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Effects of potassium ferrate on extracellular polymeric substances (EPS) and physicochemical properties of excess activated sludge

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ABSTRACT

The activated sludge process of wastewater results in the generation of a considerable amount of excess activated sludge. In many wastewater treatment plants, the bottleneck of the sludge handling system is the dewatering operation. This paper investigated the effect of potassium ferrate pretreatment on the physicochemical properties of the excess activated sludge at various dosages of potassium ferrate. The particle size, extracellular polymeric substances (EPS) content and chemical components, and sludge disintegration degree were measured to explain the observed changes of physicochemical properties. It was expected that potassium ferrate could enhance the filterability and dewaterability of the sludge. However, the results showed that potassium ferrate had a negative effect on the filterability by measuring the capillary suction time (CST), but improved the settleability and dewaterability extent by determining the water content in the dewatered cake, although the flocs size reduced slightly. Loosely bound EPS (LB-EPS) content, polysaccharides (PS) and proteins (PN) contents in LB-EPS all increased with increasing the amount of potassium ferrate. However, Tightly bound EPS (TB-EPS) content, PS and PN contents in TB-EPS did not changed significantly at first, and decreased slightly under higher dosage of potassium ferrate. EPS, especially LB-EPS played more important role in the observed changes of the settleability and filterability than the sludge particle size.

1. Introduction

The biological treatment of wastewater produces large quantities of excess activated sludge, the water content of which is generally greater than 95%. Therefore, it is necessary to dewater the sludge to obtain a product dry enough to allow a reduction in volume, facilitation of transportation, decreasing of energy used in case of drying or incineration. Unfortunately, it is well known that the sludge is hard to be dewatered [1]. Using centrifuges or belt presses, only 20–25% dry solids (DS) can be obtained. Reducing the volume of the sludge and improving the dewaterability are hence of very important. Obtaining higher DS can only be done with pretreatment processes changing sludge structure.

Effective dewatering requires the attack of the microorganisms which live in aggregates (e.g. sludge flocs). A common feature is that the microorganisms are embedded in a matrix of extracellular polymeric substances (EPS). EPS are mainly responsible for the structural and functional integrity of the aggregates and are considered as the key to the physicochemical and biological properties. The total mass of EPS (EPS and water held within the EPS-structure) has been found to represent up to 80% of the mass of the activated sludge, and are the major component of the activated sludge flocs matrix [2]. One of the main reasons for difficulty in activated sludge dewatering is attributed to the presence of EPS. Polysaccharides (PS) and proteins (PN), which entrap the water and cause a high viscosity, are the main compositions of EPS [3]. By binding cells and particulate matter together, EPS also change the particle size distribution of the sludge, which again affects the dewatering property.

The flocs matrix is likely to have a dynamic double-layered EPS structure of loosely bound EPS (LB-EPS) diffused from the tightly bound EPS (TB-EPS) that surrounds the cells [4–6]. Moreover, it was found that only the LB-EPS was related to the sludge dewaterability [5,6].

Considering the high water binding property of EPS and high EPS mass in the activated sludge, it is envisaged that the dewatering efficiency of the activated sludge can be increased by degrading the EPS. From previous experimental results and reviews, advanced oxidation methods improved the dewaterability of the sludge by affecting the EPS in two ways: (1) they have the potential to degrade EPS and; (2) they affect the multifunctional groups of the EPS and promote their participation in several interactions [7,8]. The results by Neyens et al. [8] indicated that the peroxidation of the sludge enhanced the flocculation and dewaterability. The responsible mechanism was not fully understood, but the oxidative pretreatment might be based on particle

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Table 1
Characteristics of the excess activated sludge samples.

Parameters	Average value
pH	6.97
TS (mg/L)	7207
TVS (mg/L)	3686
COD (mg/L)	10,893
Filtrate COD (mg/L)	778
SVI (mL/g)	143
CST (s)	96.6
Water content (%)	99.2

oxidation and rearrangement of the surface components of the sludge flocs.

Potassium ferrate is a strong oxidant reagent that can oxidize organic compounds efficiently [9–11]. It is an environmental-friendly oxidant, commonly applied in wastewater treatment, as it yields no noxious or polluting byproducts. By applying pretreatment with potassium ferrate, organic compounds, such as EPS in activated sludge are destroyed, and ferric coagulating species are generated simultaneously, which may lead to increased flocculation and dewaterability of the sludge. Potassium ferrate oxidation pretreatment may offer a promising technology for the improvement of the dewaterability and reduction of the excess activated sludge. Several researchers have reported that Fenton's reagent and H_2O_2 can improve the dewatering of the activated sludge efficiently [7,12]. However, as far as is known, little has been reported on the effect of potassium ferrate on the physicochemical properties of the sludge, especially dewaterability.

The aim of this study is thus to explore the possibility of applying potassium ferrate pretreatment in increasing the filterability and dewatering efficiency of the excess activated sludge. CST and moisture in dewatered sludge cake were selected as the indexes to investigate the characteristics of sludge filterability and dewaterability. Sludge floc size, EPS content and chemical constituents, sludge disintegration degree were selected as the parameters to explain the mechanism involved in the filterability and dewaterability of the sludge. It will be important to elucidate the role that EPS have in the dewatering phenomena in the sludge will be important in addressing sludge-liquid separation problems.

2. Materials and methods

2.1. Activated sludge samples

The sludge samples were taken from Ningbo municipal wastewater treatment plants in Ningbo, China. Table 1 shows the characteristics of the sludge samples. The sludge was stored in filled plastic containers placed in an ice cooler during the transportation from the plant site to the laboratory. 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. [13] and modified slightly. The final product, solid potassium ferrate was collected and stored in a vacuum desiccator prior to further use.

2.3. Sludge pretreatment with potassium ferrate

The sludge samples (350 mL) in a 500 mL beaker were rapidly mixed with different dosages of potassium ferrate (0.0101, 0.0510, 0.1008, 0.2015, 0.3545 and 0.5178 g, respectively) at a speed of 400 rpm for 3 min to start the pretreatment processes, and then slowly stirred to allow to react for 7 min. The CST, particle size, moisture content in the sludge cake, the LB-EPS and TB-EPS contents and chemical constituents of the treated sludge were investigated. Each test was performed in triplicate.

2.4. Sludge filterability and dewaterability test

The CST was measured by a CST instrument (Triton, Model 304M, Essex, UK) as detailed in APHA [14] with a CST paper purchased from Triton Ltd. The CST for distilled water was stable at 11 s. The test was made in triplicate with a relative standard deviation of 5%. 50 mL sludge samples were centrifuged at 2215 × *g* for 10 min, and then the sludge cake were dried by an oven at 105 °C to evaluate the dewaterability. The solids content was analyzed following the Standard Methods [14].

2.5. Sludge particle size

The particle size was determined by a Malvern Mastersizer/E instrument with a 300 mm lens which enables the measurement of particles in the range of 0.9-546 μm. The activated 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.

2.6. Extraction of EPS and determination of EPS components

The sludge sample was fully mixed prior to commencing the extraction procedure. EPS extraction was carried out using a sonication/thermal extraction process according to Li and Yang [6]. The activated sludge was harvested by centrifugation (6000 × *g*, 10 min) and washed with distilled water prior to extraction to remove the loose slime polymers found in the sludge. The dewatered sludge pellet was resuspended in a 0.05% w/w NaCl solution with several glass beads, sonicated at 20 kHz for 2 min, shaken horizontally at 150 rpm for 10 min, sonicated again for an additional 2 min. The liquor was centrifuged at 8000 × *g* for 10 min to separate solids and supernatant. The collected supernatant was regarded as the LB-EPS of the sludge sample (LB-EPS). The residual sludge pellet left in the centrifuge tube was resuspended in a 0.05% (w/w) NaCl solution, sonicated for 2 min, then heated at 60 °C for 30 min, finally centrifuged at 11,000 × *g* for 30 min to collect supernatant. The collected supernatant was regarded as the TB-EPS in the sludge sample (TB-EPS).

2.7. 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 suspended solid (SS) and chemical oxygen demand (COD) were determined according to APHA [14]. DD_{SCOD} was defined by Li et al. [15], comparing the potassium ferrate oxidation and the maximum soluble COD ($SCOD_{NaOH}$).

$$DD_{SCOD}(\%) = \frac{SCOD_S - SCOD_{S0}}{SCOD_{NaOH} - SCOD_{S0}} \times 100$$

$SCOD_S$ and $SCOD_{S0}$ values are for treated and untreated sludge samples, respectively. $SCOD_{NaOH}$ was obtained via an alkaline hydrolysis procedure in which the initial sludge samples was mixed with 0.5 M NaOH at room temperature for 24 h.

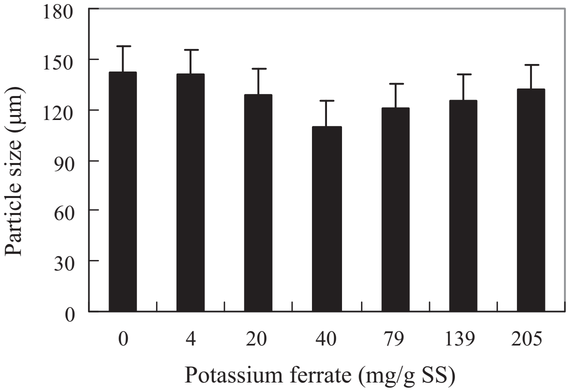

Fig. 1. Effect of potassium ferrate pretreatment on average particle size of the excess activated sludge.

The LB-EPS and TB-EPS extractions were analyzed for total organic carbon (TOC), protein and polysaccharide. TOC was measured by a TOC analyzer (Elementar, Liqui TOC/TNb, Germany), and expressed as mg TOC/g SS. Proteins were determined by an adaptation of the Lowry method [2] using casein (Shanghai Sangon Biotechnology, China) as the standard. Polysaccharides were determined using the anthrone method with a glucose standard. All samples were made in triplicate.

2.8. Statistical analysis

Statistical analysis was carried out with SPSS software version 11.0 for Windows (SPSS, Chicago, IL, USA). Correlations were considered statistically significant at a confidence interval ($P < 0.05$).

3. Results and discussion

3.1. Effect of potassium ferrate on sludge physicochemical characteristics

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 (XRD figure not shown). The purity of the potassium ferrate sample measured by UV/vis spectroscopy method was 99%.

One of the main influences on the sludge dewaterability is the particle size distribution [16]. Flocculation changes the particle size distribution of the sludge, binding small particle together, thereby influencing the sludge dewatering characteristics. As shown in Fig. 1, initially, the flocs size decreased with the increase of potassium ferrate. The sludge particle size reduced by 23% when potassium ferrate of 40 mg/g SS was added, but increased slightly only when the dosage of potassium ferrate was greater than 40 mg/g SS. Compared with the untreated sludge, the flocs sizes were all low regardless of different dosages of potassium ferrate. Similar changes in particle size also were observed by Kwon et al. [17] with Fenton reagent and Kim et al. [18] with hydrogen peroxide. Ozonation did not appear to greatly affect particle size at lower ozone doses [19–21]. Higher ozone doses resulted in an increase in small particles. Park et al. [22] reported that the mean particle size decreased from 70 mm to 40 mm with an ozone dose of 0.5 g O_3/g TSS. With a further ozone dose, the highest peak of particle size distribution was identified around 5 mm [23].

According to Jorand et al. [24], the formation model of sludge floc consists of primary particles, microflocs and macroflocs. The polymers II link the microorganisms inside the primary and microflocs, whereas the polymers I may link microflocs together and also link isolated primary particles. The polymers I and II correspond to LB-EPS and TB-EPS in present study, respectively. Potassium ferrate disintegrated the sludge macrofloc into microfloc, resulting in the reduction of the sludge flocs. However, under high dosage of potassium ferrate, the increase of particle size was probably attributed to the re-flocculation by the production of the coagulant, Fe(III), during the oxidation reaction of potassium ferrate.

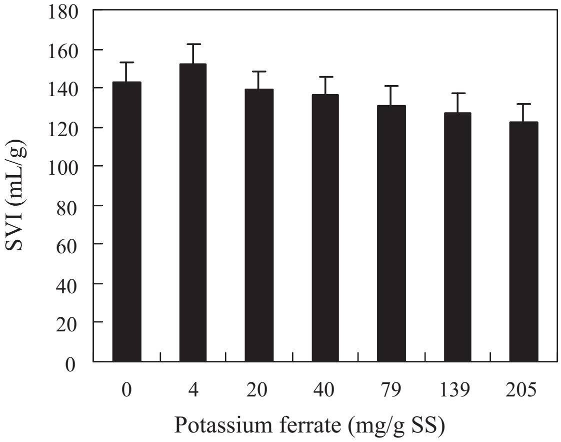

Fig. 2. Effect of potassium ferrate pretreatment on the settleability of the excess activated sludge measured by SVI.

In general, the sludge with large size and compact structure has good flocculating ability, resulting in good settleability. Flocculation does not only affect the settleability of the sludge, but it also has a significant impact on the dewaterability of the sludge flocs. Therefore, larger particle size can reduce CST values and improve dewaterability.

The SVI values decreased with the increase of the potassium ferrate (Fig. 2), suggesting that the settleability of the sludge was improved by potassium ferrate pretreatment. SVI decreased from 142.9 mL/g for the untreated sludge to 122.1 mL/g for the sludge pretreated by potassium ferrate of 205 mg/g SS. Hydrogen peroxide oxidation also enhanced the sludge settleability [18]. The strongly flocculated flocs have higher degree of compressibility of the activated sludge determined as SVI. Sponza [25] 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 enhancement was due to the re-flocculation produced by the coagulant, Fe(III), in spite of the reduction of the flocs size.

The CST values increased with the increase of potassium ferrate, especially when the dosage was high. The CST value of the excess sludge which did not treated by potassium ferrate was 96.6 s, but CST value mounted up rapidly to be 297 s when potassium ferrate of 20 mg/g SS was added. The filterability of the sludge was reduced by 207% at 205 mg/g SS of potassium ferrate, as compared to that of the untreated sludge (Fig. 3). The great increases of the LB-EPS (Fig. 5) concentration made by potassium ferrate oxidation might explain the observed adverse impact of the sludge filterability measured by CST. The small sludge particle may clogged the filter paper, resulting in reduction of the filterability. EPS have a high affinity for water and thus are highly hydrated. High EPS concentrations therefore increased the viscosity of sludge and decreased its filterability. The similar results were observed by ultrasound conditioning. Zhang et al. [26] found that slight reductions in floc size and slight increase in EPS concentration improved sludge dewaterability after lower ultrasound dosage. But sludge dewaterability was significantly deteriorated after higher ultrasound energy dosage due to the disruption of floc structure and integrity, and to the release of intracellular and extracellular materials [27]. As shown in Fig. 3, the moisture content in dewatered sludge cake reduced slightly when

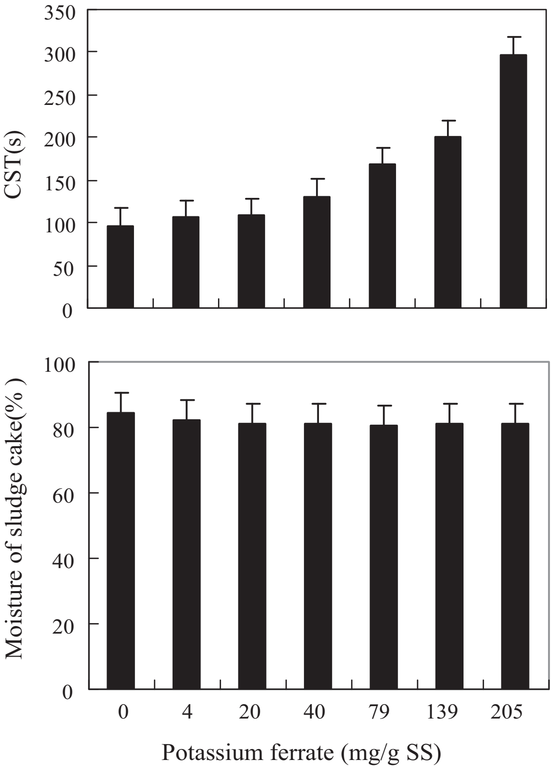

Fig. 3. Effect of potassium ferrate pretreatment on the filterability measured by CST and dewaterability measured by the moisture of the sludge cake.

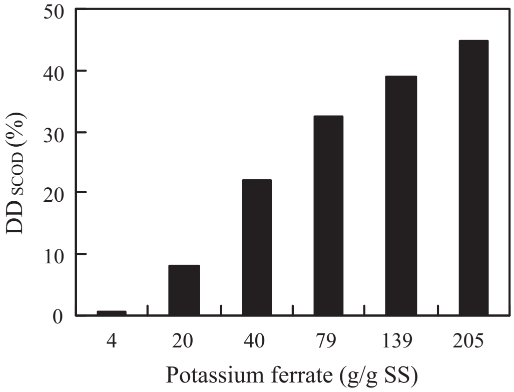

Fig. 4. Effect of potassium ferrate pretreatment on the disintegration level of the excess activated sludge.

the excess sludge was pretreated by potassium ferrate. For example, when the addition of potassium ferrate was 79 mg/g SS, water content in cake was 80.6%, which was reduced about 5% compared with the untreated sludge. Mechanical dewatering is significantly improved after hydrogen peroxide treatment [28]. CST and specific resistance to filtration (SRF) values all decreased after Fenton's reagent conditioning [29].

According to the CST data, potassium ferrate was detrimental to the filterability of the sludge, but was favorable, at least not detrimental, to the dewaterability extent based on the moisture content in sludge cake. In fact, the dewatering efficiency of the activated sludge depends on not only the rate of dewatering, but also the extent of dewatering. The rate of dewatering is usually measured in the laboratory using a CST test or SRF, whereas the extent of dewatering is usually measured by the dewatered cake solids as the percent dry solids or water content. It is possible therefore that the activated sludge is easily filterable, but there is a high amount of residual water in the dewatered sludge [30]. The present study implied that the sludge pretreated by potassium ferrate was not easily filterable, but the extent of dewaterability was not affected. The water classification is more complex and is classified into four categories, including free water, interstitial water, vicinal water and water of hydration [31]. When the sludge aggregates are disintegrated, interstitial water, vicinal water, water of hydration, and EPS bound between these macroflocs are all released. The reduction of the filterability might be attributed to the increased viscosity of LB-EPS, interstitial water bound again by these increased LB-EPS, and/or smaller particles. The original release of bound water was offset by the increase of LB-EPS and the reduction of the particle size. However, the release of bound water between these macroflocs aided to improve the extent of dewaterability measured by centrifugal method because the interstitial water bound again by released LB-EPS can be removed by centrifuges. The original sludge floc not treated by potassium ferrate was large in size, but contained a lot of bound water. Even if the water passed through the filter cake quickly (e.g. CST was low), the water content inside small pores and capillaries, as well as water bound inside the floc matrix, might remain high (e.g. moisture content was high). The treated sludge particles were smaller and sludge was sticky (e.g. CST was high), but the vicinal water which could not removed by centrifugation or other mechanical means was released and reduced accordingly (e.g. DS content was high). The present results again confirmed our previous study that the CST strongly related to the "free" water in the activated sludge, whereas the bound water content was related to the DS content in the sludge cake. The results also suggested that CST test alone is inadequate to determine the grade of separation of sludge solids from the water, and another parameter indicating the dewatering extent, such as DS content (or moisture content) in sludge cake should be applied together with it.

The sludge disintegration degree increased with the dosage of potassium ferrate (Fig. 4). This was due to the fact that floc structure was disrupted and extracellular and intracellular biopolymers were released. According to Yu et al. [32], a proper sludge disintegration degree was vital for the sludge dewaterability based on CST and SRF. They found that approximately 1.5–2% of DD_{SCOD} was the optimum value during microwave irradiation to improve the dewaterability of the sludge. In present study, the higher DD_{SCOD} possibly resulted in the increase of CST and reduction of the filterability of the sludge.

3.2. Effect of potassium ferrate pretreatment on EPS production and components

The precise role of EPS on dewaterability usually appeared ambiguous. An increase in the level of EPS caused the sludge to become more difficult to dewater [33]. However, there are findings that the CST decreased as the amount of EPS increased [34], and no simple relationship between the quantity of EPS present and the sludge dewaterability was observed [4]. To understand the action of potassium ferrate oxidation technology, the essential role by EPS needs to be understood.

In present study, the LB-EPS content did not change significantly after the sludge was pretreated by low dosage of potassium ferrate, and increased substantially when potassium ferrate dosage was beyond 40 mg/g SS (Fig. 5). When the sludge disintegrated further due to higher dosage of potassium ferrate, the sludge flocs were broken and cells destroyed, and intercellular materials were released. The TB-EPS were also diffused from inner layer of the flocs to the outer layer. As a result, the content of LB-EPS increased greatly. The contents of TB-EPS did not change basically when the potassium ferrate oxidation was not strong enough because TB-EPS lied in inner of the sludge flocs. Whereas the weak forces binding the EPS components together could not stand the strong

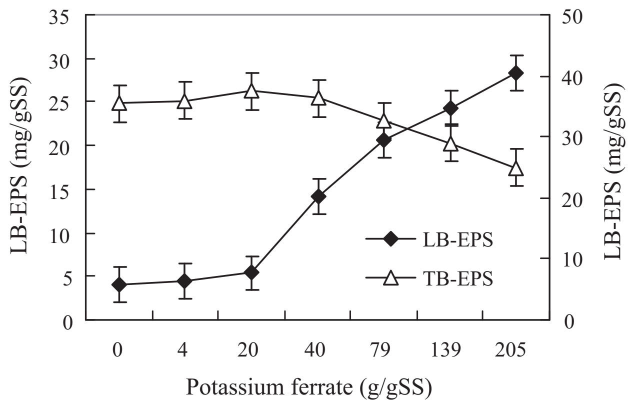

Fig. 5. Effect of potassium ferrate pretreatment on the LB-EPS and TB-EPS contents of the excess activated sludge.

oxidation of potassium ferrate resulted from higher dosage. The colloidal stability of the floc was destroyed, resulting in the release of the TB-EPS into LB-EPS. The above results suggested that one of the reasons for potassium ferrate pretreatment worsening filterability and improving dewatering of the sludge was the sludge aggregates to be disintegrated, more bound water trapped into aggregates to be released, and more TB-EPS transformed to be LB-EPS. These smaller particles and sticky sludge suspension made it difficult to pass the filter paper, resulting in high CST values, and reduction of the filterability. It was pity that the pretreatment and conditioning by other advanced oxidation method, e.g. Fenton's reagent, did not report the information about EPS of the sludge. Therefore, the results were not compared with other investigations.

PS in LB-EPS increased gradually with the increase of potassium ferrate. However, those in TB-EPS increased slightly with the increase of potassium ferrate at first, and then decreased slightly. PS in LB-EPS increased about 4 times at 40 mg/g SS of potassium ferrate, but PS in TB-EPS did not show any significant change. Similar results were also observed for PN in LB-EPS and TB-EPS. Initially, it did not change obviously at low dosage, and then increased sharply under high dosage. In the case of dosage of 40 mg/g SS, PN in LB-EPS increased by approximately 6 times. PN in TB-EPS did not changed significantly, and then decreased under high dosage (Fig. 6).

Based on the above results, we found that the dosage lower than 20 mg/g SS did not cause any obvious variation, but beyond 20 mg/g SS of potassium ferrate, the parameters changed significantly. Under the present dosage, potassium ferrate did not exhibit very strong oxidation to oxidize EPS in the sludge, which may be due to the low dosage, or the oxidation by itself and decomposed before it oxidized EPS. Detailed investigation should be done in the future.

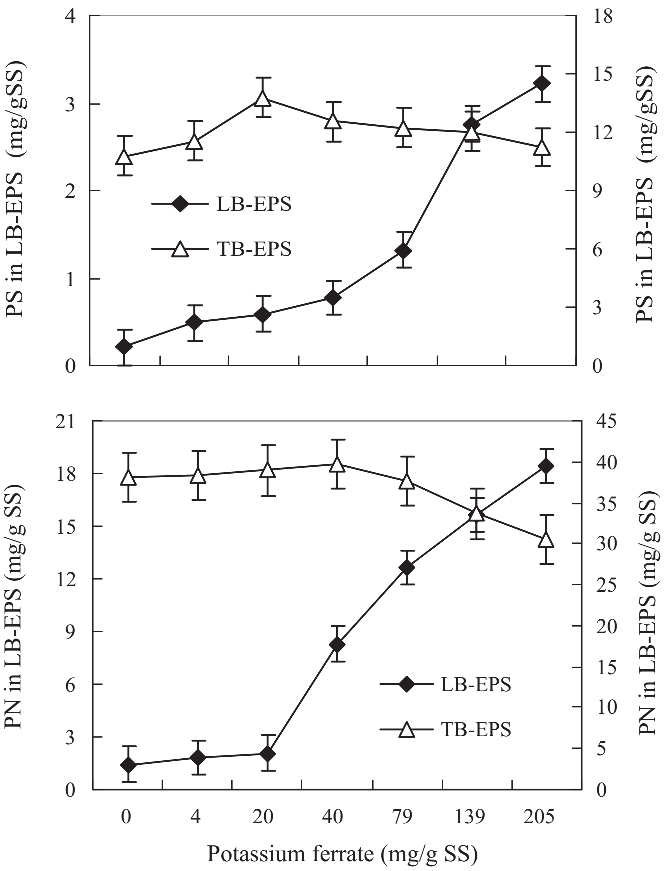

Fig. 6. Effect of potassium ferrate pretreatment on the polysaccharides (PS) and protein (PN) contents in LB-EPS and TB-EPS of the excess activated sludge.

3.3. Relationship between physicochemical properties parameters

Jin et al. [34] and Higgins and Novak [35] have reported that the "supracolloidal" particles in the range of 1–100 μm had the greatest negative effect on the sludge dewaterability, and that as the concentration of the particles in this size range increased, the dewaterability decreased. In this study, the particle size and filterability of the sludge all decreased, but no correlation between CST and particle size was found (Table 2).

The establishment of a relationship between sludge dewaterability and the EPS content will enable decisions to be made that ensure maximum dewaterability of the excess activated sludge. It was widely reported that the EPS impacted the sludge dewaterability [33,34,36]. The presence of LB-EPS had a clearly negative effect on dewaterability, and no correlation was found between TB-EPS and dewaterability based on measurements of the SRF

Table 2
Summary of Pearson's correlation coefficient (R^2) and P-value between flocs physicochemical parameters.

Parameters	SVI (mL/g)		Moisture content in sludge cake (%)		CST (s)	
	R^2	P	R^2	P	R^2	P
Particle size (μm)	0.211	0.12	0.531	0.25	0.0238	0.25
LB-EPS (mg/g SS)	−0.900	0.02	0.430	0.14	0.964	0.01
TB-EPS (mg/g SS)	0.777	0.10	0.126	0.26	−0.973	0.01
LB-EPS/TB-EPS	−0.888	0.03	0.339	0.22	0.920	0.01
PS in LB-EPS (mg/g SS)	−0.855	0.05	0.281	0.25	0.998	0.01
PN in LB-EPS (mg/g SS)	−0.893	0.04	0.414	0.14	0.967	0.01
PS in TB-EPS (mg/g SS)	0.000	0.39	0.323	0.21	−0.278	0.18
PN in TB-EPS (mg/g SS)	0.746	0.16	0.0717	0.38	−0.954	0.01
PN/PS in LB-EPS	−0.279	0.33	0.0521	0.26	0.031	0.13
PN/PS in TB-EPS	0.728	0.15	0.611	0.11	−0.685	0.11

[6,36]. Table 2 shows that LB-EPS, LB-EPS/TB-EPS, PS in LB-EPS, PN in LB-EPS were negatively correlated with SVI, and positively with CST. However, TB-EPS, and PN in TB-EPS were negatively correlated with CST. No correlation between the parameters and the moisture content in sludge cake was found. It was suggested that the increase of LB-EPS and its chemical components would favor the settleability, but worsen the filterability of the sludge. The results are in agreement with our previous works and other works [6,36]. However, it was surprised to find that the TB-EPS and its PN content would improve the filterability of the sludge. It may be explained that the TB-EPS was in inner layer of the sludge floc, and high level of TB-EPS could aggregate the floc and make the sludge particle more compact. It was previously observed that, when EPS concentration was increase to a sufficient level, there was a direct impact on sludge dewaterability that was unrelated to the sludge particle size distribution [37]. Therefore, of these two factors, EPS concentration, especially, LB-EPS, and particle size, the former was deemed more influential with respect to sludge flocculation, settleability and dewaterability of the sludge.

4. Conclusions

The effects of potassium ferrate pretreatment on the physicochemical characteristics of the excess activated sludge were investigated with different dosages of potassium ferrate. The following conclusions may be drawn from this study:

(1) Potassium ferrate pretreatment slightly enhanced sludge settleability measured by SVI and dewaterability measured by DS content, but was detrimental to the filterability of the sludge based on the CST results.
(2) Low dosage did not lead to significant change of LB-EPS and TB-EPS contents, while higher dosage caused the increase of LB-EPS, PS and PN in LB-EPS, and reduction of TB-EPS and PN in TB-EPS.
(3) LB-EPS, PS and PN in LB-EPS, and the ratio of LB/TB were correlated significantly with SVI. LB-EPS, LB/TB, PS and PN in LB-EPS were positively correlated with CST. But TB-EPS, PN in TB-EPS were negatively correlated with CST.
(4) The greatly increased LB-EPS concentration was determined to be the major reason for the observed changes in sludge settleability and filterability.

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