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Evaluation of a pretreatment method using cation exchange resin to enhance the sludge solubilization and disintegration for improving the efficiency of anaerobic digestion

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ABSTRACT

Cation exchange resin (CER) has been widely utilized as a method to extract extracellular polymeric substance. It was demonstrated in the author's preliminary study that CER could also be used as a sludge pretreating method to enhance the efficiency of subsequent anaerobic digestion. In this study, the solubilization and disintegration of sludge during CER pretreatment were reported. Proteins, polysaccharides, fluorescent dissolved organic matter (DOM), and molecular weight distribution of DOM were measured to assess and explain the solubilization of sludge. Particle-size distribution and scanning electron microscope were determined to evaluate the disintegration of sludge. Besides, it also indicated that the dewatering and settling ability of sludge were deteriorated. Proteins, polysaccharides, particle size, and monovalent to divalent cation ratio were revealed to have significant correlations with the sludge dewaterability and settleability during the CER conditioning process.

Keywords: Sludge; Pretreatment; Cation exchange resin; Dissolved organic matter; Disintegration; Dewaterability

1. Introduction

In China, the amount of sewage sludge produced in activated sludge technology has increased to almost 30,000,000 tons (80% moisture content), but 80% of them were stabilized incompletely [1]. Thus, the wastewater treatment plants (WWTPs) are facing a rising challenge for excess biomass treatment and disposal. Anaerobic digestion (AD) is the most cost-efficient method due to its high-energy recovery and limited environmental impact. However, the application of AD to biosolids was limited by long retention times (20–30 d) and a low degradation efficiency of organic matters (30–50%) [2]. Hydrolysis in the procedure of AD was reported to be the limiting step [3,4], for most of the organic matters were compartmentalized within the microorganisms [5], and the microbial-originated extracellular polymeric substance (EPS) had many sites to adsorb organics [6].

In order to accelerate the progress of hydrolysis and enhance the efficiency of AD, diverse sludge pretreatment disintegration, including mechanical,

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physical, thermal, chemical, and biological actions, have been investigated. These techniques may induce the destruction of floc structures and the release of EPS. The binding between EPS and divalent cations, such as Ca^{2+} and Mg^{2+}, is one of the main intermolecular interactions in maintaining the microbial aggregate structure [7]. Previous studies have revealed that cations can be removed from the sludge matrix by CER, thereby releasing floc-bound EPS to the aqueous phase [8–12]. Nevertheless, most of these studies have only utilized CER as a method to extract EPS to investigate its characters. Very few publications discussed the feasibility to use CER as a sludge pretreatment method prior to AD. In fact, CER pretreatment was cost-efficient due to the recycle of cationic resin, and it is convenient to operate.

In the authors' preliminary study (unpublished), a lab-scale pretreatment device pretreating waste activated sludge (WAS) by Na^+ form cationic resin was set up. It was revealed that CER conditioning could remove most of the bivalent cations in the sludge, release the biopolymers incorporated in the tightly bound EPS to the bulk, and enhance the efficiency of the subsequent AD. However, the changes of sludge properties during CER pretreating were not investigated.

As a result, in the present paper, the solubilization and disintegration of sludge during CER conditioning were investigated. It was well-known that protein, polysaccharide, and humic substance were the major components of EPS [13–15]. In order to assess the solubilization of sludge, the concentrations of protein (PN) and polysaccharide (PS) were estimated, fluorescent dissolved organic matter (FDOM) was measured by the three-dimensional excitation–emission matrix (EEM) fluorescent spectroscopy, and the variation of molecular weight (MW) distribution of dissolved organic matter (DOM) was investigated. Besides, in order to better understand the disintegration of sludge flocs, a particle size and shape analyzer, and a scanning transmission electron microscope (SEM) were employed to analyze particle-size distribution and the surface characteristics of flocs, respectively. The dewaterability and settleability of WAS, as well as their correlations with PN, PS, and particle size were also studied. The results of the research are expected to help further our knowledge on the enhancement of sludge solubilization and disintegration using this innovative pretreatment method.

2. Material and methods

2.1. Source of WAS

WAS were collected directly from Quyang wastewater treatment plant located in Shanghai, China. The plant treats mainly domestic wastewater, and the treating process is PASF (removes phosphorous by activated sludge and biofilm technology). The sludge samples were collected from the aeration tank of this WWTP. The major characteristics of the original WAS were: total suspended solids of 10.82 ± 1.35 g/L, volatile suspended solids of 6.84 ± 0.65 g/L, and pH of 7.8 ± 0.1. All sludge and wastewater samples were stored at 4℃ before use, and all experiments were completed in 48 h.

2.2. Experimental setup

A lab-scale installation, shown in Fig. 1, was used to pretreat WAS in this work. Raw sludge was put into the contact reactor which was a 6.0 L cylindrical receptacle and the haptoreaction took place here with about 100 rpm, mixing by a stirrer (JJ-1, Guohua, China). And then a peristaltic pump (BT100–2 J, Baoding Longer, China) was used to convey the sludge to the settling tank, which was a 7.0 L cylindrical container with a cone bottom. After sedimentation, the sediments were conveyed to the contact reactor by another identical peristaltic pump, while the supernatant was filtrated through the membrane system equipped with 0.1 m^2 flat-sheet membranes (SINAP, porous size of 0.1 μm). Then, the bivalent and trivalent cations in the filtrate were removed by the CER whose

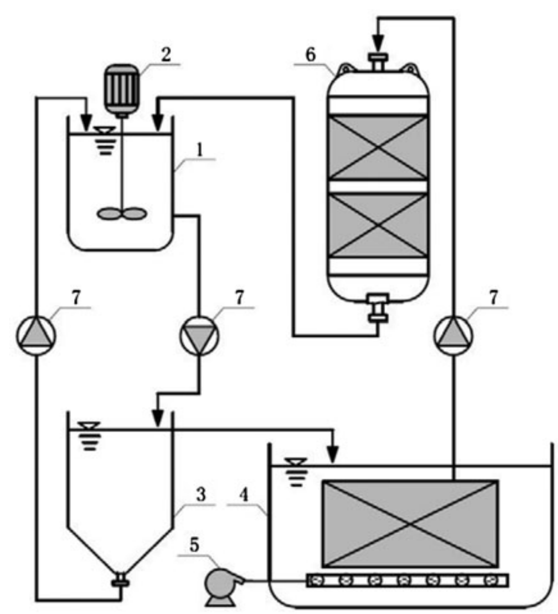

Fig. 1. Diagram of the lab-scale CER pretreating deceive. 1: contact reactor, 2: stirring system, 3: settling tank, 4: membrane system, 5: blower, 6: resin softener system, 7: peristaltic pump.

average particle size was 0.50–0.65 mm in sodium form (IRC748, Rohm & Haas, German), and after that the filtrate was refluxed to the contact reactor. Additionally, the membrane module was cleaned through aeration.

The pretreatment process was circulatory with a flow rate of 6.0 L/h, and sludge was sampled once in an hour. In order to deduct the influence of shear force, a blank test was carried out in another 6.0 L cylindrical vessel at the same rotation rate (100 rpm) when the CER pretreatment was running.

Besides, the resin column in the CER pretreating device was taken to regenerate the resin after CER pretreatment. Firstly, 15 L HCl solution of 2 M was used to filter the resin column with a speed of 10.0 L/h. And then the resin column was filtered by 15 L deionized water with a speed of 10.0 L/h. After that 15 L NaOH solution of 2 M was used to turn the resin into sodium form with a speed of 10.0 L/h. And the resin was finally washed by 15 L deionized water with a speed of 10.0 L/h.

2.3. Analytical methods

Proteins and polysaccharides were all determined spectrophotometrically using a spectrophotometer (photolab 6600 UV–vis, WTW, EU). Proteins were determined using Lowry method [16] with BSA as standard, while polysaccharides were quantified by the Anthrone method [17] with glucose as standard.

A luminescence spectrometry (F-4500 FL spectrophotometer, Hitachi, Japan) was used to measure the EEM of the filtrate obtained after centrifugation (13,000 rpm, 20 min, 4℃) and filtration (< 0.45 μm). The excitation and emission slits were maintained at 10 nm, the scanning speed was set at 1,200 nm/min, and the voltage of the photomultiplier tube was kept at 700 V. All samples with pH values were adjusted to 8.0 before EEM analysis, using 0.1 M HCl solution [18]. EEM spectra were collected with subsequent scanning emission spectra ranging from 300 to 550 nm at 5 nm increments through varying the excitation wavelength from 200 to 400 nm at 5 nm increments. To excise the second-order Raleigh light scattering, a 290 nm emission cutoff filter was used [19,20]. Additionally, the spectrum of deionized water was recorded as the blank. After EEM measurement, the method of fluorescence regional integration (FRI) was applied to extract valuable information about FDOM transformation and compositions [19–22]. The software MatLab 7.0 (MathWorks Inc., USA) was employed to deal with the EEM data.

Filtrate, obtained after centrifugation (13,000 rpm, 20 min, 4℃) and filtration(< 0.45 μm), were fractionated by a gel filtration chromatography analyzer, which consists of a TSK G4000SW type gel column (TOSOH Corporation, Japan) and a liquid chromatography spectrometer (LC-10ATVP, SHIMADZU, Japan). Polyethylene glycols (PEGs) with MW of 1,215,000, 124,700, 11,840, and 620 Da (Merck Corporation, Germany) were used as standards for calibration. The elution at different time intervals were collected by an automatic fraction collector, and then analyzed automatically by UV spectroscopy and a dissolved organic carbon analyzer to obtain a MW distribution curve.

Floc particle size was measured using an EyeTech-combo Particle Size and Shape Analyzer with particle size detection range of 0.1–600 μm. Samples were diluted and shaken up before analysis to obtain appropriate particle counts.

Before the surface observation of the sludge samples using SEM equipped with an electron microscope (XL30, Philips, Holland), the samples were washed with 0.2 M phosphate buffer solution for tree replicates and fixed with 10% formaldehyde solution for 4 h at 4℃, then dehydrated by successively passing through 20, 40, 60, 80, and 100% ethanol. To obtain microscopy images of the granules clearly, the dewatered samples were frozen at −20℃ for 24 h, followed by drying in a vacuum freeze-drying instrument (LGJ-10, Ningbo, China).

The dewatering properties of WAS before and after pretreating were characterized by normalized capillary suction time (CST). CST values were measured by CST apparatus (Type 304, Triton, UK) equipped with an 18 mm diameter funnel and CST papers (9 cm × 7 cm, length × width). These values were then corrected by dividing them by the mixed liquor suspended solids [23,24]. The settling ability was determined by SVI in accordance with standard methods [25].

2.4. Statistical analysis

In this work, statistical analysis was carried out by the software SPSS version 11.0 for Windows (SPSS, Chicago, IL, USA). Pearson's correlation coefficient (r_p) was used to estimate the linear correlations. The value of r_p is always between −1 and + 1, where −1 means a perfect negative correlation, +1 denotes a perfect positive correlation and 0 indicates absence of relationship. Correlations were considered statistically significant at 95% confidence interval ($p < 0.05$). The blank and CER pretreatment were performed in triplicate, and then average values and standard deviations were obtained.

3. Results and discussion

3.1. Effect of CER pretreating method on the sludge solubilization

Fig. 2 showed the effect of CER pretreatment on the concentrations of both PN and PS in the sludge supernatant. The PN and PS in the raw sludge supernatant were measured at 33.92 g COD/L and 7.76 g COD/L, respectively. And the PN and PS of the blank were ranging from 33.92 to 56.02 g COD/L and 7.76 to 10.57 g COD/L, respectively. It is obvious that the PN increased by 379% after pretreated for 1 h and 545% for 3 h, and the level of PN was relatively steady after pretreated for 3–5 h. While the PS increased by 213% after pretreated for 5 h. As a result, it was revealed that organic matters in the inner fraction of the sludge flocs were transformed into soluble and released to the slime, owing to the effect of CER pretreatment, whereas the effect of shear force on the release of protein and polysaccharide in the sludge was negligible.

According to the FRI analysis, the EEM spectra in this report were divided into five regions (Fig. 3), each region was associated with different compounds: simple aromatic proteins (I and II), soluble microbial byproduct-like material such as tryptophan (III), fulvic-like acid (IV), and humic acid-like organics (V). Compounds of regions I, II, and III could be categorized as protein-like substances. A multiplication factor for each region (MF_i), which was defined to be equal to the inverse of the fractional projected area per region [19], was calculated in Table 1. Normalized EEM area volumes ($\Phi_{i,n}$, $\Phi_{T,n}$,) and percent fluorescence response ($P_{i,n}$) were then calculated. The $P_{i,n}$ values of each region were shown in Fig. 4.

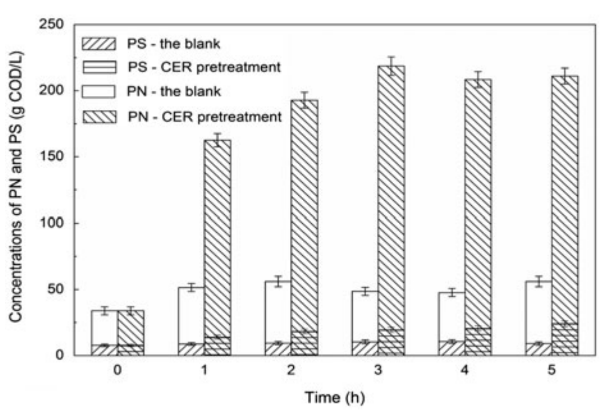

Fig. 2. Effect of CER pretreatment on proteins (PN) and polysaccharides (PS) in the sludge supernatant.

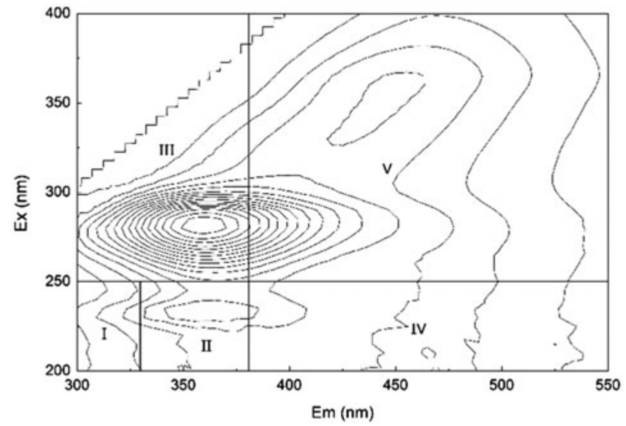

Fig. 3. Regions operationally defined in fluorescence EEM contours of FDOM.
I and II: simple aromatic proteins, III: soluble microbial byproduct-like material, IV: fulvic-like acid, V: humic acid-like organics.

Table 1
FRI parameters for operationally defined EEM regions

EEM region	No. of EEM data points per region (N)	Projected excitation–emission area (nm^2)	Fractional projected area per region	MFi
I	77	1,500	0.033	30.30
II	110	2,500	0.055	18.18
III	374	8,500	0.189	5.29
IV	1,008	7,200	0.160	6.25
V	312	25,350	0.563	1.78
Summation	1,881	45,050	1	

No significant changes of the $P_{i,n}$ value for each region was observed for the blank, implying that stirring had little influence on the composition of FDOM. While slight decrease in $P_{i,n}$ were observed for regions I and II of the CER pretreatment from 12.1 to 9.9% and from 25.3 to 23.3%, a significant increase from 19.6 to 34.1% and a remarkable decrease from 21.3 to 15.5% were shown for regions III and IV. Besides, the $P_{i,n}$ for region V of the CER pretreatment was fluctuant ranging from 15.3 to 21.7%.

The regions I, II, and III were categorized to be protein-like region, and the sum of $P_{i,n}$ for these three regions was regarded as the percentage of protein-like region. As shown in Fig. 4, protein-like region percent of the blank increased slightly. Whereas, a significant increase of the protein-like region was observed after pretreating CER for 1 h, and the $P_{i,n}$ values were reduced slimly after 2 h of pretreating. The Pearson's correlation coefficients (r_p) of the protein-like region

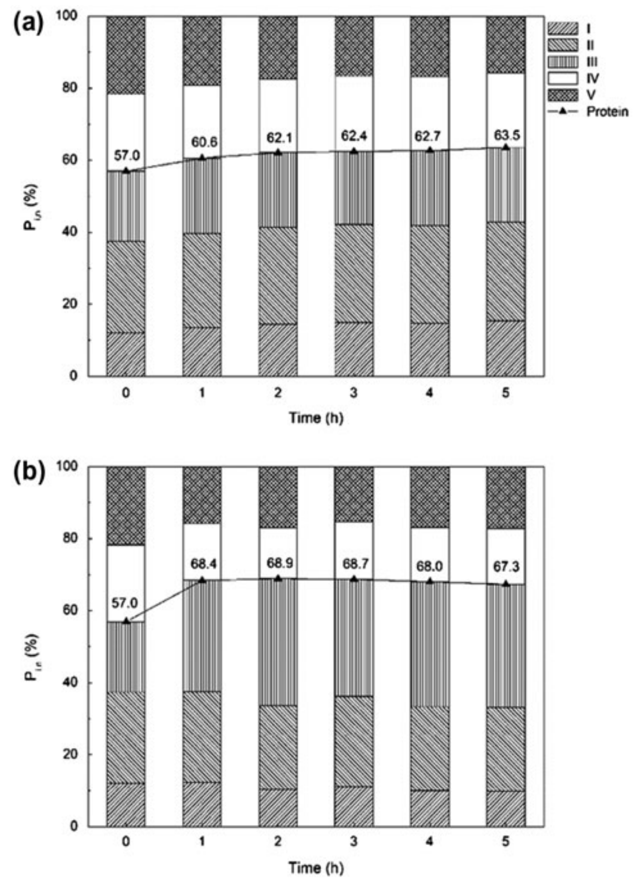

Fig. 4. Distribution of FRI in FDOM from samples of the blank test (a) and CER pretreatment (b).

percent with the result of chemical analysis were 0.85 ($p < 0.05$) for the blank test and 0.96 ($p < 0.01$) for the CER pretreatment, demonstrating the validness of FRI analysis. Furthermore, according to the chemical and FRI analysis, protein was revealed to be the largest fraction of extracted EPS in this work, which was in agreement with previous studies [8,13,14].

The evolution of MW distribution of DOM could be observed in Fig. 5. Low MW molecules, especially less than 10,000 Da, existed in the sludge pretreated after 5 h, while they are absent in the raw sludge, and a peak ranging from 100 to 1,000 Da exited in the distribution curve of the pretreated sludge. It indicated that DOMs with low MW were generated after pretreating for 5 h. Number-average molecular weight (Mn), weight-average MW, and the coefficient of MW distribution (MW/Mn) were employed to evaluate and understand the MW distribution of the dissolved organics better. A high MW/Mn ratio implied a broad distribution of MW. The MW, Mn, and MW/Mn ratio of the raw sludge were 159.7 kDa, 76.2 kDa, and 2.1,

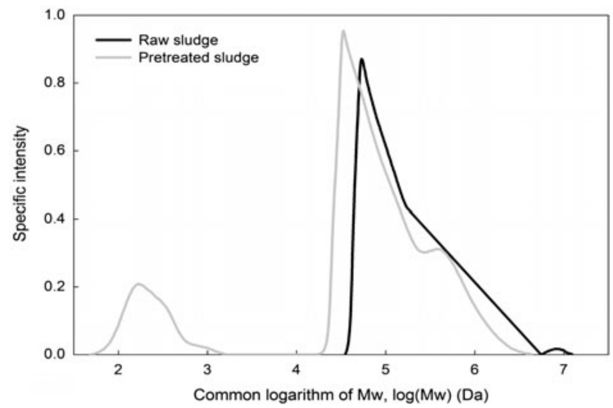

Fig. 5. GFC chromatograms of DOM in raw sludge and sludge pretreated after 5 h.

respectively, while the MW, Mn, and MW/Mn ratio of the pretreated sludge were 189.7 kDa, 1.3 kDa, and 140.9, respectively. It was suggested that sludge pretreated after 5 h had much wider MW distribution than that of raw sludge.

3.2. Effect of CER pretreating method on the sludge disintegration

The changes of particle-size distribution of sludge flocs were illustrated in Fig. 6 based on cumulative volume distribution. It was shown that the size range was 0.30–184.72 μm. Furthermore, it was noticeable that the floc size distribution curves were shifted to the smaller particles after CER pretreating, whereas the distribution curves of the blank tended toward the larger size classes due to the flocculation by stirring. It could be

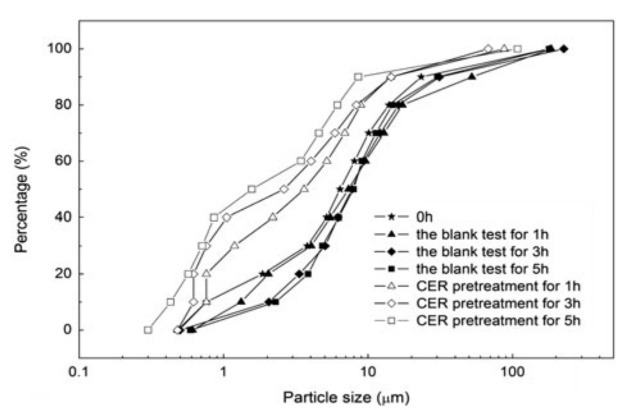

Fig. 6. Variations of particle-size distribution of sludge flocs.

concluded that CER pretreatment may induce the breakage of flocs, thus reduce the particle size.

The SEM images of the granular sludge were also shown (Fig. 7). It was clearly seen that the structure of raw sludge sample was loose, wrinkled, and coarse with few cavities, while the surface of the sludge pretreated for 5 h was more compact and less porous. It indicated that the sludge could be disintegrated through CER pretreating, and then organic matters could be released to the aqueous phase.

3.3. *Effect of CER pretreating method on the sludge dewaterability and settleability*

As shown in Fig. 8, the variation of the normalized CST values of the blank was negligible (1.39–3.60 s L/g), meaning that the dewaterability of unpretreated sludge was relatively stable. While CER pretreatment could deteriorate sludge dewaterability dramatically, for the corrected CST values it increased from 2.19 s L/g at the beginning to 11.95 s L/g after 5 h pretreating. It was also revealed that the SVI values of pretreated sludge were 3.2-, 4.7-, 2.5-, 2.8-, and 2.7-fold than that of the blank at first to fifth hour, respectively, indicating that CER pretreatment would result in the deterioration of sludge settleability. In contrast to the blank test, whose SVI values were steady between 54 and 130, the SVI values of pretreated sludge increased from 108 at the beginning to 347 after 5 h pretreating.

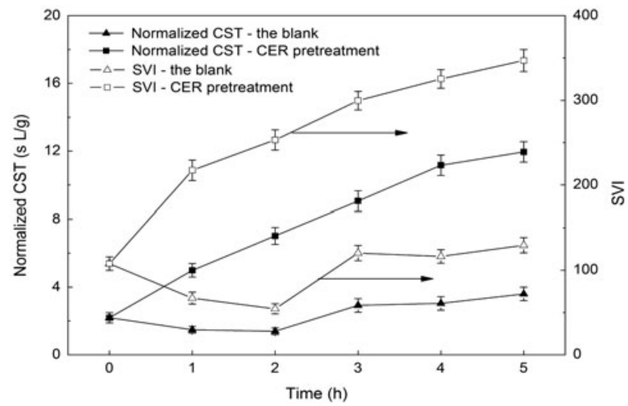

Fig. 8. Effect of CER pretreatment on dewaterability and settleability of sludge.

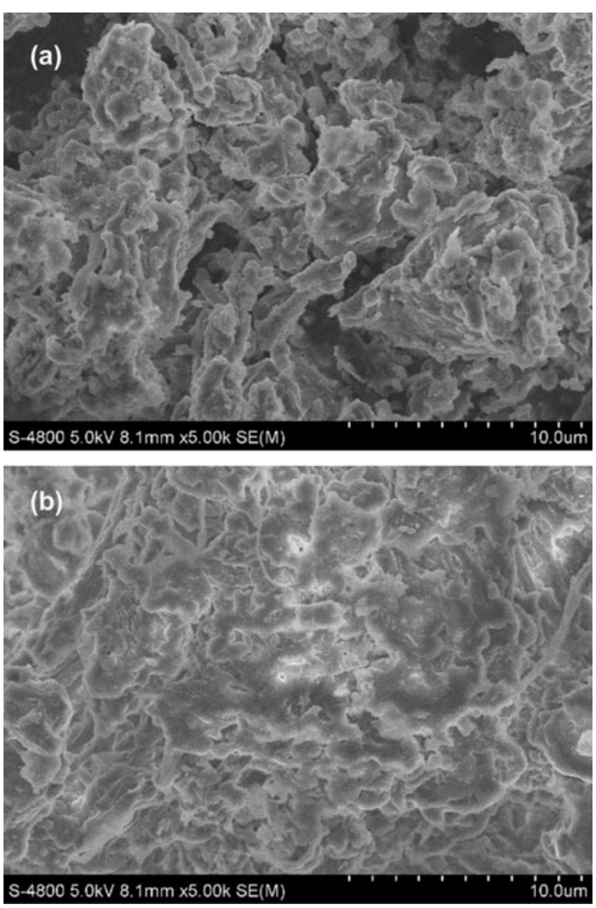

Fig. 7. SEM of raw sludge (a) and sludge pretreated for 5 h (b).

Above all, CER pretreatment to sludge would cause poor dewatering and settling ability. The reason for these results might be that CER could not only remove the divalent cations, thus causing the EPS to fall apart, but also increase the concentration of DOM (mainly protein and polysaccharide) and reduce floc size.

Thus, the correlations between protein, polysaccharide, and normalized CST, as well as SVI values were estimated to better understand the effects of these compounds on sludge dewaterability and settleability. The Pearson's coefficients were shown in Table 2. Poor correlation was found between protein, polysaccharide, and normalized CST, as well as SVI values in the blank, while the protein and polysaccharide had significant positive correlations with the sludge normalized CST and SVI values in the CER pretreatment. These statistical results suggested that the increase of DOM could contribute to the deterioration of sludge dewaterability and settleability. It is probably because proteins and polysaccharides contributed significantly to the water binding capacity of sludge floc matrix [26,27]. Furthermore, it could also be deduced that polysaccharide had more positive correlation with the normalized CST and SVI values than protein.

Table 2
Coefficients of Pearson correlation between protein, polysaccharide, normalized CST and SVI

	Blank test		CER pretreatment	
	Protein (g COD/L)	Polysaccharide (g COD/L)	Protein (g COD/L)	Polysaccharide (g COD/L)
Normalized CST (s L/g)	0.03	0.38	0.82[a]	0.97[b]
SVI	0.27	0.22	0.91[a]	0.98[b]

[a]$p < 0.05$.
[b]$p < 0.01$.

In addition, normalized CST and SVI values increased with the decrease of particle size during CER pretreatment, suggesting that the reduction of floc size had adverse influence on sludge dewaterability and settleability, which was consistent with previous publications [28,29]. The decrease of floc size could lead to an exposure of more floc surfaces, strengthen the hydrophobicity of sludge flocs, and accordingly contribute to the apparent deterioration in the sludge dewaterability and settleability.

Besides, the bivalent and trivalent cations were removed by CER and replaced by sodium, resulting in the increase of monovalent to divalent cation ratio, which was reported to show negative correlation with sludge dewatering and settling ability [13,28].

4. Conclusions

The CER pretreating method could enhance sludge solubilization and disintegration dramatically. Organic matters (especially protein and polysaccharide) could be released to the bulk and become soluble, and protein was the major constituent of DOM. DOMs with low MW were generated and MW distribution became much broader. SEM photographs suggested that sludge was broken up. Furthermore, the increasing proteins, polysaccharides, and the monovalent to divalent cation ratio, as well as the decrease of particle size showed significant correlations with the deteriorating dewaterability and settleability. The above studies better demonstrated the feasibility to use CER as a pretreating strategy before AD.

Acknowledgments

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