, and then increased drastically with the addition of NaCl, Na_2SO_4, $NaNO_3$, and $NaClO_4$, respectively. The trend of changes in the R_{CST} was similar to the significant increases in R_{CST} observed after ozonation of sludge (Zhang et al., 2016).

To fully understand the role of electrolytes, the production of oxidative radicals was also monitored after 5 min of electrolysis pretreatment (Fig. 1b). In the presence of free radicals of oxychloride ($^{\cdot}OCl$) in the reaction mixture, the DMPO–OCl adduct should yield seven characteristic peaks (Sundarapandiyan et al., 2014). The ESR spectrum of the DMPO–OH adduct must exhibit a spectrum with the characteristic intensity of 1: 2: 2: 1 (Lei et al., 2015). From ESR spectra, it can be seen that $^{\cdot}OH$ was the dominant oxidative radical in the presence of Na_2SO_4, $NaNO_3$ and $NaClO_4$, while the oxychloride free radical ($^{\cdot}OCl$) was the dominant radical species in the presence of NaCl (Fig. 1b). Compared with the peak intensities in the ESR spectrum in the absence of electrolyte, the addition of electrolytes enhanced the production of oxidative radicals.

2.2. Effect of dosage of NaCl

With respect to the effect of NaCl on sludge dewaterability, different amounts of NaCl ranging from 0 to 14 mmol/L were added into the electrolysis system. As shown in Fig. 1c, after 20 min of electrolysis pretreatment, the R_{CST} was increased from 32.6% at a NaCl concentration of 0 mmol/L to 93.9% with the addition of 10 mmol/L NaCl, and then decreased to 81.4% at the NaCl concentration of 14 mmol/L. In view of both effectiveness and economy, the optimal dosage of NaCl in the present study appears to be 10 mmol/L.

The *in situ* production of active chlorine species (Cl_2, HOCl and $^{\cdot}OCl$) and their roles in electrolysis pretreatment of

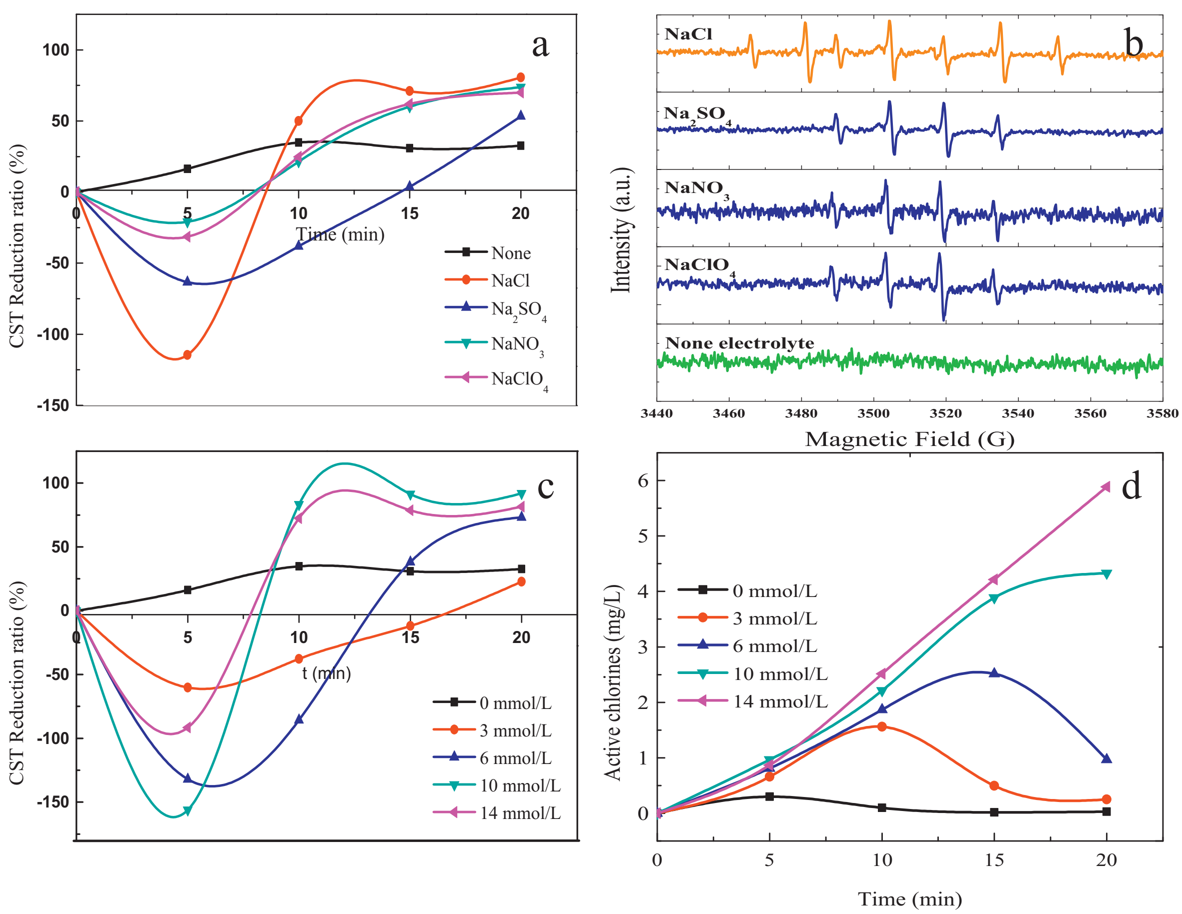

Fig. 1 – (a) Effect of supporting electrolytes on the R_{CST}; (b) Electron Spin Resonance (ESR) spectra of samples collected during electrolysis pretreatment of sludge after 5 min of electrolysis; (c) Effect of NaCl dose on the R_{CST}; (d) Effect of NaCl dose on the production of active chlorine species.

municipal sludge were investigated at different NaCl concentrations, which is presented in Fig. 1d. It was observed that the concentration of active chlorine species increased on increasing the concentration of NaCl in the first minutes. This result was consistent with a previous study, which discovered that a high concentration of Cl^- was beneficial for the production of active chlorine (Zeng et al., 2016). As electrolysis time was extended further, the concentration of active chlorine decreased when the NaCl concentration was lower than 6 mmol/L. This was due to the consumption of active chlorine, which oxidized the sludge cells. However, the increase of R_{CST} was very marginal beyond the NaCl concentration of 14 mmol/L (Fig. 1d). The reason may be attributed to the generation of Cl_2. When the NaCl concentration increased up to a critical concentration, a large amount of Cl_2 was discharged from the anode. Actually, the concentration of Cl in the reactor was measured and found to decrease by 4.50 mmol/L within 20 min. The discharge of Cl_2 reduced the mass transfer of the generated active chlorine into the bulk sludge, leading to a significant decrease of the R_{CST} after 20 min of pretreatment. Several references are available on the effect of the concentration of Cl^- on Cl_2 production (Faxon et al., 2015; Ullal et al., 2002).

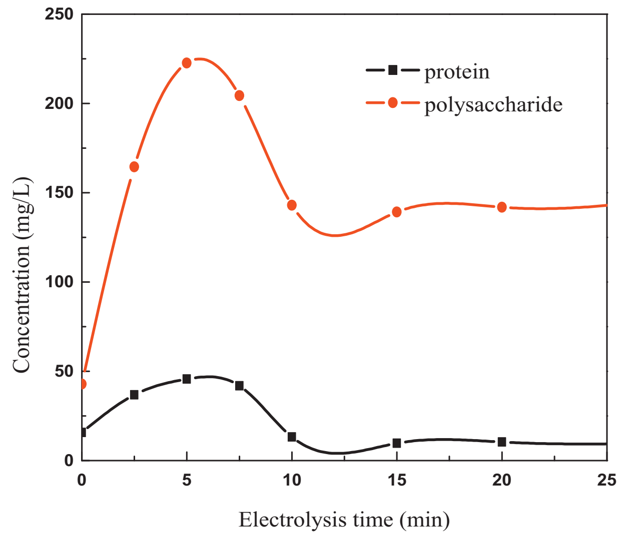

Fig. 3 – Concentrations of proteins and polysaccharides in the supernatant during sludge pretreatment.

2.3. Possible mechanisms of electrolysis pretreatment enhanced by electrolytes

Based on the experimental results mentioned above, the mechanism of electrolysis pretreatment enhanced by electrolyte was studied for addition of 10 mmol/L NaCl.

The morphological characteristics of the original and treated sludge samples were analyzed using AFM (Fig. 2). The surface morphologies of the different sludge samples were distinctly different. The surface of the original sludge cells was visually smooth, and no obvious fragmentation or breakages were observed (Fig. 2a). After the electrolysis pretreatment, the sludge cells became denser and more amorphous, indicating that the sludge cells were disintegrated (Fig. 2b). It was confirmed that the depicted morphological defects were typical for the bulk of sludge samples. It can be deduced that the sludge cells were disrupted by the oxidative radicals generated from the electrolysis system. This observation was consistent with a previous report which found that the intercellular substances such as DNA, proteins and polysaccharides were leaked out of damaged cells treated with a high-pressure jet device (Xie et al., 2016).

It is well known that the major organic components of EPS are proteins and polysaccharides (Li et al., 2017). Therefore, measurements of proteins and polysaccharides in sludge supernatant were critical to the understanding of the effects of electrolyte on sludge disintegration in the electrolysis process (Fig. 3). In the first 5 min, the soluble proteins and polysaccharides increased from 15.6 and 42.9 mg/L in the original sludge sample to around 45.8 and 222.6 mg/L in the supernatant, respectively. This was probably related to the effect of the generated active chlorine on sludge cells. As proteins and polysaccharides are the main constituents of EPS of sludge cells, the oxidation of EPS could result in the release of the polysaccharides and proteins into the supernatant. After 5 min of treatment, the concentrations of soluble proteins and polysaccharides decreased, indicating that some of the released proteins and polysaccharides could be

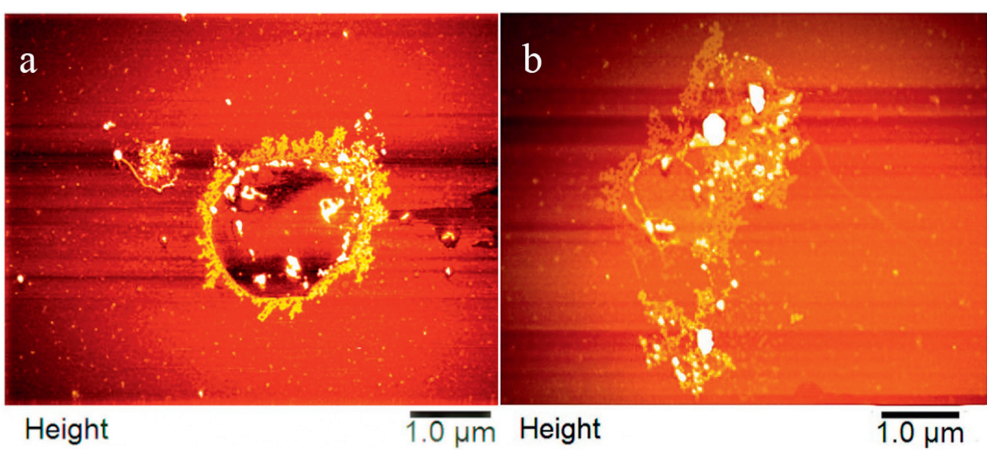

Fig. 2 – Atomic force microscopy images of sludge cells before (a) and after (b) the electrolysis pretreatment.

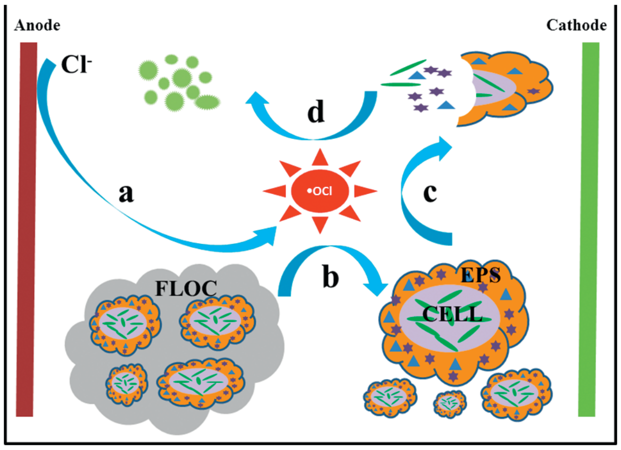

Fig. 4 – Proposed electrolysis mechanisms of the sludge pretreatment enhanced by electrolytes.

degraded by the oxidation from the oxidative radicals. In this study, the increases in the concentrations of soluble proteins and polysaccharides (Fig. 3) were correlated with the decrease of R_{CST} (Fig. 1a). These results were consistent with previous reports, which suggested a direct relationship between EPS content and the dewaterability of municipal sludge (Chen et al., 2017).

Based on the analyses of R_{CST}, supernatant concentrations of polysaccharides and proteins, and the surface morphologies of sludge cells, mechanisms for the electrolytic sludge pretreatment enhanced by electrolytes were proposed, which are shown in Fig. 4. These include: (a) The oxidative radicals were generated in electrolysis process, which was affected by the types and concentrations of the added electrolytes (Fig. 1b and d). (b) The sludge flocs were oxidized and disaggregated into smaller particles. These small particles could associate with more water molecules, leading to decrease of the R_{CST} (Fig. 1a). (c) The EPS outside sludge cells, which played an important role in the sludge dewaterability, were oxidized by the oxidative radicals, resulting in an increase in the supernatant concentrations of proteins and polysaccharides (Fig. 3). (d) As the electrolysis time was extended further, more oxidative radicals were generated and organic substances released from sludge cells could be oxidized into low-molecular-weight organics or even inorganic components, which resulted in a drastic increase of the R_{CST} (Fig. 1a) and a decrease in the concentrations of proteins and polysaccharides in the sludge supernatant (Fig. 3). The four stages and phenomena described above were not completely separable.

2.4. Comparison of different pretreatments for sludge dewaterability

Economic analyses of the electrolysis pretreatment with and without 10 mmol/L NaCl addition for the sludge dewaterability improvement were conducted using a desktop scaling-up method (Appendix A Table S1) (Song et al., 2016; Wang et al., 2018). The total cost of the electrolysis pretreatment with and without 10 mmol/L NaCl addition was 182.9 and 200.6 USD/ton DS (dry solids content) to obtain an R_{CST} of 93.9% and 32.6%, respectively. Table 1 presents a comparison of the economic efficiencies of enhancing sludge dewaterability by 10 mmol/L NaCl addition and electrolysis pretreatment, Fenton oxidation (Buyukkamaci, 2004; Lu et al., 2003), and Fe(II)-PMS oxidation (Liu et al., 2016). The sludge dewaterability via electrolysis with NaCl addition can achieve higher CST reduction efficiency and lower cost than other pretreatment technologies. Compared with Fe(II)-PMS oxidation to enhance sludge dewatering with addition of 0.9 mmol/g VSS of PMS and 0.8 mmol/g VSS of Fe(II), the addition amount for NaCl (10 mmol/L, about 0.5 mmol/g VSS) was low and did not cause environmental problems (Liu et al., 2016). In addition, the cost of NaCl was relatively low in our study. Hence, it is economical and energy-saving to add NaCl in electrolysis pretreatment technology at the industrial scale.

3. Conclusions

The addition of electrolytes to electrolysis pretreatment had a significant effect on improving sludge dewaterability. It was proved that the addition of electrolytes enhanced the production of oxidative radicals. The maximum R_{CST} (93.9%) was obtained with the addition of NaCl (10 mmol/L). The electrolysis mechanisms for the sludge pretreatment enhanced by electrolytes were proposed. Active chlorine species generated from the electrolysis system with the addition of NaCl decomposed sludge flocs, cracked sludge cells, disrupted EPS, and degraded proteins and

Table 1 – Comparison of different pretreatments for sludge dewaterability.

Pretreatment	CST		Oxidant dosage	pH	Total cost (USD/ton DS)	References
	Raw sludge (sec)	Reduction rate (%)				
Fenton	–	–	Fe(II) 6 g/L H_2O_2 3 g/L	3.0	250.6	(Lu et al., 2003)
Fenton	30.5	48.5	Fe(II) 5 g/L H_2O_2 6 g/L	3.0	288.5	(Buyukkamaci, 2004)
Fe(II)–PS	210.0	88.8	Fe(II) 1.5 mmol/g VSS $S_2O_8^{2-}$1.2 mmol/g VSS	6.9	323.7	(Zhen et al., 2012)
Fe(II)-PMS	201.8	90.1	Fe(II) 0.8 mmol/g VSS HSO_5^-0.9 mmol/g VSS	6.8	336.2	(Liu et al., 2016)
Electrolysis	206.9	32.6	None	6.5	200.6	This study
Electrolysis-NaCl	206.9	93.9	10 mmol/L	6.5	182.9	

polysaccharides, resulting in the release of bound water inside cells and the subsequent improvement of sludge dewaterability.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jes.2018.03.007.

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