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Improved sludge dewaterability by combining SDS pre-treatment conditioning with PDMDAAC flocculation

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ABSTRACT

Wastewater treatment generates considerable amounts of sludge. Reducing sludge volume by dewatering is an important part of many wastewater treatment processes. We investigated the effects of a combination of sodium dodecyl sulfate (SDS) and polydimethyldiallylammonium chloride (PDMDAAC) on anaerobically digested sludge dewaterability. Capillary suction time (CST) and specific resistance to filtration (SRF) were measured as indicators of sludge dewaterability. Extracellular polymeric substances (EPS) content, particle size, zeta potential, and bound water in sludge floc were also measured. We found that CST increased from 348.5 to 530.5 s as the SDS dosage increased. However, with a dose 0.03 g g^{-1} DS PDMDAAC only, SRF and CST were 3.78×10^{12} m kg^{-1} and 79.2 s; with a combined dose of 0.025 g g^{-1} DS SDS and 0.03 g g^{-1} DS PDMDAAC, SRF and CST decreased to 3.21×10^{12} m kg^{-1} and 68.7 s. These results indicate that above a certain dose, SDS reduces sludge dewaterability and that an optimum dose of SDS in sludge pre-treatment would improve subsequent PDMDAAC conditioning. Sludge conditioned with a combination of SDS and PDMDAAC increases dewaterability more than conditioned with SDS only or PDMDAAC only. Freeing EPS-bound water with SDS followed by re-flocculation using PDMDAAC gave improvement in sludge dewaterability.

Keywords: Sludge dewatering; Sodium dodecyl sulfate; Polydimethyldiallylammonium chloride; Extracellular polymeric substances; Bound water

1. Introduction

Large quantities of sludge are generated by wastewater treatment processes, and over 90% of the sludge is water [1]. Dewatering is a vital part of the sludge treatment process as it reduces sludge volume and, consequently, the transportation and disposal costs of the sludge. The mechanical press, which consumes low energy, is widely used to reduce sludge volume [2]. However, extracellular polymeric substances (EPS) are highly hydrated and able to bind a large volume of water in sludge. This significantly limits sludge dewatering [3]. The choices of conditioning chemicals are important for maximum sludge dewatering. Many studies have been conducted to increase sludge dewaterability by using conditioning chemicals [4–6]. Typical chemical additives include aluminum sulfate, ferric chloride, polyelectrolytes, and surfactants [7]. Surfactants decrease the surface tension between aqueous and non-aqueous liquids. By releasing EPS from sludge, surfactants could also alter cell structures and thus affect the properties of the sludge [8]. Flocculants are widely used to improve sludge dewatering through physical and chemical conditioning processes. Flocculants overcome the repulsion between charged particles and increase the particle size of

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sludge, leading to improved stability and dewaterability of the sludge [9]. The cationic surfactant (cationic cetyltrimethylammonium bromide) combined with the cationic flocculant (cationic polyacrylamide, CPAM) increases sludge dewaterability [10].

Sodium dodecyl sulfate (SDS) is an anionic surfactant. The amphiphilic molecules have two parts, one of which, the hydrophilic head is hydrophilic, and the other, the hydrophobic tail chain is hydrophobic. SDS causes the sludge matrix to break up and increases the solubilization of EPS in sludge. It also increases soluble protein and carbohydrate concentrations [11]. It has been observed that the addition of SDS on its own leads to a decrease in sludge dewaterability [12]. However, Besra et al. [13] studied the dewatering of kaolin suspensions by CPAM flocculants in the presence of SDS and found that the addition of CPAM and SDS substantially reduced cake moisture. Polydimethyldiallylammonium chloride (PDMDAAC) is a green cationic organic polymer with a high positive charge density that has been widely used in sludge dewatering because of its adsorption and binding capabilities [14].

There are few studies of the effects of a combination of SDS and cationic flocculants on the physicochemical properties of sludge. We examined changes in dewaterability and floc properties of sludge conditioned by SDS and SDS combined with PDMDAAC (SDS–PDMDAAC). We observed the characteristics of EPS and the surface characteristics and microstructure of sludge treated with both conditioners and compared them.

2. Materials and methods

2.1. Materials

The sludge was obtained from the Gaobeidian Wastewater Treatment Plant, Beijing, China. The sludge was treated by thermal hydrolysis at 160°C for 30 min followed by anaerobic digestion at 40°C for 20 d. The characteristics of the sludge are given in Table 1. The SDS was supplied by Tianjin Guangfu Fine Chemical Research Institute, China, and the 40% w/w aqueous solution of PDMDAAC, which had 100% charge density, was supplied by SNF Flocculant Co., Ltd., China.

2.2. Batch experiments

Sludge samples were prepared at room temperature of 24°C ± 1°C. In each case, a 800 mL of sludge sample was added in a 1,000 mL beaker. Then, sludge sample was homogenized by stirring at 300 rpm for 1 min using a magnetic stirrer. The conditioner was added (0.025–0.2 g g^{-1} DS SDS) and the tank was stirred for 5 min. After this time, 400 mL of the sample was poured out to measure the sludge properties. Then 0.03 g g^{-1} DS PDMDAAC was added to the remaining 400 mL of conditioned sludge, and the mixture was rapidly stirred at 300 rpm for 1 min and then stirred at 100 rpm for 5 min.

Table 1
Sludge characteristics

Indicator	Sludge
Moisture content (%)	95.8
VS/TS (%)	55.3
CST (s)	348.5
SRF (m kg^{-1})	5.82E + 12
$d_{0.5}$ (μm)	19.3
Zeta potential (mV)	−13.2

2.3. Analytical methods

2.3.1. Sludge dewaterability assessment

Capillary suction time (CST) and specific resistance to filtration (SRF, m kg^{-1}) were measured to determine sludge dewaterability. CST was measured with CST apparatus (Model 319, Triton, UK) using an 18 mm diameter funnel and Whatman No. 17 chromatography-grade filtration paper. The SRF measurement was made in a Büchner funnel using a quantitative filter paper. The Büchner funnel was filled with 100 mL sludge, and a constant pressure of 0.1 MPa was maintained by a vacuum pump. The SRF of the sludge was calculated by:

$$\mathrm{SRF} = \frac{2PA^2b}{\mu\omega} \tag{1}$$

where P (kg m^{-2}) represents the pressure applied, A (m^2) is the filter area, b is the slope of the curve plotting the ratio of the time of filtration to the volume of filtrate (t/V) against the filtrate volume (V), μ (kg s m^{-2}) is the kinetic viscosity, and ω (kg m^{-3}) is the dry solid weight per unit volume of sludge on the filtrate media.

2.3.2. EPS characterization

2.3.2.1. EPS extraction

EPS were classified as soluble, loosely bound, and tightly bound (S-EPS, LB-EPS, and TB-EPS) according to the strength of the bond between EPS and sludge micelles. A sludge sample was centrifuged in a 50 mL tube at 3,000 g for 10 min. The supernatant was collected as S-EPS. The residual sediment in the tube was re-suspended to 50 mL with a 0.05% (w/w) sodium chloride solution, sonicated at 20 kHz for 2 min, shaken horizontally at 150 rpm for 10 min, sonicated again for an additional 2 min, and centrifuged at 8,000 g for 10 min. The supernatant was collected as LB-EPS. The sludge pellet in the tube was re-suspended as before, sonicated for 3 min, heated at 60°C for 30 min, and centrifuged at 10,000 g for 20 min, the supernatant was collected as TB-EPS [15].

2.3.2.2. EPS composition

The supernatant collected from the liquid fraction of the EPS was filtered through a 0.45 μm syringe–driven filter (Millipore Co., USA) before analysis. The protein (PN) content of the supernatant was determined by the BCA method using a BCA protein assay kit, and polysaccharide content was determined using the anthrone method with standard glucose [16].

Three-dimensional excitation–emission matrix (3D-EEM) fluorescence spectra was obtained using a fluorescence spectrophotometer (F-4500, Hitachi, Japan) with an excitation range 200–400 nm and an emission range 220–550 nm. The spectra were recorded at a scan rate of 12,000 nm min^{-1}, using excitation and emission slit bandwidths of 5 nm.

2.3.3. Other indicators

Bound water content was measured by thermogravimetric analysis (TGA). The sludge was first frozen at −20°C, and free water was totally removed by a freeze dryer (FD-100). The analysis was performed with TA Instruments (SDT-Q600, America) apparatus that simultaneously performs TGA and differential scanning calorimetry. The mass of the samples ranged from 5 to 10 mg. The heating rate applied to the samples was 10°C min^{-1}, with a maximum temperature of 600°C, under N_2 flowing at 100 mL min^{-1}. When the temperature approached 105°C, the water in the sludge had been thoroughly eliminated [17].

Sludge floc size was determined by a Mastersizer 2000 particle size analyzer (Malvern Instruments Ltd., UK). The $d_{0.5}$ value given is the median value.

Zeta potential, an indicator of the electrostatic potential at the surface of the flocs, was measured by a Malvern Zetasizer 3000 (Malvern Instruments Ltd., Malvern, UK).

All samples were measured in duplicate.

3. Results and discussion

3.1. Effect of SDS and SDS–PDMDAAC on sludge dewaterability

Foam might be generated by surfactants in the presence of SDS due to a decrease in the surface tension of the sludge. However, there was no obvious adverse effect from foam when the SDS dosage was <0.2 g g^{-1} DS [18]. We set an upper limit of 0.2 g g^{-1} for SDS dosage. The effects of SDS only and PDMDAAC dosage only on sludge dewaterability are shown in Fig. 1a. The CST values of sludge increased as the SDS increased; the CST value of the raw sludge increased from 348.5 to 530.5 s when the dosage of SDS was 0.2 g g^{-1} DS, indicating a decrease in dewaterability. At the beginning of filtration, the small sludge particles clogged the filter paper, making extraction difficult, and we did not obtain values for SRF. This is consistent with other reports that SDS conditioning reduces sludge dewaterability [5,12]. However, when we used PDMDAAC alone for sludge conditioning, there was a sharp decrease in CST values as PDMDAAC dosage increased, and the minimum of 79.2 s was reached when the dosage of PDMDAAC was 0.03 g g^{-1} DS.

When the dosage of SDS was in the range 0.025–0.1 g g^{-1} DS, the SRF and CST values for the sludge were lower than for sludge conditioned with only 0.03 g g^{-1} DS PDMDAAC (Fig. 1b). SRF and CST decreased from 3.78 × 10^{12} m kg^{-1} and 79.2 s to the minimum values of 3.21 × 10^{12} m kg^{-1} and 63.5 s. Both SRF and CST increased after the minimum was reached as the SDS dosage increased. These results show that sludge dewaterability was determined by the doses of SDS and PDMDAAC, and these two chemicals had a synergistic effect on sludge dewaterability. Purcell et al. [19] found that SDS and polymer poly(vinylpyrrolidone) (PVP) interact

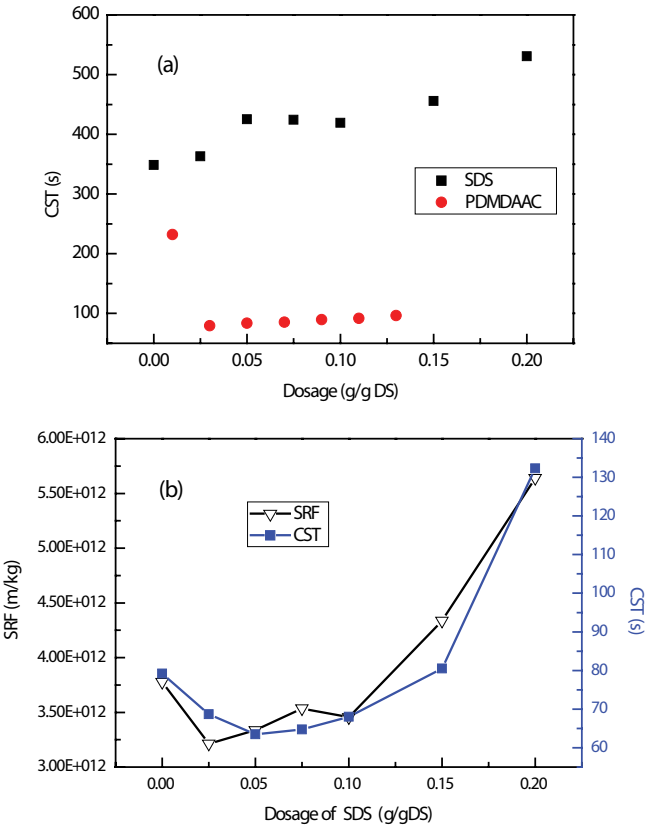

Fig. 1. Effect of SDS and PDMDAAC on sludge dewaterability: (a) SDS only and PDMDAAC only and (b) combined SDS with 0.03 g g^{-1} DS PDMDAAC.

cooperatively at the surface. We also characterized EPS and other sludge properties to further understand the mechanism of sludge dewatering.

3.2. Effect of SDS, combined SDS with PDMDAAC on EPS

3.2.1. EPS distribution and composition

EPS are high-molecular-weight biopolymers originating as bacterial secretions, cell lysis and hydrolysis products, leakage of exocellular constituents, and adsorbed organic matter from the surrounding wastewater. The distribution and composition of EPS affect sludge dewaterability [20]. Fig. 2a shows that EPS was solubilized and released into the liquid phase as the SDS concentration increased. When the SDS dosage was 0.075 g g^{-1} DS, extractable EPS and protein content (PN) of EPS increased to maximum values of 2,242.62 and 1,696.71 mg L^{-1}, and polysaccharide content increased from 377.87 mg L^{-1} to a maximum of 599.53 mg L^{-1}. EPS are active in the formation of sludge particles [21], which aggregate to form larger flocs, thereby increasing sludge dewaterability [22]. The release of EPS in the sludge affects flocculation and results in CST increasing and decreased dewaterability.

Proteins and polysaccharides are major constituents of EPS [23,24]. The proteins and polysaccharides contain polar groups, so EPS easily interacts with surfactants, leading to the release of EPS [25]. The proteins and polysaccharides

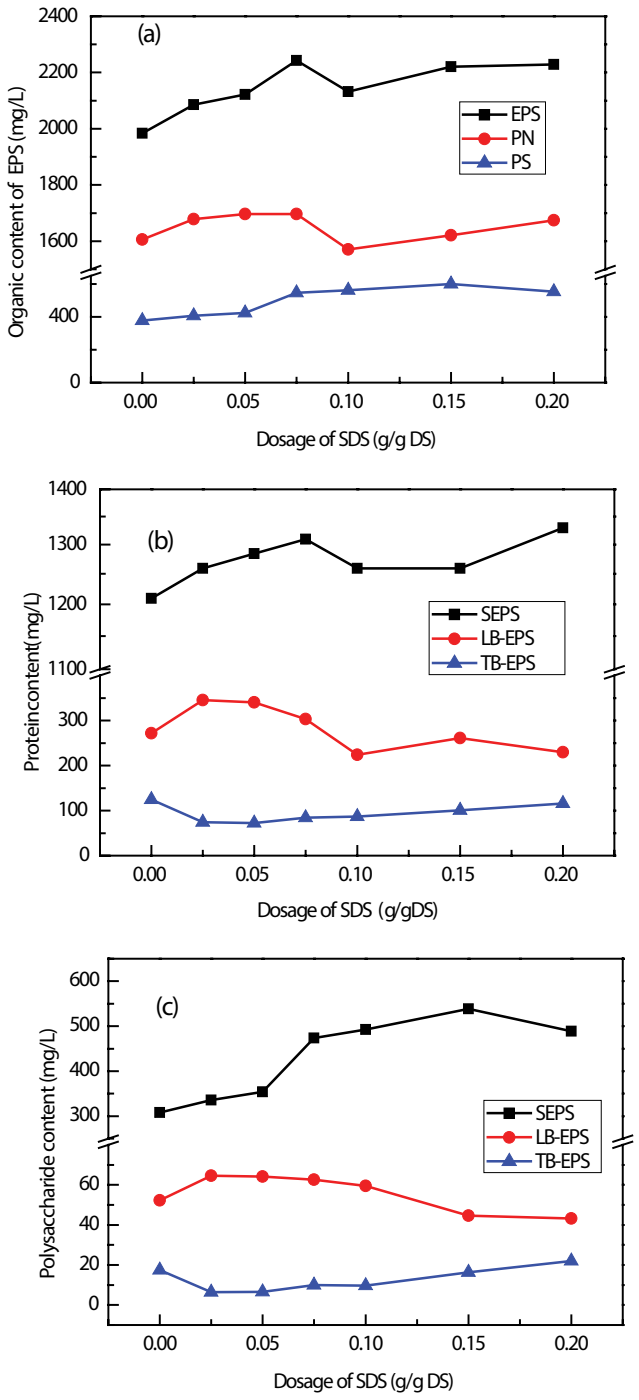

Fig. 2. Effect of SDS dosage on content of (a) different EPS fractions, (b) protein, and (c) polysaccharide.

freed from S-EPS increase with the addition of SDS, especially at higher dosages (Figs. 2b and c). As SDS increased, the protein and polysaccharide content of S-EPS increased from 1,209.5 and 308.06 mg L^{-1} to maximum values of 1,329.25 and 538.61 mg L^{-1}. The protein and polysaccharide content of LB-EPS both initially increased but then decreased and became steady. Protein and polysaccharide content in LB-EPS were higher than that in LB-EPS of the raw sludge when the SDS dosage was in the range 0.025–0.075 g g^{-1} DS. This is because SDS deflocculated the sludge, which resulted in more proteins and polysaccharides being freed from LB-EPS. Protein and polysaccharide content in LB-EPS both decreased while S-EPS in the sludge increased significantly when the dosage of SDS was in the range 0.1–0.2 g g^{-1} DS, indicating that SDS likely transformed some of the LB-EPS fraction into S-EPS. Protein and polysaccharide content in TB-EPS first decreased and then increased. LB-EPS and TB-EPS are highly hydrated, but the addition of SDS caused them to solubilize into an aqueous phase, which led to the release of bound water from the sludge [26].

EPS content of the sludge decreased significantly with a dosage of SDS and 0.03 g g^{-1} DS PDMDAAC compared with SDS conditioning only (Figs. 2a and 3a). The protein content of S-EPS was almost unchanged when the SDS dosage increased from 0 to 0.1 g g^{-1} DS (Fig. 3b). This was likely due to strong cationic groups and active adsorption groups in the PDMDAAC molecule, which can adsorb colloids [27]. However, as the SDS dosage continued to increase, the PDMDAAC dosage had no obvious effect on S-EPS. Thus the increase in S-EPS caused a significant increase in SRF and CST, and sludge dewaterability decreased. A comparison of Figs. 2b and c with Figs. 3b and c shows that the protein and polysaccharide contents of LB-EPS which was treated with SDS-PDMDAAC were significantly lower than that treated with a low dose of SDS. There was little difference in TB-EPS protein and polysaccharide content, which suggests that SDS-PDMDAAC fractionated EPS only on the sludge surface.

3.2.2. 3D-EEM analysis

3D-EEM shows the composition of organic matter in water. The effect of SDS on EPS as shown by the 3D-EEM spectra and released EPS fluorescence are shown in Fig. 4. Three fluorescence peaks were detected: Peak A ($\lambda_{ex}/_{em}$ = 225/340), aromatic proteins; Peak B ($\lambda_{ex}/_{em}$ = 280/335), tryptophan-like proteins; and Peak C ($\lambda_{ex}/_{em}$ = 330/410), humic substances [28]. The results given by 3D-EEM are consistent with the conclusion derived from Fig. 2. The intensity of released EPS fluorescence gradually increased as the dosage of SDS increased (Figs. 2a–g). The intensity of peaks A, B, and C increased from 1,149, 803.4, and 446.9 to 1,619, 1,600, and 1,194 when the SDS dosage increased from 0 to 0.2 g g^{-1} DS. Humic substances contained more hydrophobic fractions, indicating that protein, protein-like substances, and humic substances were more easily solubilized and released from the sludge by SDS and that bound water was freed.

3.3. Effect of SDS, combined SDS with PDMDAAC on zeta potential and particle size

Zeta potential is a measurement of the charge at the sludge surface. The charge difference between the sludge surface and the surrounding liquid affects sludge dewaterability [29]. Fig. 5a shows the effect of SDS alone and a combination of SDS with 0.03 g g^{-1} DS PDMDAAC on zeta

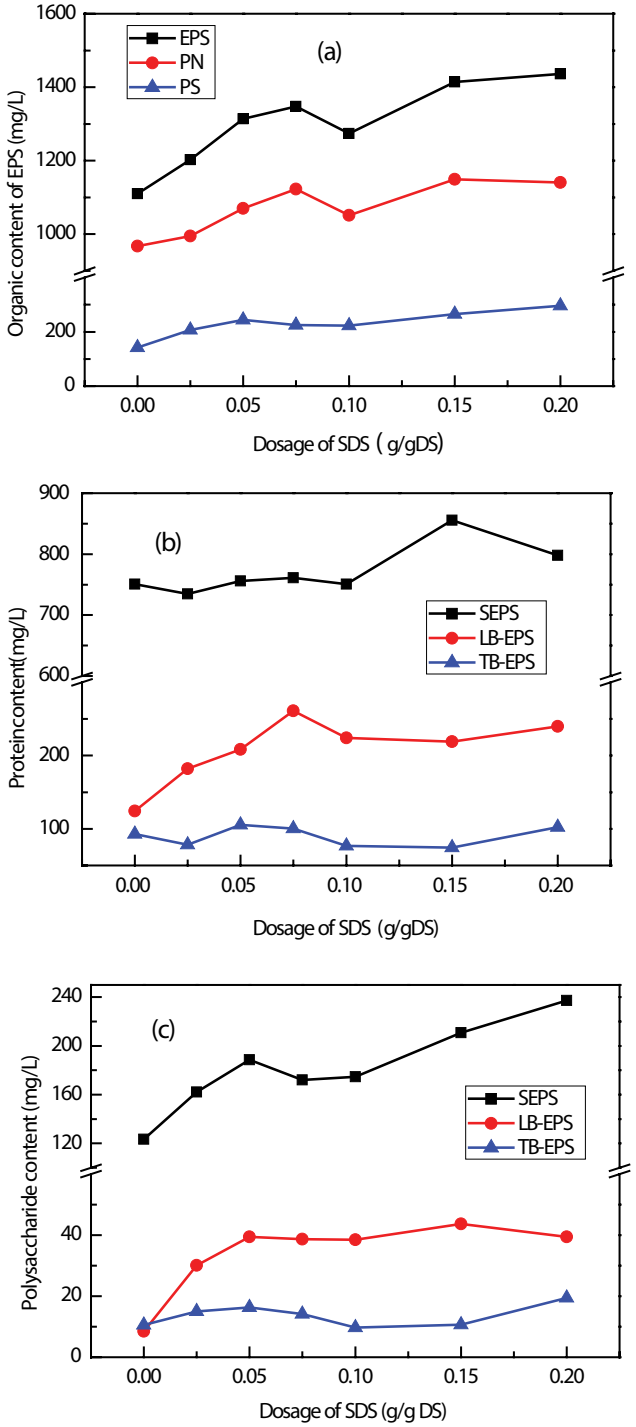

Fig. 3. Effect of combined SDS with PDMDAAC (0.03 g g^{-1} DS) on content of (a) different EPS fractions, (b) proteins, and (c) polysaccharides.

potential. The zeta potential of the sludge was initially measured as –13.2 mV. It decreased significantly with the addition of SDS, reaching a minimum of –15.72 mV at higher SDS concentrations. The surfaces of both sludge flocs and EPS are normally negatively charged. The anionic surfactant SDS increased the negative charge, leading to a disaggregation of EPS and sludge flocs. The addition of cationic flocculants neutralizes the negative surface charge of sludge particles, which results in the particles becoming destabilized and aggregated. PDMDAAC is a highly-charged cationic polymer flocculant which is effective through adsorptive neutralization and polymer bridging [30]. Zeta potential reached a maximum of –7.37 mV when the dosage of SDS was 0.075 g g^{-1} and a dose of 0.03 g g^{-1} PDMDAAC-DS was added, and it decreased with increased SDS dosage (Fig. 5a). This indicates that excess SDS attenuated the cationic neutralization by PDMDAAC.

Average floc size ($d_{0.5}$, μm) of the sludge is 21.27 μm (Fig. 5b). Average floc size decreased slightly as SDS increased, reaching a minimum of 19.03 μm when the SDS dosage was 0.15 g g^{-1} DS. Wilen et al. [31] found that EPS greatly affects sludge flocculation. When SDS is added, EPS on the sludge surface gradually solubilizes, affecting flocculation. However, there is no obvious change in floc size, which indicates a stable floc structure during SDS treatment, consistent with the results of the 3D-EEM analysis. However, when the PDMDAAC dosage was 0.03 g g^{-1} DS, average floc size increased significantly (Fig. 5b) and reached a maximum $d_{0.5}$ of 68.7 μm when the SDS dosage was 0.05 g g^{-1} DS. As the SDS dosage further increased, $d_{0.5}$ gradually decreased. Previous studies have shown that sludge particle size has a significant effect on sludge properties; smaller flocs can easily clog the cake during filtration, thereby reducing sludge dewaterability [32]. Higher levels of SDS increase the quantity of fine particles in the sludge and decrease dewaterability.

3.4. Effect of SDS, combined SDS with PDMDAAC on bound water

Bound water influences sludge dewaterability, and decreasing the bound water content of sludge increases dewaterability [33]. Fig. 6 shows that bound water content decreased from 11.36% to a minimum of 4.16% as SDS increased. The explanation is as follows. The surface of the protein molecule is highly hydrophilic with $–NH_4^+$, –COO–, and –OH, and the polar group forms a hydrated layer on the sludge particle surfaces. Protein is freed from the surface of the sludge when the SDS is added, reducing the quantity of hydrophilic groups, reducing the adsorbed water content, and making the sludge surfaces less hydrophilic. Polysaccharides form strong water-floc bonds. As the polysaccharides move from the flocs into the liquid phase, the flocs become less capable of forming water bonds, and bound water in EPS is released and converted into free water [34].

Bound water in the sludge decreased to 4.03% with 0.05 g g^{-1} DS SDS pre-treatment and 0.03 g g^{-1} DS PDMDAAC re-flocculation. EPS were freed after the SDS pre-treatment, which facilitated the conversion of bound water to free water. However, the quantity of bound water increased when the dosage of SDS was 0.15 and 0.2 g g^{-1} DS. The quantity of bound water reached 12.72% when the dosage of SDS was 0.2 g g^{-1} DS. Increased SDS affects the charge neutralization capability of PDMDAAC, leaving more water bound to sludge flocs.

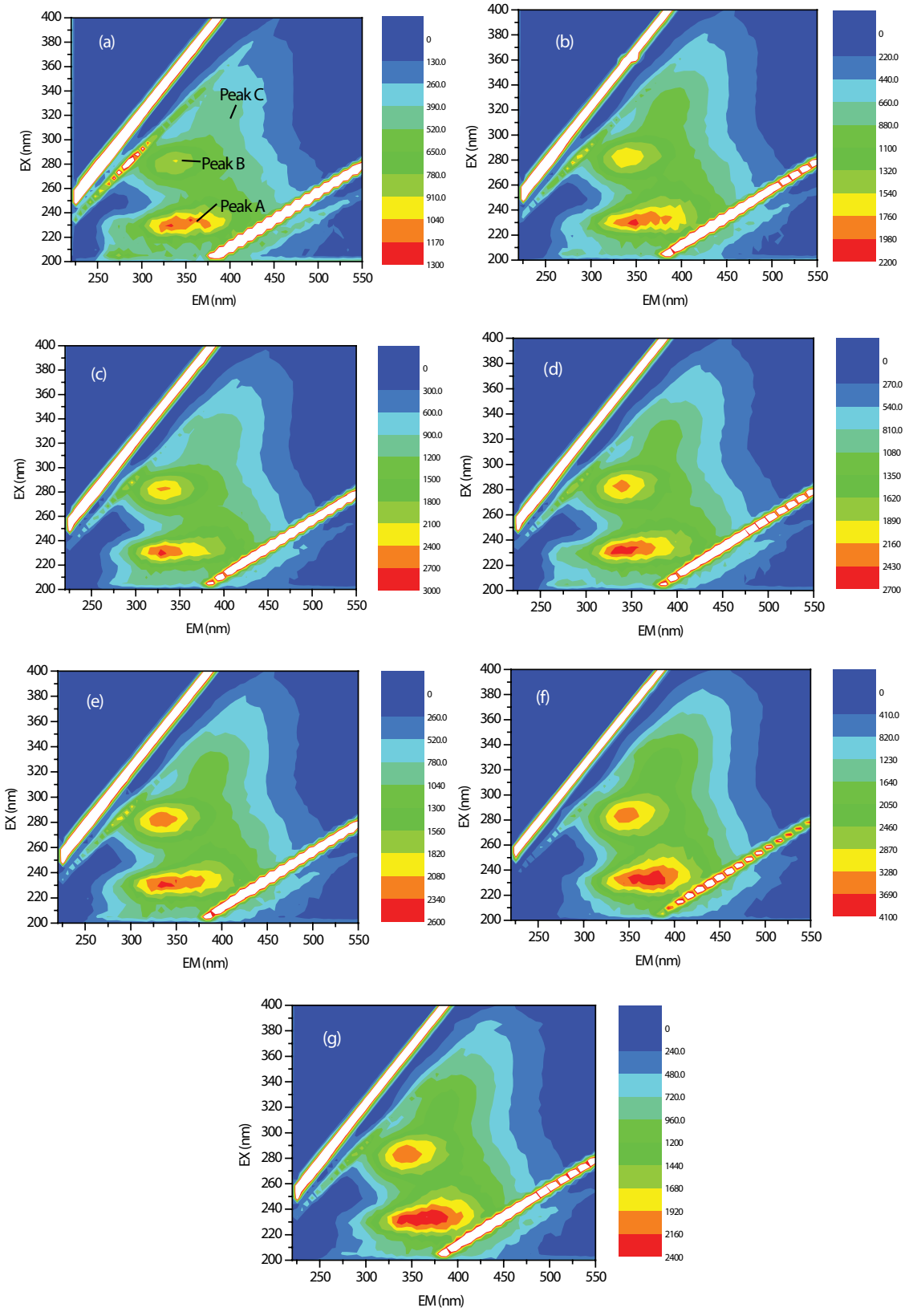

Fig. 4. Effect of SDS dosage on EEM profile of the released fluorophores: (a) raw sludge, (b) 0.025 g g^{-1} DS, (c) 0.05 g g^{-1} DS, (d) 0.075 g g^{-1} DS, (e) 0.1 g g^{-1} DS, (f) 0.15 g g^{-1} DS, and (g) 0.2 g g^{-1} DS.

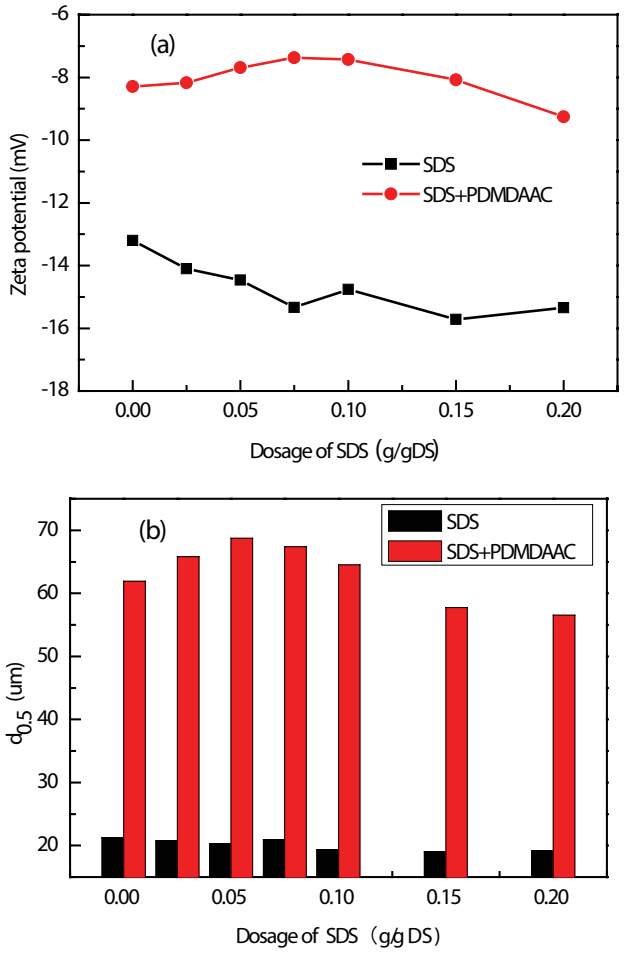

Fig. 5. Effect of SDS, combined SDS with PDMDAAC (0.03 g g^{-1} DS) on (a) zeta potentials and (b) particle size of sludge.

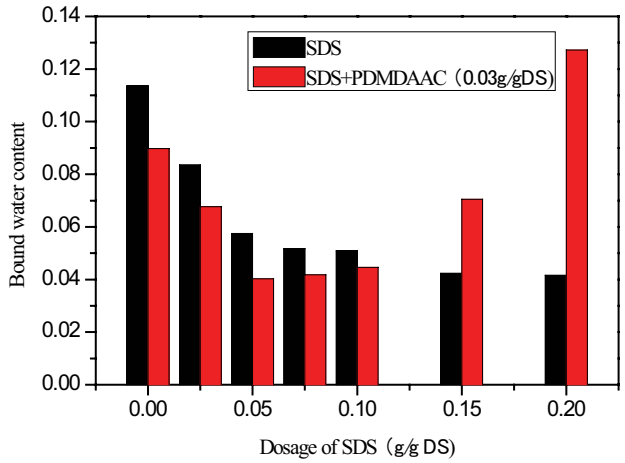

Fig. 6. Effect of SDS, combined SDS with PDMDAAC (0.03 g g^{-1} DS) on bound water content of sludge.

4. Conclusion

We investigated the effects of the anionic surfactant SDS and the highly-charged cationic polymer flocculent PDMDAAC on the dewaterability of anaerobically digested sludge. The results show that the SDS increases the release of EPS from sludge floc surfaces, which disaggregates sludge floc and reduces sludge dewaterability. CST increased from 348.5 s to a maximum of 530.5 s when the SDS dosage increased from 0 to 0.2 g g^{-1} DS. However, a fraction of bound water was freed from the floc. Sludge dewaterability was increased by a combination of SDS pre-treatment combined with PDMDAAC for re-flocculation. Compared with a dose of 0.03 g g^{-1} DS PDMDAAC only, with the addition of combined 0.025 g g^{-1} DS SDS with 0.03 g g^{-1} DS PDMDAAC, SRF and CST decreased from 3.78 × 10^{12} m kg^{-1} and 79.2 s to 3.21 × 10^{12} m kg^{-1} and 68.7 s. The results indicate that an appropriate amount of SDS for sludge pre-treatment increases subsequent PDMDAAC dewatering and that the combination of SDS with PDMDAAC treatment was much more effective than conditioning with either SDS only or PDMDAAC only in increasing sludge dewaterability.

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