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Influence of organic flocculants on the flocculation performance of aerobic sludge

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ABSTRACT

This study used cationic polymers poly (methacryloyloxyethyl trimethyl ammonium chloride-acrylamide, MPAM) synthesized in laboratory and poly (acryloyloxyethyl trimethyl ammonium chloride-acrylamide) (APAM) commercially available for aerobic sludge flocculation. The relationship between the dosage and the flocculation effect was investigated, and the influence of the polymer structure and cationicity on the flocculation performance was compared. The results revealed that the polymers with 10%–50% cationicity were suitable to flocculate the aerobic sludge. The flocculation efficiency of MPAM was better than APAM, which can attribute to the enhanced inhibitive hydration effect through compressing the double electrode layer by the methyl group in methacryloyloxyethyl trimethyl ammonium chloride (DMC). MPAM with 50% cationicity exhibited superior dewatering efficiency, a low moisture content of 75% and a specific transmittance of 97% at dosage of 90 mg L^{-1}. Consequently, MPAM, especially the polymer with 50% cationicity, is suggested as a promising and sustainable candidate in aerobic sludge dewatering agent. Furthermore, the possible flocculation mechanism involved in the flocculation process is proposed.

Keywords: Methacryloyloxyethyl trimethyl ammonium chloride; Acryloyloxyethyl trimethyl ammonium chloride; Flocculation; Aerobic sludge; Cationicity

1. Introduction

The constant increase in the production of organic wastes, especially from the alcohol production sector, and the concern for environmental issues associated to its management, such as gaseous emissions or wastewater treatment, has led all countries to adopt various regulatory measures in order to minimize waste and to enforce good management [1]. Alcohol production industry is one of the highest organic pollutant concentration industries [2]. The wastewater has high content of organic matter. PH is lower than 4.0 and chemical oxygen demand (CODcr) is more than 30,000 mg L^{-1}. Generally, this kind wastewater is disposed by the biochemical treatment method due to the advantages of high removal rate, relatively low cost and simple operation [3–5]. Nevertheless, the residual sludge after biochemical treatment is always a tough situation, especially the aerobic sludge [6–8]. Because it has high water content (>97%), in the presence of organic components (bacterial cells and extracellular polymeric substances), colloidal specialty, and biological gel structure properties [9,10]. The methods of aerobic sludge treatment have been investigated for some decades, especially those concerning solid–liquid separation [5]. The most prevalent method is using the chemical condition by inorganic or organic coagulants for the sludge flocculation process [11,12]. Among all coagulants, polyacrylamides (PAMs) have been extensively used in the process of sludge dewatering owing to their advantages such as low dosage, high efficiency, facile operation, and affordable price [13–15].

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However, the sludge derived through commercially available PAMs treatment followed by mechanical dewatering always contains high moisture (>80%) which will severely affect subsequent disposal in landfills or the incineration or composting of aerobic sludge. These complications caused sludge dewatering as a universal issue [12].

Previous literature concerning the sludge treatment and disposal suggested that the structure, rheological characteristics, and dewatering ability of sludge is directly influenced by the flocculation of organic polymer flocculants with different structure and charges [16]. Otherwise, the dewatering and drying effect are closely related to the sludge properties and the characters of used coagulants [17]. Despite the fact that the various types of commercial PAMs have been developed and used successfully in sludge dewatering, the acryloyloxyethyl trimethyl ammonium chloride-acrylamide (APAM) and methacryloyloxyethyl trimethyl ammonium chloride-acrylamide (MPAM) were still the two most commonly used of all PAMs, and it still needs to improve their efficiency and develop the property of polymers for the enhancement of sludge dewatering performance. Regretfully, a significant drawback in the application exists: the polymer MPAM is concentrated on the low cationicity, especially on the concentration lower to 30% (the molar ratio of cationic monomers to total monomers) and the molecular weight is not high enough, which would make an unsatisfactory result in some occasions for flocculation. Furthermore, influence of these two polymers structure on the flocculation performance for different kinds of sludge remains to be studied. As a result, APAM is used mostly for sludge dewatering despite the sludge properties.

The aim of this work was to provide insights on the flocculation efficiency of fresh, aerobic sludge using cationic polymers, characterizing the filter cake moisture content, supernatant properties (transmittance and CODcr) and zeta potential, analyzing the influence of polymer structure on the flocculation performance of aerobic sludge and give a suggestion on the aerobic sludge treatment.

2. Experimental

2.1. Sludge sample

The study was performed on aerobic sludge samples collected after thickening from the plant using cassava starch producing alcohol, located in Lianyungang, Jiangsu province, China. This plant treats their wastewater by biochemical technology and has removed most of the organic carbon, nitrogen as well as phosphorous. CODcr of the wastewater has reduced from 30–40 thousand to 500. Then, the aerobic sludge is disposed of by a cationic polymer. Unfortunately, the effect of flocculation and dewatering is too poor after treatment. Fig. 1 is the flow chart of wastewater treatment by this plant (the red frame is the point for aerobic sludge at this experiment).

Before sludge used in the flocculation step, it is characterized at the beginning. Adequate amounts of sludge from aerobic treatment tank were taken out carefully. A small amount of sludge was separated into solid and supernatant by centrifuge, for measuring pH, CODcr, transmittance (T), and the zeta potential. Then some well-stirred sludge was put into a pre-weighed watch-glass, and the watch-glass was put into the oven with 105°C after weighed. When the sludge was baked to constant weight, the weight loss was weighed, and then the moisture content (MC) of sludge could be calculated. The result was listed in Table 1.

2.2. Flocculants

Five kinds of high molecular weight polyacrylamide Poly (methacryloyloxyethyl trimethyl ammonium chloride-co-acrylamide, MPAM) with serial cationicities (the molar ratio of cationic monomer DMC to total monomers DMC and AM) were synthesized in laboratory. Aqueous solution polymerization with one stage addition of the monomers and stepwise increasing temperature was adopted to synthesize the copolymers [18,19]. Intrinsic viscosities (η) were used to assess the polymer molecular weight. Four kinds of Poly (methacryloyloxyethyl trimethyl ammonium

Table 1
Sludge characteristics

pH	7.81
MC, %	98.12
T, %	68.6
CODcr, mg L^{-1}	474.5
Zeta, mV	–21.66

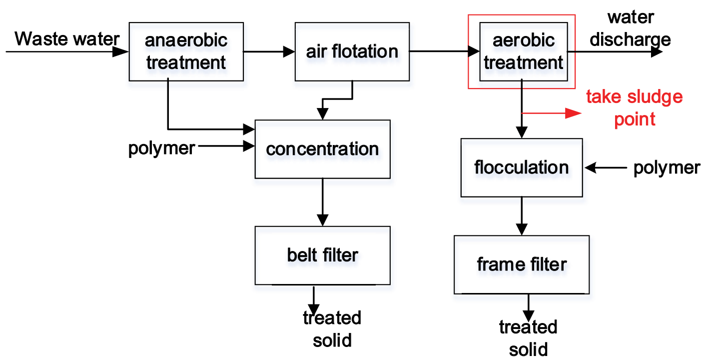

Fig. 1. Flow chart of waste water treatment by the plant.

chloride-co-acrylamide, APAM), with serial cationicities were purchased from SNF FLOERGER Company, (France).

2.3. Characteristics of flocculants

In this section, MPAM with high molecular weight, 50% cationicity (MPAM-50) and APAM with the similar cationicity (APAM-50) were chosen for comparative analysis. Fourier transform infrared (FTIR) spectra of copolymers were recorded by infrared spectrometer (FTLA2000, ABB Bomen Inc., Canada). ^{1}H unclear magnetic resonance (^{1}H NMR) spectra were recorded by ^{1}H NMR spectrometer (Avance III-500-Hz, Bruker Corporation, Germany) in deuterium oxide.

2.4. Flocculation design

- The flocculants with different cationicities were preliminarily compared and selected, respectively, using floccules size and supernatant transparency as the criterion for further experiment. The flocculants, obviously to display a big floccules size and transparent supernatant were selected for next stage use.
- The selected agents were used to do the flocculation test. The parameters of filter cake moisture content, transmittance (T), CODcr, and zeta potential were characterized to investigate the relationship between the dosage and the flocculation effect.
- The different flocculation effect between MPAM and APAM were analyzed and the possible flocculation mechanism involved in the flocculation process was proposed.

2.5. Flocculation test

Sludge samples of 100 mL were used to glass beakers for flocculation. 45–105 mg L^{-1} dosage of flocculants was added to the glass beaker with a rapid stirring at 200 rpm for 30 s to achieve complete mixing of the flocculants and sludge, then a slow stirring at 50 rpm for 1 min to enhance the floccules growth, and no stirring for 2 min for the floccules settling, filtrated under vacuum of –0.085–0.100 Mpa for 2 min [20]. After filtrated, filter cake was transferred to a pre-weighed watch-glass for drying to constant weight in 105°C oven, and its moisture content (MC) was calculated through the weight loss. The transmittance (T) and zeta potential of supernatant were conducted on a UV-2000 spectrophotometer (Unocal Shanghai Co., Ltd., China) and JS94H micro electrophoresis (Shanghai Zhongchen Digital Equipment Co., Ltd., China), respectively. CODcr was measured according to standard methods [21].

3. Results and discussion

3.1. Characterization of copolymers

The FTIR spectra of MPAM-50 and APAM-50 were investigated and the results were shown in Fig. 2. It was found that the absorption peaks of the MPAM-50 and APAM-50 were similar except for the bending vibration of methine in APAM-50 at 1,365 cm^{-1} [13,22]. It means that the two polymers had a similar functional group.

The characteristic peaks in the ^{1}H NMR spectra of MPAM-50 and APAM-50 were indicated in Fig. 3. Comparing these two spectra, we find that the absorption peaks of the protons of $–CH_2–$ (a), $–CH_2–$ (c), –CH– (b), $–COOCH_2–$ (e), $–CH_2–N^+$ (f), and $–(CH_3)_3–N^+$ (g) in MPAM and APAM were almost the same related to δ = 1.64, 1.73, 2.18, 4.58, 3.77, and 3.26 ppm, respectively, except for $–CH_3$ (d) and –CH– (d′), which were belong to DMC and DAC, respectively [22,23]. The spectral analytical results indicated the formation of MPAM and confirmed the structure of purchased polymers APAM.

3.2. Preliminary screening flocculants

Previous literature concerning in the sludge treatment and disposal suggested that different charge densities of organic polymer flocculants would directly influence the flocculating and dewatering ability of sludge [17,24]. Different charge densities of polymers mean different cationicities. In case of flocculant, the higher the intrinsic viscosity is, the less the amount of input and the better the performance is during flocculation progress. Here, the intrinsic viscosities of MPAM used were 15.51–14.13 dL g^{-1} and the cationicities were 10%–90%, while the intrinsic viscosities of APAM were 16.47–12.90 dL g^{-1} and the cationicities 10%–70%. The intrinsic viscosities of polymers were similar but cationicities were different. To select the polymers, sufficient dosage of flocculant was added to the glass beaker and stirred. The floccules size and supernatant transparency were recorded to evaluate the flocculation ability. Fig. 4 illustrated the effect of cationicity on the floccules size. It shows that the difference of their floccules size distribution was evident. The floc size increased from 3 to 5 mm, then decreased to 1 mm with the increasing cationicity. Furthermore, MPAM-50 and APAM-50 exhibited higher Floc size than others. The supernatant was transparent when the cationicities of polymers were 10%–50%. However, 70%–90% was turbid through observation. Generally speaking, the polymer with higher cationicity had a higher charge neutralization capacity. More particles were tightly absorbed on the polymer chain to form

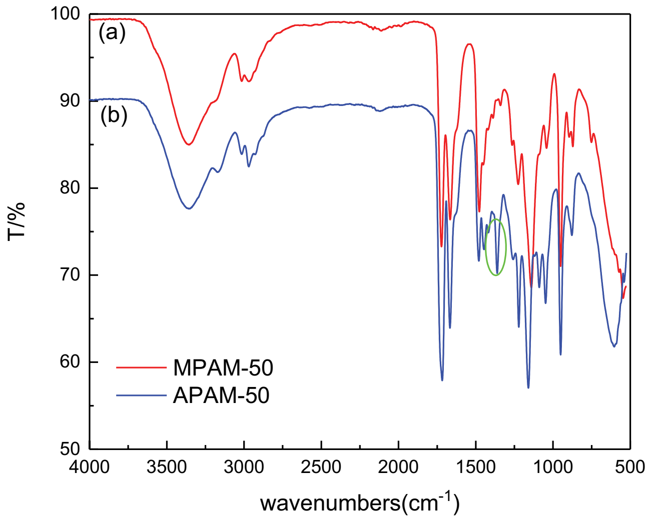

Fig. 2. FTIR spectra of (a) MPAM-50 and (b) APAM-50.

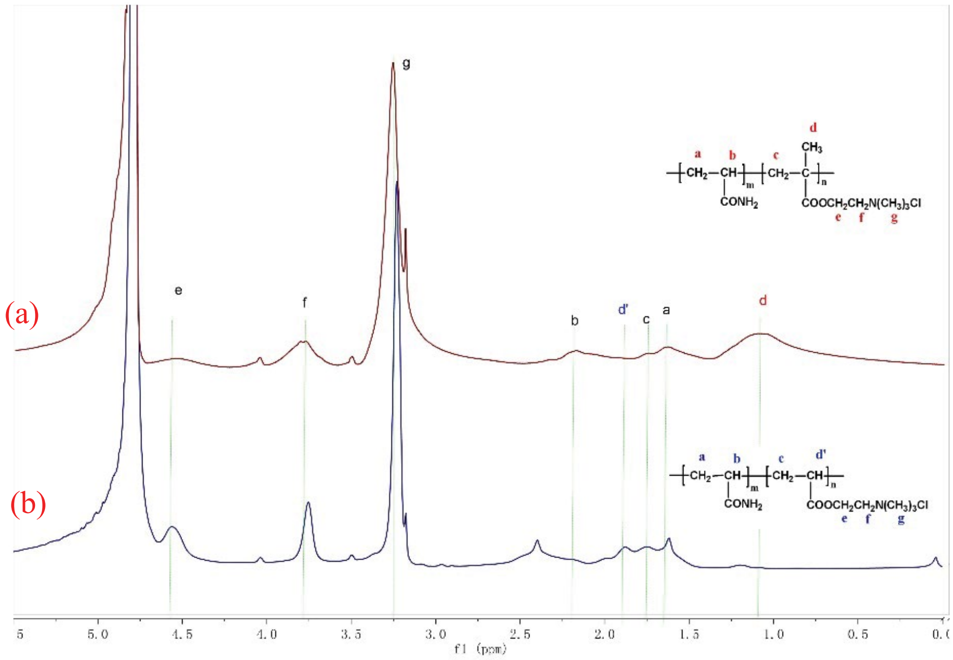

Fig. 3. ^1H NMR spectra of (a) MPAM-50 and (b) APAM-50.

a larger and compact floccule. Meanwhile, the repulsion between charged units could induce the chain expansion and embed the sludge solution, leading to smaller floccule size and turbid supernatant. In this condition, the charge density of 10%–50% cationicity, especially at 50% cationicity, was suitable to flocculate the aerobic sludge, which was beneficial for the large floccule and transparent supernatant formation.

3.3. Flocculation test

According to the preliminary selecting flocculants results, four polymers were selected to flocculation test. They were MPAM-10, MPAM-50, APAM-10, and APAM-50. The aerobic sludge was treated by the selected flocculants.

3.3.1. Effects of the dosage on the sludge dewatering performance

Moisture content (MC) has been widely used as means of gauging sludge dewatering. Generally, it is considered that the higher MC value indicates the worse sludge dewaterability [12]. The impact of polymer dosage on sludge dewatering performance was investigated and the results were displayed in Fig. 5.

It shows that the MC of filter cakes for the four flocculants decreased with increasing flocculant dosage before reaching the minimum value at 90 mg L^{-1}, and then increased within the flocculant dosage from 90 to 105 mg L^{-1}. When the flocculant dosage was low, the number of the positive charges and the surface active sites of the flocculants were insufficient, and which could not effectively neutralize and adsorb sludge particles, thus resulting in a high MC [20,25]. However, superfluous flocculant dosage would make the colloidal system highly positive charged, which re-stabilized sludge particles through electrostatic repulsion, thereby reducing the dewatering effect [14].

The fact that the minimum MC value of MPAM-10 was 80.93%, higher than MPAM-50 (75.23%) and of APAM-10 was 90.51%, higher than APAM-50(84.05%), respectively, indicated the higher cationicity of flocculant achieved lower moisture content, which means the more cationic unit, the higher the dewatering efficiency. The reason may be that the aerobic sludge with a certain negative charge was a stable aqueous colloidal system. When the cationic polymer flocculants only with proper cationicity, the balance of the original aqueous colloid system would be broken up, and large amount of suspending particles together were absorbed to form flocs based on the entire function demonstration of charge neutralization and electric double layer compression, bridging and net-capturing [26].

Notably, the minimum MC values of MPAM-10 (80.93%) and MPAM-50 (75.23%) were both lower than APAM-10 (90.51%) and APAM-50 (84.05%). Furthermore, MPAM-50 exhibited the best result in a full dosage range among the four flocculants. The discrepancy of the dewatering efficiency of these results revealed that MPAM was more suitable for the sludge dewatering. Particularly, the filter cakes treated by APAM looked like gelation texture, containing much colloid with intercellular water. It was difficult to dehydrate by simple filtration. However, the cakes by MPAM were compact flocs, as shown in Fig. 6. The reason may be that methacryloyloxyethyl trimethyl ammonium chloride (DMC) had one more methyl group than acryloyloxyethyl trimethyl ammonium chloride (DAC), which made the hydrophobic property of polymer MPAM better than APAM, resulting in better inhibitive hydration through compressing the electric double layer in sludge surface.

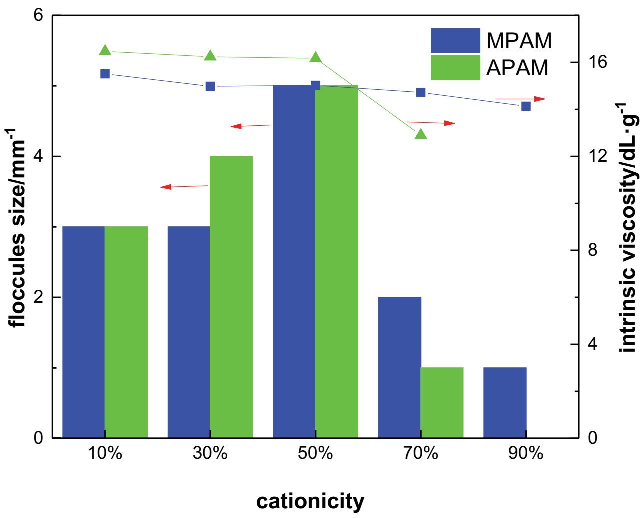

Fig. 4. Effects of the polymer cationicity on the floccule size.

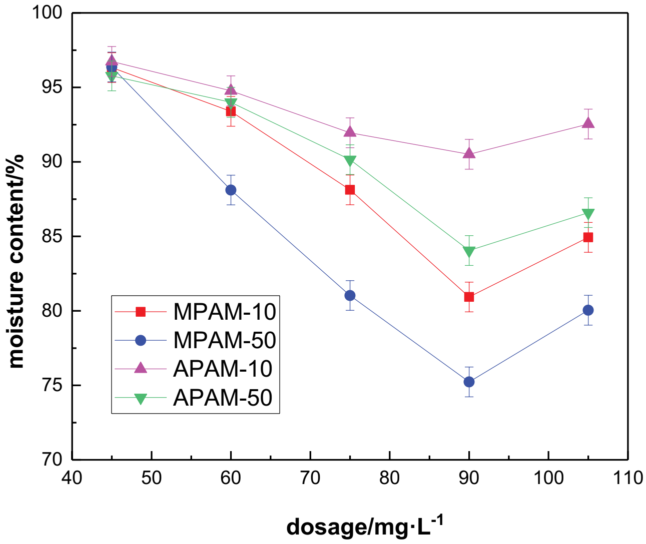

Fig. 5. Dosage of flocculants on MC.

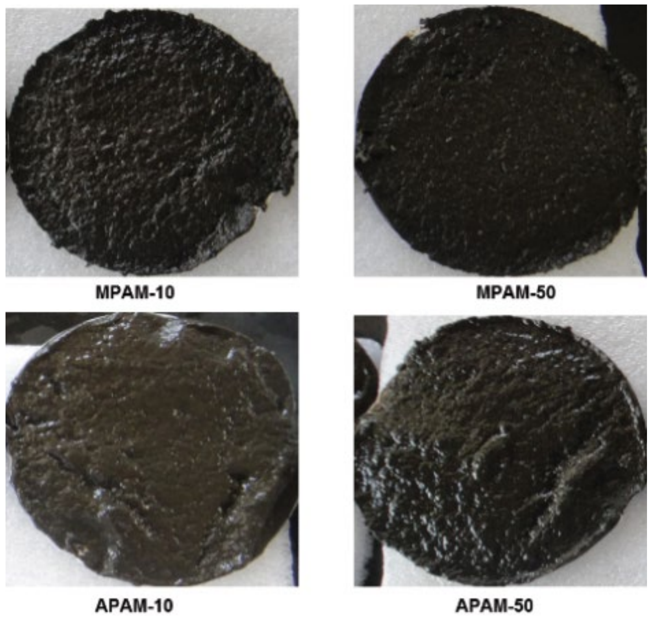

Fig. 6. Filter cake figures.

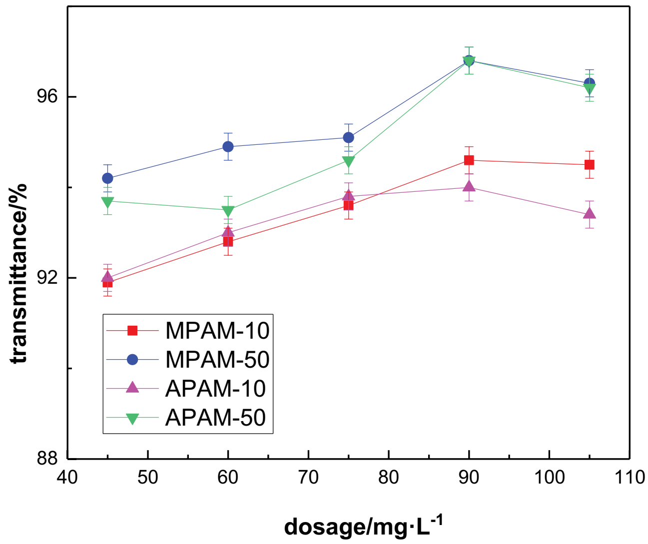

Fig. 7. Dosage of flocculants on transmittance.

3.3.2. Effects of the dosage on the supernatant

The supernatant property plays an important role in evaluating flocculation efficiency. Transmittance and CODcr reduction are intuitive and have been extensively used to characterize the supernatant state, in which a bigger value indicates better flocculation efficiency [27]. The relationship between the supernatant properties (transmittance and CODcr) and different dosages was studied. As shown in Figs. 7 and 8, the transmittance increased within the flocculant dosage from 45 to 90 mg L^{-1}, then decreased a little, while CODcr exhibited a contrary dependence. Fig. 7 showed that the transmittance could reach 96.8% using APAM-50 and MPAM-50, and APAM-10 exhibited the worst results of 93.4% transmittance with an adequate dosage. Meanwhile, Fig. 8 showed that MPAM-10 presented the highest CODcr of 399–390 mg L^{-1} when dosage of 45–75 mg L^{-1}, and APAM-10 presented the similar bad efficiency as MAPM-10 with the CODcr of 360–370 mg L^{-1} when dosage of 90–105 mg L^{-1}. Otherwise, another two polymers MPAM-50 and APAM-50 had the similar results about 350 mg L^{-1}.

The discrepancy of the transmittance and CODcr after treated by the four flocculants revealed that the flocculant with the more cationic unit, the higher the flocculation efficiency was. The reason for that was the CODcr reduction has a positive correlation with the molecular weight and cationicity of flocculant. MPAM-10 had smaller molecular weight than APAM-10. The long molecular chain could span and reduce the gap between particles, and the likelihood of collision among the polymer and colloidal particles increased. The particles absorbed on the polymer chain could aggregate and form large and compact floc through the bridging effect and sweeping mechanism [25]. Increasing the molecular weight of flocculant could improve its bridging action and netting ability, leading to reducing the number of colloidal particles, and improving the supernatant transmittance and CODcr reduction. Thus, APAM-10 performs better than the MPAM-10. Meanwhile, increasing the cationicity of flocculant could enhance its charge neutralization effect, and thereby the negative charged colloidal could be neutralized and destabilized by cationic flocculant to form large and compact flocs, and the strong charge repulsion between the polymer chains generated by the cationic unit was beneficial for the stretch and extension of molecular chain, thus strengthening the bridge effect, and further increasing the supernatant transmittance and CODcr reduction [20,28].

3.3.3. Effects of the dosage on the zeta potential

The zeta potential of the sludge colloidal system was also measured to understand the dewatering action of cationic flocculants clearly, which displayed in Fig. 9. The zeta potential could represent the charge density and the damage degree of electric double layer of suspended colloid particles with negative charges. The higher zeta potential of the particles indicates the lower negative charge density and higher damage degree of electric double layer, which lead to lower repulsive force between particles and lower moisture content of the filter cake [17].

The curve shown in Fig. 9 revealed that, while increasing the flocculant dosage, the zeta potential of the sludge system increased from 45 to 105 mg L^{-1}. The aerobic sludge carried negative charge and the zeta potential was –21.66 mV. With the increase of flocculant dosage, charge neutralization with the particles increased and charged particles in the system decreased leading to the increase of zeta potential. The zeta potential of the supernatant conditioned with MPAM-50 and APAM-50 was much higher than those of MPAM-10 and APAM-10 at the same dose. This finding indicated that the flocculant with higher cationicity had higher charge neutralization ability in the flocculation process.

3.4. Flocculation mechanism

Based on the results of filter cake moisture content, CODcr, transmittance, and zeta potential, the possible flocculation mechanism involved in the flocculation process was summarized and displayed in Fig. 10. Since the aerobic sludge came from starch processing technology, containing much of polysaccharide organic components. The surface of sludge particles could form hydrogen bonding with water to the stable colloidal. Because of the presence of methyl group in DMC unit in MPAM, hydrophobic performance was extremely increased, thereby greatly improving the dehydration property of the flocculant. As a result, the negatively charged sludge particles were neutralized completely and aggregated to form large and compact flocs under the charge neutralization and bridging by adding MPAM flocculant. However, when using APAM flocculant, although the sludge could be bridged and neutralized completely, it aggregated to form large but hydration flocs, lending to low treatment efficiency.

4. Conclusion

Cationic polymers MPAM with different cationicities were efficiently prepared using water solution polymerization of one stage addition of the monomers and stepwise increasing temperature. Two MPAM samples with different cationicity and two compared APAM samples were selected to treat on the aerobic sludge, which are collected from the plant using cassava starch producing alcohol. The efficiency of MPAM at dewatering and transmittance was better than APAM, which attributes to the enhanced inhibitive hydration effect through compressing the double electrode layer by the methyl group in DMC. In addition, the performance of the higher cationicity polymer was slightly better than that of the lower cationicity polymer due to the positive effect through improving the charge neutralization. Consequently, MPAM, especially the polymer with 50% cationicity was suggested as a promising and sustainable candidate for activated sludge dewatering.

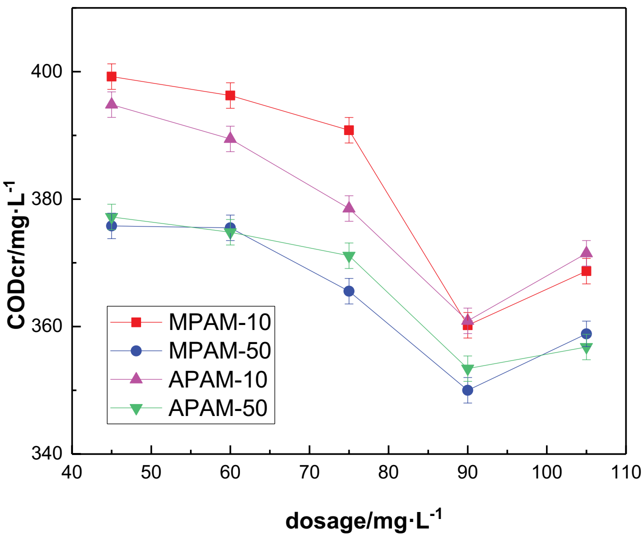

Fig. 8. Dosage of flocculants on CODcr reduction.

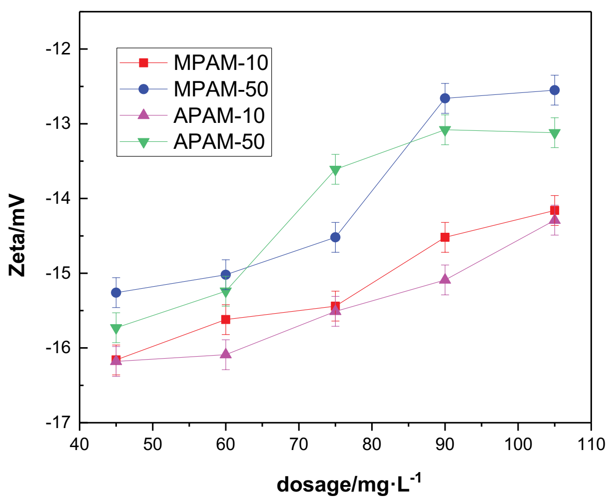

Fig. 9. Dosage of flocculants on zeta potential.

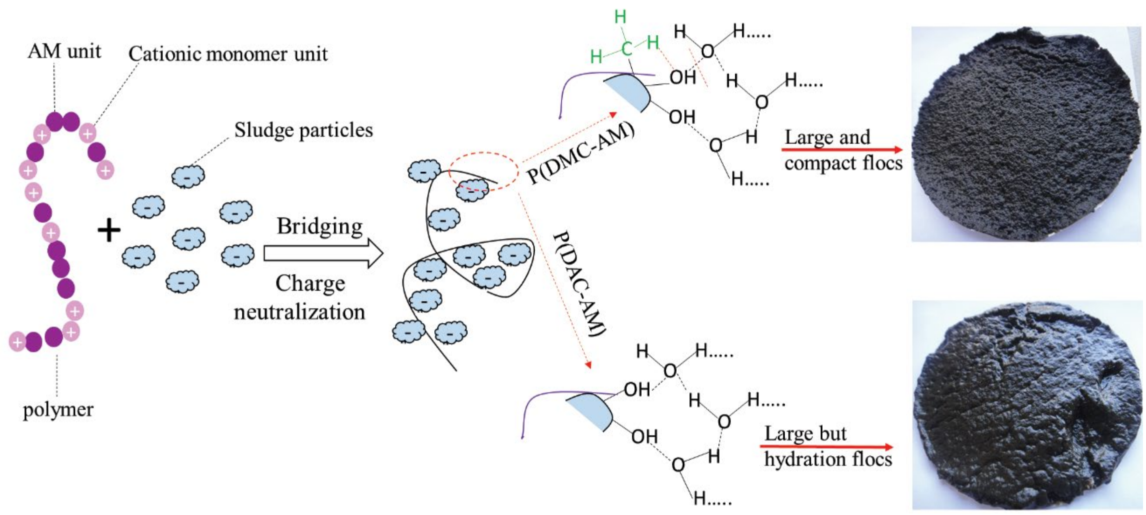

Fig. 10. Possible flocculation mechanism of MPAM and APAM.

Abbreviations

DMC	—	Methacryloyloxyethyl trimethyl ammonium chloride
DAC	—	Acryloyloxyethyl trimethyl ammonium chloride
MPAM	—	Poly (methacryloyloxyethyl trimethyl ammonium chloride-acrylamide)
APAM	—	Poly (acryloyloxyethyl trimethyl ammonium chloride-acrylamide)
PAMs	—	Polyacrylamides
T	—	Transmittance
MC	—	Moisture content
FTIR	—	Fourier transform infrared spectroscopy
^{1}H NMR	—	^{1}H unclear magnetic resonance
MPAM-10	—	MPAM with 10% cationicity
MPAM-50	—	MPAM with 50% cationicity
APAM-10	—	APAM with 10% cationicity
APAM-50	—	APAM with 50% cationicity

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