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Application of Enteromorpha as a new kind of coagulant aid in municipal sludge treatment

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ABSTRACT

The large-scale blooms of Enteromorpha disrupts normal ecosystem functioning and brought serious environmental problems in past years. This study focused on the recycling of it with the aim of "treating waste by waste." In this paper, Enteromorpha polysaccharide (Ep) was applied as a new coagulant aid in sludge treatment process. Initially, orthogonal test was conducted and then the effect of Ep on coagulation behavior was studied and coagulation performance was evaluated in terms of solid content, turbidity, filtrate rate, capillary suction time, and resistance to filtration. Zeta potential and scanning electron microscopic (SEM) were also measured to investigate coagulation mechanism. In addition, cost analysis of sludge treatment was also discussed in this paper. Results of orthogonal test showed that coagulation efficiency was significantly affected by hydraulic conditions. Sludge treatment efficiency could be apparently improved by Ep addition and the optimum polyacrylamide (PAM) and Ep dosages were 15 and 3 mg/L, respectively. When Ep was used in combination with PAM, the average particle size of sludge was increased, and the treated sludge possessed better dehydration ability. In addition, results of cost analysis showed that Ep addition could reduce the cost of sludge treatment by reducing PAM dosage.

Keywords: Enteromorpha polysaccharide; Sludge dewatering; Coagulant aid; PAM; Cost analysis

1. Introduction

Wastewater treatment processes produce large amounts of sludge, which commonly contains over 90% water [1], and has been addressed as an environmental issue for the past decades [2,3]. Sludge handling and disposal treatments account for up to 50% of total wastewater treatment costs for large plants [4]. Except for economic aspect, the treatment of sludge is also faced with the pressure of sustainable disposal according to current laws and regulations [5]. Therefore, the reduction of sludge volume by water separation is the most important part of sludge treatment. However, colloidal particles in nature normally carry charges on their surface and provide certain potential

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energy barrier, which lead to the stabilization of suspension in water [6]. The free water could be readily removed by gravity separation, but bound water, which involves the interaction between water and solid surface, requires more energy to be released from the sludge [3]. To solve this problem, pretreatment technologies such as ultrasonication, coagulation, electrolysis, microwave irradiation, and alkaline pretreatment have been employed [7–10]. Among them, chemical coagulation is considered as an efficient way since coagulation process could compress the extracellular polymeric substance structure for sludge conditioning. Addition of adequate coagulants would be helpful for sludge particles to agglomerate into larger particles [11]. The destabilization of colloids is usually induced by charge neutralization, agglomeration, and colloid collision, while large particles are formed by fixing the destabilized particles to the long polymeric chains. Therefore, coagulant with a high molecular weight is critical to the performance of sludge dewatering. Recently a series of polyacrylamide (PAM), including anionic, cationic, and nonionic PAM, are the most commonly used conditioner in coagulation process prior to dewatering [12]. But dehydration effect of PAM is easily affected by water quality. More importantly, PAM may partially hydrolyze into toxic monomers, such as acrylic acid and organic amine, which may result in secondary pollution in sludge treatment process [13]. Therefore, it is necessary to reduce the risk caused by PAM in sludge treatment by decreasing its dosage. To achieve this purpose, many products, which are denominated as coagulant aids, are used together with traditional coagulant in sludge treatment [14]. In recent years, natural polymers have drawn great attention with potential for applications in water treatment due to their advantages such as high biodegradability, non-toxic to human beings, and minimal second contamination [15,16].

Enteromorpha is one of the dominant seaweeds in littoral zone, which distributes worldwide from the intertidal to the upper subtidal zones [17]. As a kind of common fouling algae, Enteromorpha often contributes to the formation of “green tide.” Large-scale blooms of Enteromorpha disrupts normal ecosystem functioning as the floating Enteromorpha block sunlight from photosynthetic marine plants under the water surface. In addition, the dead algae may consume all the oxygen in water, which results in the death of fish [18]. For tourist cities, a great quantity of floating Enteromorpha gathered to the shore and posed a serious threat to tourism. Hence, the government had to spend lots of labor and financial resources on Enteromorpha disposal every year [19,20]. Currently, a common way for disposal of Enteromorpha is open burning, which causes serious secondary pollution and resource wastes. Therefore, it is of great significance to find an effective way for comprehensive utilization of Enteromorpha, as well as for the restoration and improvement of the ecological environment. But relevant references about Enteromorpha reuse are few since studies are mainly focused on its chemical and bioactive characterization [21,22]. The chemical composition of Enteromorpha was presented in Table 1, which showed that the proportion of polysaccharides is high up to 54% in dry matter [23,24]. According to the research by Qi et al., the molecular weight of Enteromorpha polysaccharides (Ep) ranges from 55 to 511 kDa, and Ep have linkages of (1 → 4)-linked β-L-arabinopyranose, which consist of xylose, galactose, arabinose, rhamnose, and glucose [23].

To improve the dewatering efficiency and reduce secondary pollution in sludge treatment process, Enteromorpha (the by-products of ocean green tides), was used together with PAM to access its aid effects. In this way, lower PAM dosage was needed to achieve certain coagulation efficiency, and, meanwhile, a new kind of non-toxic coagulant aid could be provided.

2. Materials and methods

2.1. Waste sludge

The municipal sludge used in this study was sampled from the secondary settling tank of Guangda Wastewater Treatment Plant (WWTP) in Jinan, China. The plant treats 200,000 m^3/d of wastewater (90% domestic and 10% industrial sewage) by anaerobic-anoxic-oxic technology, which produces sludge about 60–80 m^3/d, and the final effluent meets China’s pollutant discharge standard for municipal WWTP (GB18918-2002). All the samples in this study were transported to the laboratory within 60 min after sampling, and precipitated 60 min before coagulation experiments. All the samples were analyzed within 24 h, and then new waste sludge should be sampled again from the WWTP for next experiments. The characteristics of the sludge were shown in Table 2.

2.2. Preparation of coagulant and coagulant aid

PAM was purchased from Kolon Co., Korea, and its properties were listed in Table 3. PAM solution (1 g/L) was prepared by dissolving PAM directly in deionized water, and then stirred for 24 h using a magnetic stirrer until PAM dissolved completely. The dissolved PAM was used as coagulant.

Table 1
The chemical composition of Enteromorpha

Composition	Polysaccharide	Protein	Cellulose	Fat	Magnesium	Potassium	Sodium	Calcium	Other
Percentage (%)	53.71	27.03	10.16	0.88	2.73	1.19	0.87	0.72	2.71

Table 2
Characteristics of waste sludge collected from Guangda WWTP

MLSS (mg/L)	MLVSS (mg/L)	Moisture content (%)	pH	Calorific value (MJ/kg)
5,260 ± 125	1,975 ± 78	97.8 ± 0.2	6.46 ± 0.22	7.761 ± 0.25

Notes: MLSS: mixed liquor suspended solids, MLVSS: mixed liquor volatile suspended solid.

Table 3
Properties of PAM purchased from Kolon Co., Korea

Model number	Molecular weight (Dalton)	Cationic degree (%)	Polymerization degrees (%)	Solid content (%)
K6641	8,000,000	62.5 ± 2.5	≥99.9	≥90

Fresh Enteromorpha was gathered from Qingdao, China. The preparation method of Ep was shown as follows: (1) Enteromorpha was dried at the temperature of 40℃ and then shattered into powder by a magnetic crusher; (2) the powder was mixed with deionized water at a mass ratio of 1:75 and then heated in water bath for 4 h at temperature of 90℃; (3) the extract of Enteromorpha was centrifugalized for 20 min at a speed of 5,000 r/min; (4) supernatant was concentrated by rotary evaporator and then alcohol precipitation was conducted to obtain Ep. The sediment Ep was dried by lyophilizer and used as coagulant aid. The molecular weight of Ep was 300,000–2,000,000 Dalton, which was measured by gel permeation chromatography. Ep solubility was 23.58 g/L and zeta potential of Ep solution was −35 ± 5 mV.

2.3. Jar test

Coagulation experiments were conducted by a jar test apparatus (ZR4-6, Zhongrun Water Industry Technology Development Co. Ltd., China) at a room temperature of 20 ± 1℃. Test sludge (1,000 mL) was poured into each of the 1,400 mL plexiglass beakers and a six-paddled stirrer was used for mixing. Initially, PAM was used as coagulant and different programs designed by orthogonal software were conducted to determine the optimum coagulation procedure.

Under the optimal conditions, Ep was used together with PAM for sludge treatment. According to previous study, when organic polymer was used as coagulant aids, the best results could be obtained when polymer was added after the addition of the primary coagulant [25]. Therefore, PAM was added firstly at the start of coagulation test, and then Ep was dosed 30 s later in this study, which was denoted as PAM-Ep. According to the results of orthogonal test, the coagulation procedure was shown as follows: initially sludge sample was mixed up by shear rate at 200 rpm for 30 s; then PAM was added, with simultaneously 30 s of rapid mixing, followed by Ep addition for another 30 s stirring. After that, the sample was exposed to a slow mixing at 40 rpm for 1 min, followed by 5 min of settlement period to allow the aggregated sludge particles to settle down. After quiescent settling, the sample was filtrated by vacuum suction. The liquid was directly used for measuring residual turbidity (with 2100P turbidimeter, Hach, USA), zeta potential (with zeta sizer 3000HSa, Malvern Instruments, UK), and capillary suction time (CST) (with Triton Type 304 M CST-meter, Triton Electronics Ltd., England). The sludge after coagulation was dried and then the morphology structure of sludge was observed with Scanning electron microscopy (SEM) (JEOL, JSM 7600F, Japan).

2.4. Measurement of sludge particle sizes

The particle sizes of sludge before and after coagulation were both measured by a laser diffraction instrument (Mastersizer 2000, Malvern, UK). The sludge was stirred at a medium-low speed (50 rpm) during the measurement to avoid sludge particles settling. Suspended sludge particles were monitored through the sample cell of Mastersizer and then transferred back into the jar by a peristaltic pump (LEAD-1, Longer Precision Pump, China) using a 0.5 cm internal diameter tube at a flow rate of 2.0 L/h. The pump was designed to be located downstream the Mastersizer so the sludge particles would not be disturbed before measurement. The inflow and outflow tubes were positioned opposite each other at a depth just above the impeller in the holding ports. Size measurements of the sludge particles were repeated five times and the results (the median volumetric diameter (d_{50})) were recorded automatically by computer.

2.5. Evaluation of sludge dewaterability

In this paper, sludge dewaterability was evaluated by the indicators of CST and resistance to filtration (SRF). CST was measured by Triton Type 304 M CST-meter directly, and SRF can be determined by Eq. (1) shown as follows [26–29]:

$$\mathrm{SRF} = \frac{2PA^2a}{\mu w} \tag{1}$$

where A is filtration cross-section area; P is applied pressure; μ is liquid viscosity; w is the mass of cake solids deposited per volume of filtrate; a is the slope of the linear plot of $(t - t_s)/(V - V_s)$ vs. $(V + V_s)$ t is filtration time or duration (t_s is filtration time at the beginning of constant pressure period; V is filtrate volume; V_s is filtrate volume at the beginning of constant pressure period). The corresponding derivation of formula and the calculation of *SRF* value can be found in the paper by Qi et al. [30]. In addition, the flow chart of all the experiment in this study was shown in Fig. 1.

3. Results and discussion

3.1. Orthogonal test

Coagulation processes were significantly affected by coagulant dosages and hydraulic conditions. Therefore, orthogonal test was conducted at the temperature of 20 ± 1˚C to determine the optimal condition for sludge pretreatment. The project of factors was shown in Table 4. As can be seen, three factors (PAM dosage, rapid stirring period, and slow stirring period) were selected and four levels were conducted, respectively. For all coagulation experiments, rapid mixing speed was maintained at 200 rpm while the slow mixing speed was constant at 40 rpm. PAM of different dosages (40–70 mg/L (sludge)) was used as coagulant, respectively, and the coagulation procedure was changed as shown in Table 4. Indicators in this paper included settling velocity, CST, filtration rate, supernatant turbidity, and light transmittance. The results of orthogonal test were listed in Table 5, which showed that the optimal condition varies for different indicators. Considering all indexes comprehensively, the coagulation procedure of number 10 was selected as the optimum program, because under this condition, the minimum CST, and supernatant turbidity were achieved and meanwhile settling velocity, filtration rate, and the light transmittance were nearly maximal.

Appropriate coagulant dosage will not only improve the effluent water characteristics, but also decrease the cost of water treatment processes. As can be seen from the results of orthogonal test: when PAM was used, CST and supernatant turbidity decreased as PAM dosage increased, and settling velocity, filtration rate, and the light transmittance showed an augment trend. This indicated that coagulation became more effective as PAM dosage increased, which was in consistence with the increase of zeta potential. The highest coagulation efficiency was achieved at the dosage of 60 mg/L with zeta potential of 0.7 mV. Previous studies have shown that if charge neutralization is the main path for coagulation, zeta potential should be in excellent correlation with coagulant dosage, and the optimal efficiency will be achieved when zeta potential is close to zero [31]. Therefore, charge neutralization was the main dominant mechanism of PAM coagulation system. Under this condition, coagulation was more affected by the charge effects and meanwhile precipitate seemed less important. Thus, efficient coagulation occurred at low PAM dosages, even though the zeta potentials were somewhat below zero (Table 6).

In order to give a visual presentation, the corresponding size distributions of sludge particles before and after coagulation were presented in Fig. 2, where the flocs sizes were represented by median equivalent diameter (d_{50}). As can be seen from Fig. 2, the sizes of initial sludge particles were about 60 μm, while when PAM was dosed, the peak moved to the right to a large extent, which indicated that the sludge particles became larger. In addition, there was an obvious decrease in the volume of the sludge particles with size below 100 μm, which meant that PAM addition could not only improve the average particle size, but also reduce the number of small particles. According to previous studies, smaller particles generally settle

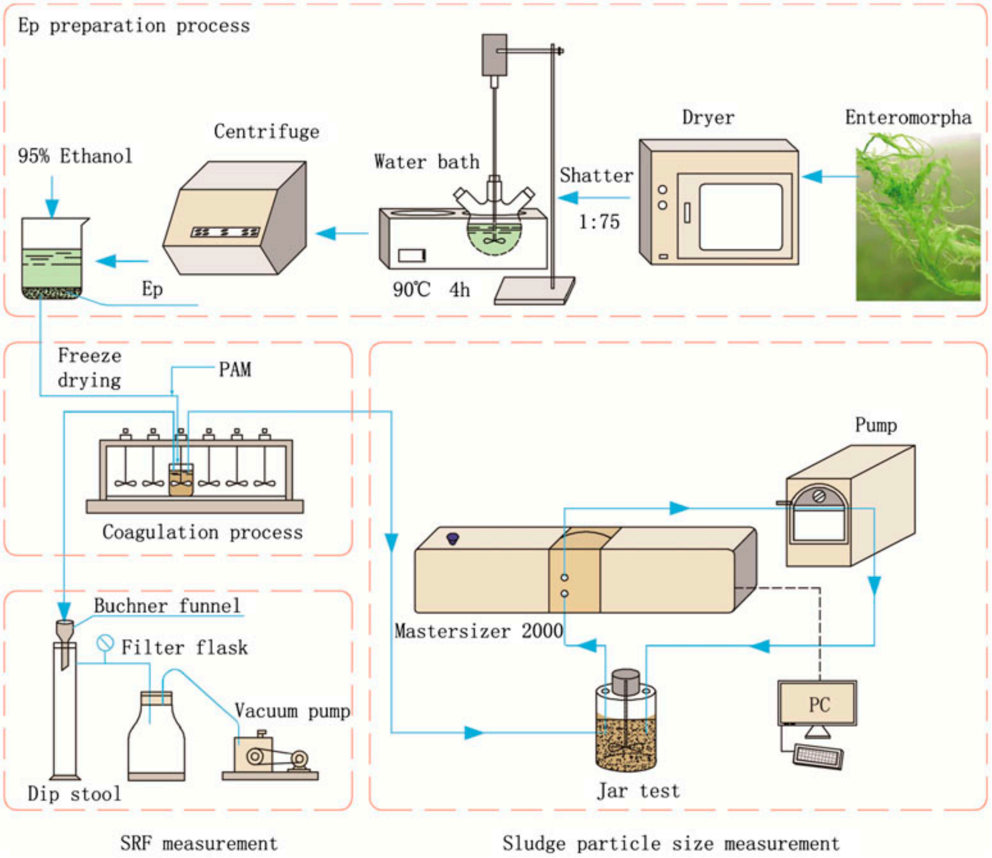

Fig. 1. Flow chart of Ep preparation, coagulation, and size measurement process.

Table 4
Orthogonal table of three factors and four levels

Number	Dosage (mg/L(sludge))	Rapid stirring period (min)	Slow stirring period (min)
1	40	0.5	0.5
2	40	1	1
3	40	3	3
4	40	5	5
5	50	0.5	0.5
6	50	1	1
7	50	3	3
8	50	5	5
9	60	0.5	0.5
10	60	1	1
11	60	3	3
12	60	5	5
13	70	0.5	0.5
14	70	1	1
15	70	3	3
16	70	5	5

Table 5
Table of the orthogonal experiment results

Number	Settling velocity (mL/min)	CST (s)	Filtration rate (mL/min)	Supernatant turbidity (NTU)	Light transmittance (%)
1	5.3	187.4	0.56	26.4	27.54
2	6.3	180.7	2.75	37.7	29.24
3	6.7	176.3	3.67	23.2	28.42
4	6.5	166.3	7.50	21.7	21.99
5	8.2	138.4	5.30	22.9	33.96
6	8	128.1	6.14	17.0	34.83
7	6.8	67.1	5.80	19.6	32.06
8	4.8	86.4	6.21	8.2	38.28
9	9.2	46.0	7.66	9.2	36.19
10	8.9	30.5	7.25	7.7	39.11
11	9.2	57.1	6.43	11.2	37.75
12	8.2	60.1	9.02	8.3	38.11
13	8.5	38.7	8.21	13.21	30.12
14	6.5	74.5	9.10	15.3	26.05
15	7.0	81.5	8.75	13.3	23.22
16	8.5	66.1	8.45	18.7	26.43

Table 6
Particles size ($d_{0.5}$) and zeta potential before and after coagulation

	PAM dosage (mg/L)				
Parameter	0	40	50	60	70
$d_{0.5}$ (μm)	59	386	558	656	670
Zeta potential (mV)	−9.5	−7.2	−3.8	0.7	2.5

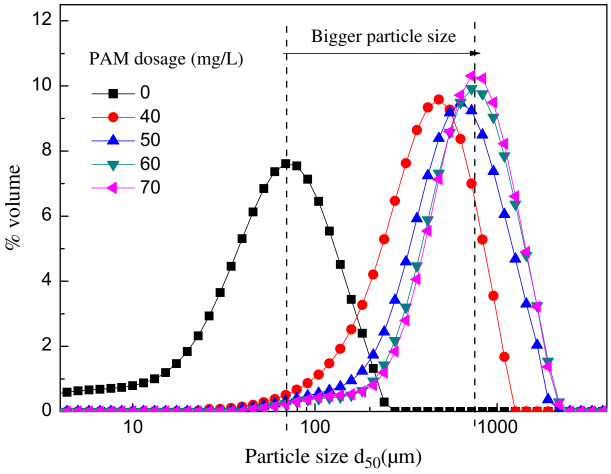

Fig. 2. Size distribution of sludge particles with different PAM dosages.

down more slowly [32]. Therefore, coagulation as pretreatment method could provide a great advantage for sludge settling velocity. But when PAM dosage increased from 60 to 70 mg/L, the shift of peak was not obvious, which can be explained as follows: there were not enough particles generated by PAM at smaller dosages, hence the effective collision could be enhanced with PAM dosage increasing. However, when PAM dosage further increased (>60 mg/L), the superfluous PAM may have negative influence on particle aggregation. Since all particles were positively charged, the repulsive force between sludge particles impeded the growth of particles. Therefore, the sizes of sludge particles could not increase further more. So the optimal PAM dosage was 60 mg/L, which was in accordance with the conclusion of Table 5 as shown above. Therefore, the following coagulation program was determined: PAM dosage 15 mg/L, rapid mixing period 1.0 min, and slow mixing period 1.0 min.

3.2. Effect of Ep on coagulation efficiency

In this section, a series of coagulation tests were conducted to study the role of Ep as coagulant aid, and meanwhile to ascertain the optimum Ep dosage for sludge dewatering. PAM-Ep and PAM were comparatively evaluated in terms of coagulation performance, including filtrate volume, solid content, turbidity, and SRF. PAM dosage was fixed at 60 mg/L (sludge) based on Section 3.1, and Ep dosage range was from 1 to 4 mg/L(sludge). Coagulation procedure was shown in Section 2.3, and the results were shown in Fig. 3.

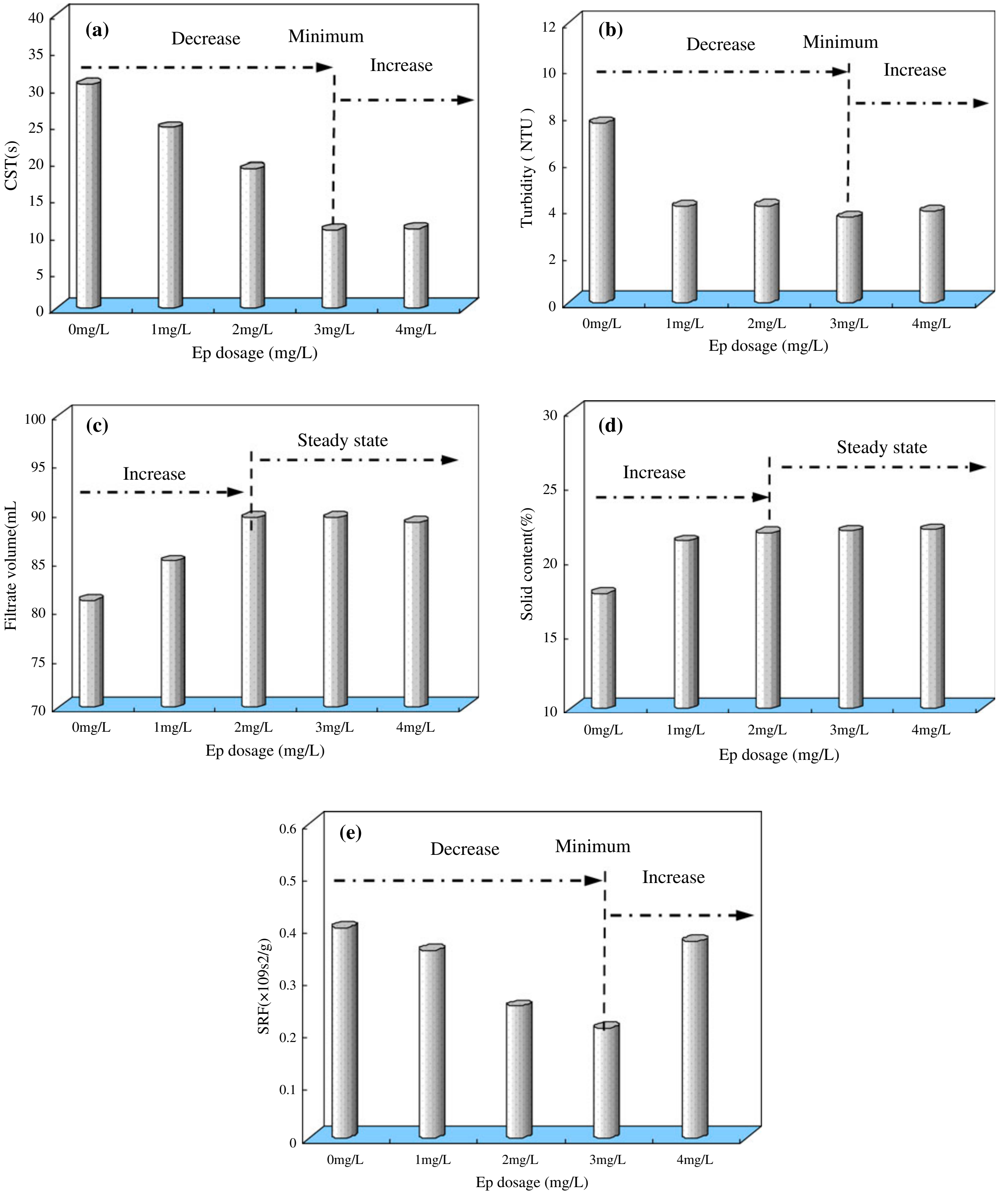

Fig. 3. Effect of Ep dosages on coagulation performance: (a) CST; (b) Turbidity; (c) Filtrate volume; (d) Solid content; (e) SRF.

As can be seen from Fig. 3, when adequate Ep was used in conjunction with PAM, the filtrate volume and solid content could be enhanced apparently; meanwhile, turbidity and SRF were reduced, which indicated that Ep could be used as a new efficient coagulant aid. Especially when Ep dosage was 3 mg/L, the coagulation efficiency was significantly improved: the CST and residual turbidity of filtrate was 10.7 s and 3.63 NTU at the PAM dosage of 60 mg/L, whereas 30.5 s and 7.7 NTU were obtained when PAM was used alone. But under this condition, zeta potential was still below zero, which suggested that charge neutralization was not the dominant mechanism for sludge treatment by PAM-Ep. PAM would quickly adsorb on the surface of the sludge particles when dosed firstly and then neutralized the negative charge on it. Hence, repulsion forces between the colloids became very weak. Then, the adsorption bridging ability of Ep could have a significantly positive effect on the gathering process of particles, which could generate larger particles with preferable settling ability [33]. SEM was also used to further explore the surface ultrastructure of treated sludge (Fig. 4). According to SEM analysis, the three-dimensional net structure of polymer was formed in PAM-Ep coagulation system, which can be attributed to the bridging role of Ep. As can be seen from Fig. 4, the uniform distribution of Ep provides a large number of channels and holes, which could form sludge cake with porous structure. Therefore, the permeability of the sludge was improved greatly and the resistance of sludge could be decreased.

3.3. Effect of Ep on sizes of sludge particles

Table 7 indicated that particles sizes in PAM-Ep coagulation system were larger than those of PAM: at constant PAM dosage of 60 mg/L, the sludge sizes could reach 791 μm when 3 mg/L Ep was used, which is larger than when PAM was used alone (656 μm). The phenomenon mentioned above may be related to the dominant coagulation mechanism. As discussed in Section 3.2, the primary mechanism of PAM was charge neutralization while extra adsorption bridge was dominant when PAM-Ep was used. Ep promoted the growth of sludge particles by adsorption bridge ability due to its natural polymers. This result was in agreement with the observation of Ray and Hogg [34], who found that flocs produced by bridging flocculation and charge neutralization were much larger than those formed simply by charge neutralization. Therefore, better coagulation efficiency could be achieved when Ep was used as coagulant aid. However, when Ep dosage was 4 mg/L, coagulation efficiency became low: SRF value achieved $0.37 \times 109\ s^2/g$, while SRF was only $0.37 \times 109\ s^2/g$ when 3 mg/L of Ep was used. As can be seem from Table 7, when Ep was overdosed (4 mg/L), zeta potential of sludge system was under −15 mV. Fig. 5 also showed that the particle size was almost overlapping when Ep dosage was larger than 3 mg/L. This can be explained as follows: the bridging role of Ep led to the increase of sludge particle aggregation at the initial aggregation stage. There were not enough macromolecule matter particles when Ep dosage was small, so the adsorption bridge effect would be enhanced with the increase of Ep dosage. But, all particles were positively

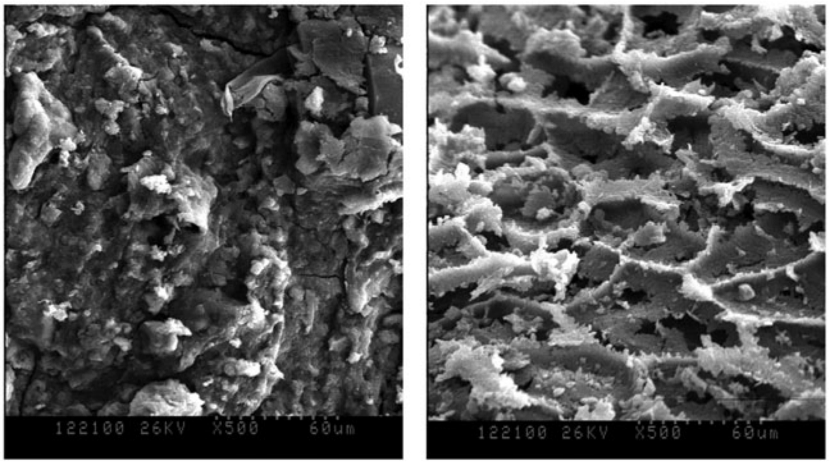

Fig. 4. Scanning electron microscope photographs of treated sludge: (Left: PAM was used, Right: PAM and Ep were used).

Table 7
Particles size and zeta potential under different Ep dosage conditions

	Ep dosage (mg/L)				
Parameter	0	1	2	3	4
$d_{0.5}$ (μm)	656	715	748	791	783
Zeta potential (mV)	0.7	−5.6	−8.4	−12.9	−15.8

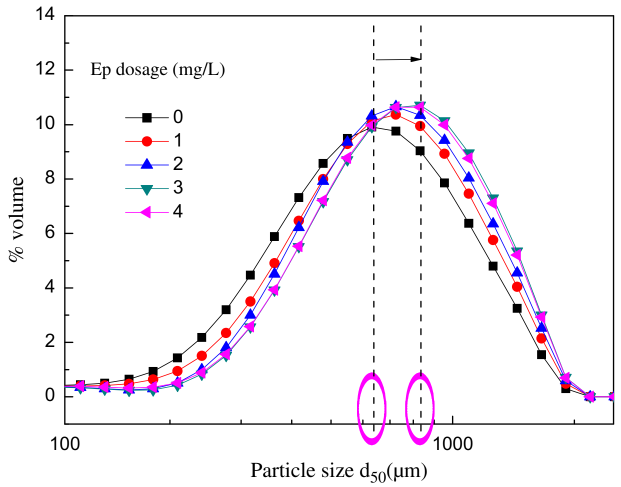

Fig. 5. Size distribution of sludge particles with different Ep dosages.

aggregated and precipitated when Ep dosage was larger than 3 mg/L. Therefore, the due role of extra Ep could not be realized. Accordingly, the floc sizes were hard to further increase. Additionally, since Ep was negatively charged (zeta potential was about −35 mV), stronger inter-particle repulsion forces would restrain the growth of sludge particles. Therefore, the coagulation efficiency could not be enhanced further when Ep dosage was larger than 3 mg/L. Consequently, based on the figure and discussion above, 3 mg/L was selected as the optimal Ep dosage.

3.4. Cost-benefit analysis

The preparation method of Ep was shown in Fig. 1, which showed that only three low energy and inexpensive equipment were needed: a vacuum oven, a water bath, and a centrifuge. Additionally, the sole reagent involved for Ep preparation was ethanol, which is also inexpensive. Above all, the raw material-Enteromorpha could be obtained from coastal cities for free. Considering all the costs including transportation, energy, equipment, and reagents, the price of Ep should be about 500–700 CNY/ton. In contrast, the price of commonly used PAM in China is about 20,000–25,000 CNY/ton. Therefore, the production cost of Ep is much lower than that of PAM.

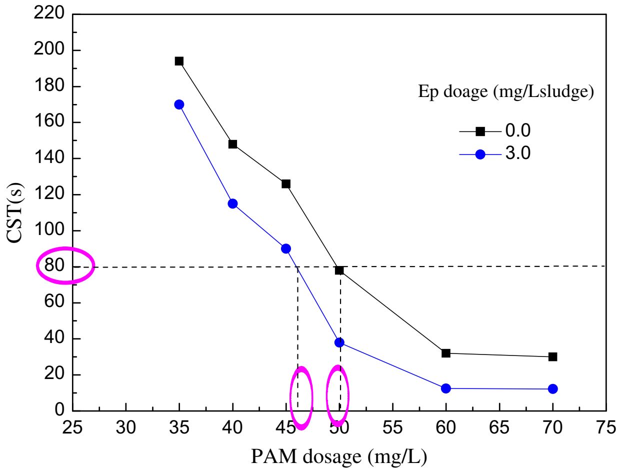

Fig. 6. Effect of Ep on CST variation under different PAM dosage conditions.

As discussed in Section 3.2, the addition of Ep could enhance coagulation performance of PAM. In other words, to achieve the same coagulation efficiency, lower PAM dosage was needed. As can be seen from Fig. 6, when 50 mg/L of PAM was used, the residual CST was 80 s. However, when 3 mg/L of Ep was added, only about 46 mg/L of PAM was needed to achieve the same CST level. Therefore, adding Ep as a coagulant aid has a great advantage for decreasing the PAM dosage (about 10%), which is a benefit for water treatment plants since the price of PAM was really high.

4. Conclusions

Results of this paper indicated that Ep could be used as a new coagulant aid in sludge pretreatment process. The following conclusions can be obtained:

1. In sludge treatment process, the optimum rapid stirring period and slow stirring period were both 1 min. When Ep was applied with PAM, the coagulation efficiency could be greatly improved, and the optimum PAM and Ep dosage was 15 and 3 mg/L, respectively.
2. Loosely and porous structure of sludge cakes was obtained due to Ep addition, which lead to larger floc sizes and better dewatering ability of treated sludge.
3. The main coagulation mechanism of PAM was precipitate charge neutralization, and Ep could

play a positive aid role by adsorption bridging effects. Therefore, better coagulation performance could be achieved by the combination of two advantages.

(4) Ep addition could reduce the cost of sludge treatment by reducing PAM dosage. In addition, the cost of Ep preparation was relatively low.

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