

Research Article

Study of Synthesis and Flocculation Properties of New Modified Hydrophobic Organic Polymer P(AM-DM-DMC12)

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A new modified hydrophobic organic polymer P(AM-DM-DMC12) is synthesized by free radical polymerization of three monomers in aqueous solution using a REDOX initiator system. Its structure is characterized by FT-IR and ¹H-NMR, the effect of reaction conditions on the product is studied, and the flocculation performance and mechanism are discussed with the aim of wastewater turbidity and COD removal rate. The results showed that the synthesized compounds accorded with the target structure, and the optimum synthetic conditions are as follows: the molar ratio of AM, DM, and DMC12 is 4 : 1 : 1, total mass fraction of monomer is 35%, the mass fraction of redox initiator is 0.06%, and the molecular weight of P(AM-DM-DMC12) is 1.02×10^7 . Moreover, the flocculation effect of P(AM-DM-DMC12) is obviously better than that of PAM, and it has the advantages of short flocculation time, low dosage, and wide application range of pH and has a good industrial application value and prospect.

1. Introduction

A flocculation sedimentation method plays an important role in the process of sewage treatment, which mainly makes use of the electrical neutralization, adsorption bridge, and electrostatic force of flocculant to coagulate with colloidal particles, solutes, and suspended solid particles in sewage to form larger floc and then destabilize and settle [1–3]. This method can not only reduce the turbidity and chroma of sewage but also remove the water of a variety of macromolecule organic substances, heavy metals, and radioactive substances, which can not only become an independent processing system but also be combined with other processing units as pretreatment, intermediate, or final processing process of sewage [4, 5], and is also often used to improve the dehydration properties of sludge [6–8]. The flocculation sedimentation method has been widely used in sewage treatment because of its simple process, convenient operation, good treatment effect, and wide application range.

The selection of flocculant is the key to the treatment of sewage by the flocculation sedimentation method [3, 9, 10].

According to chemical composition, flocculant can be divided into three categories: inorganic flocculant, organic flocculant, and microbial flocculant [11–14]. At present, most of the common traditional flocculation processes mainly combine inorganic flocculants with organic flocculants, such as using polyaluminum chloride (PAC) as flocculant and polyacrylamide (PAM) as flocculant aid, although it has certain effect on sewage treatment; their application is limited due to excessive dosage, corrosion-prone equipment, high cost, secondary pollution, and so on [1, 15, 16]. Therefore, it is vital to develop a new type of coagulant aid with high efficiency, reasonable price, and wide application range and that is nontoxic and harmless for the present situation of water treatment. For example, hydrophobic modification of ordinary cationic polymers is one of the very important and effective ways. The principle is that hydrophobic groups are embedded in polyacrylamide macromolecular chains containing hydrophilic groups to change and enrich the physical and chemical properties of polymers. Many studies have shown that hydrophobic groups have more effective contact adsorption for organic matter particles in sewage, which can optimize the

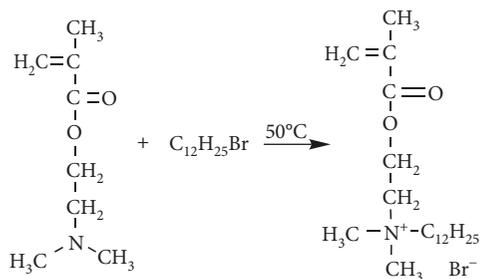


FIGURE 1: Schematic of synthesis of DMC12.

flocculation performance, reduce the dosage of flocculant, and make sewage treatment more economical [7, 17, 18].

In this study, three monomers of acrylamide (AM), dimethyl-ethyl methacrylate (DM), and hydrophobic monomer dimethylaminoethyl methacrylate-bromododecane (DMC12) were copolymerized in a redox initiator system by aqueous solution polymerization. A new type of organic polymer flocculant P(AM-DM-DMC12) was prepared. Its structure was characterized by FT-IR and $^1\text{H-NMR}$, and the synthetic conditions, flocculating properties, and flocculating mechanism were discussed to provide theoretical reference and basis for the development and application of the new flocculant.

2. Experimental

2.1. Materials and Instruments. Dimethyl-ethyl methacrylate (DM), acrylamide (AM), polyacrylamide (PAM), polyaluminum chloride (PAC), bromododecane ($\text{C}_{12}\text{H}_{25}\text{Br}$), acetone, ammonium persulfate ($(\text{NH}_4)_2\text{S}_2\text{O}_8$), sodium hydrogen sulfite (NaHSO_3), ammonium hydroxide, hydrochloric acid, absolute ethyl ether, and absolute ethyl alcohol were purchased from the National Pharmaceutical Group Chemical Reagent Co. Ltd. in China. Experimental water is deionized water.

The experimental instruments include the chroma detector (XZ-WS, Shanghai Experimental Instrument Factory), constant speed agitator (JJ-1, Changzhou Guohua Electric Co. Ltd.), ultraviolet visible spectrophotometer, electric vacuum drying oven (ZK-82A, Shanghai Experimental Instrument Factory), turbidimeter (2100AN, HACH), ultraviolet spectrophotometer (DR6000, HACH), Fourier transform infrared spectrometer (Cary 630, Agilent Technologies), nuclear magnetic resonance (AVANCE DMX500, Bruker), and pH meter (pHS-3C, Shanghai Lida Instrument Factory).

2.2. Synthesis of Target Polymers

2.2.1. Synthesis of Long Chain Hydrophobic Monomer DMC12. With DM and $\text{C}_{12}\text{H}_{25}\text{Br}$ as raw materials and acetone as reaction solvent, the viscous transparent liquid was obtained after heating in water bath to 50°C and stirring continuously for 20-24 h. After vacuum distillation and repeated washing and drying with anhydrous ether, the white powder (DMC12) was obtained and refrigerated for reserve. The reaction equation is shown in Figure 1.

2.2.2. Synthesis of P(AM-DM-DMC12). The monomers of AM, DM, and newly synthesized hydrophobic monomer DMC12 were weighed separately according to the molar ratio of 4:1:1, the solid AM was dissolved by a small amount of deionized water, and the mixture of several substances was poured into a flask with three necks. Then, the initiators ($(\text{NH}_4)_2\text{S}_2\text{O}_8$ and NaHSO_3) were put into the flask at the mass ratio of 1:1; ventilation with nitrogen and 70°C water bath mixing were for 5 h, with the speed of 200 RPM. A large amount of cotton-like flocculation was observed in the flask after being cooled to seal reaction. Then, washing and drying with anhydrous ethanol, the final product P(AM-DM-DMC12) was obtained after grinding. The reaction equation is shown in Figure 2.

2.2.3. Characterization of FT-IR and $^1\text{H-NMR}$. The structure of the product was characterized by Fourier transform infrared spectrometer (FT-IR) with the KBr pellet method and the nuclear magnetic resonance (NMR) [19, 20].

2.2.4. Determination of Molecular Weight. With reference to the relative molecular mass determination method (viscosity method) stipulated in GB 17514-2008 "Water treatment chemicals-Polyacrylamide" of Chinese national standard, the Ubbelohde viscometer was adopted for the measurement. The molecular weight M of the polymer was calculated by the following formula:

$$M = 802 \times [\eta]^{1.25}. \quad (1)$$

2.3. Flocculation Experiment. The stirring speed of the flocculation experiment is in the form of mixing and rotating at different rotating speeds from beginning to end. The first 5 minutes' rotating speed is 300 RPM, the second was rotated at 200 RPM for 20 minutes, and the last 10 minutes was rotated at 100 RPM. Therefore, the stirring speed is no longer discussed and analyzed separately during the whole experiment. In addition, the municipal domestic sewage (original turbidity is 217.8 NTU) was selected as the treatment object, and the large particulate matter was settled in order to avoid affecting the experimental results.

2.3.1. Flocculation Experiment of PAC. The target sewage was put into several 1000 mL beakers, and $1 \text{ mg}\cdot\text{mL}^{-1}$ PAC solution was added. The dosage of PAC was 10, 20, 30, 40, 50, 60, 70, and $80 \text{ mL}\cdot\text{L}^{-1}$, respectively. The turbidity and COD removal rate of each water sample were determined, respectively, after 35 minutes of stirring at mixing speed and a certain time of static settling (turbidity of the water sample was determined by a turbidimeter). The turbidity removal rate of the water sample was calculated by the following equation:

$$Q = \frac{C_1 - C_2}{C_1} \times 100\%, \quad (2)$$

where Q (%) represents the turbidity removal rate of the water sample and C_1 and C_2 (NTU) represent the original turbidity and present turbidity, respectively.

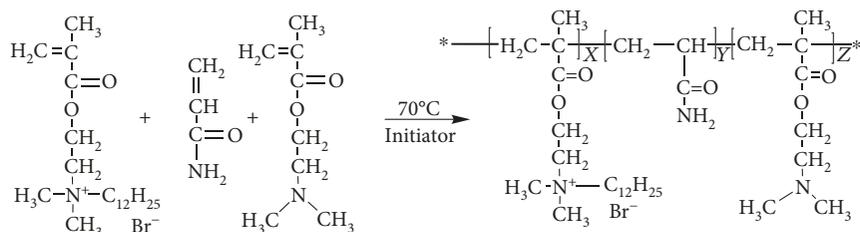


FIGURE 2: Schematic of synthesis of P(AM-DM-DMC12).

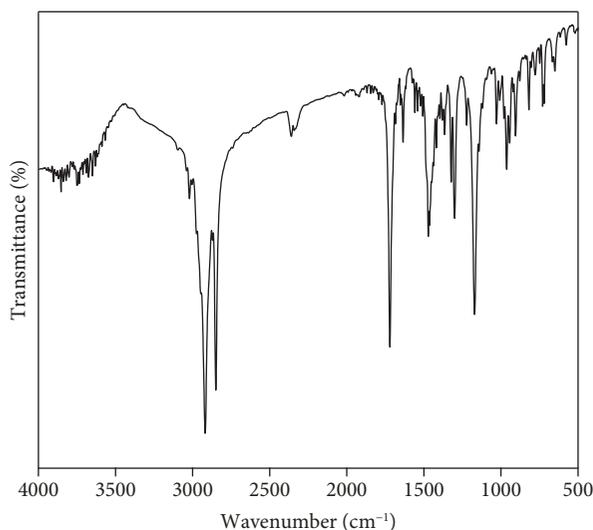


FIGURE 3: FT-IR spectrum of DMC12.

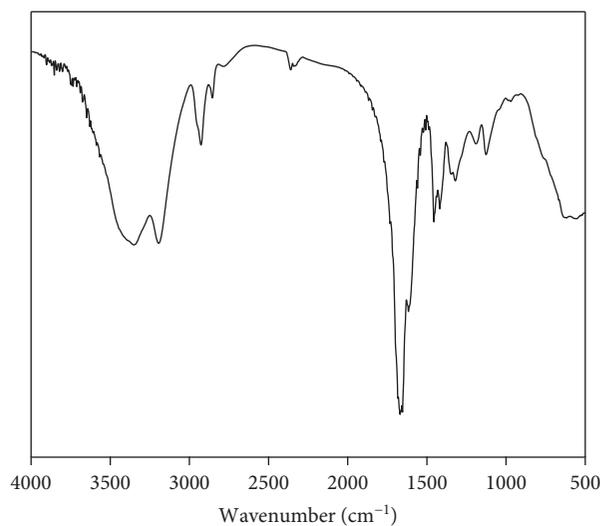


FIGURE 4: FT-IR spectrum of P(AM-DM-DMC12).

2.3.2. *Flocculation Experiment of PAC & P(AM-DM-DMC12) and PAC & PAM.* PAC was used as the main flocculant; PAM and P(AM-DM-DMC12) were used as the coagulant aid, respectively. Flocculation effects of different coagulant aids were explored through single factor experiments by changing the dosage, pH, and settling time.

The increase-decrease ratio of the dosage of flocculant and coagulant aid was 10:1, which were 60 + 1, 50 + 2, 40 + 2, 30 + 4, 20 + 5, and 10 + 6 mg · L⁻¹, respectively. The settling times were 0.5, 1, 1.5, 2, 2.5, and 3 h. Ammonia and HCl were selected for adjusting pH. Finally, the turbidity and COD removal rate of each water sample were determined.

3. Results and Discussion

3.1. *Structure and Characterization of DMC12.* The infrared spectra of the DMC12 synthesized monomer were shown in Figure 3.

From Figure 3, the stretching vibration peak near 3025 cm⁻¹ is =C-H; the stretching vibration absorption peak near 2860-2960 cm⁻¹ is saturated C-H; the characteristic absorption peak near 1728.1 cm⁻¹ is C=O in the ester group; the characteristic absorption peak near 1625.5 cm⁻¹ is C=C; the characteristic absorption peak near 1310.8 cm⁻¹ is C-N; and the characteristic absorption peak near 1178.1 cm⁻¹ is

C-O in the ester group. The oscillation absorption peaks near 726.5 cm are (CH₂)_n, n > 2.

3.2. *Structure and Characterization of P(AM-DM-DMC12).* The infrared spectra of the synthesized polymer were shown in Figure 4, and the results of ¹H-NMR spectrum analysis of the synthesized copolymer were shown in Table 1.

From Figure 4, the characteristic absorption peak of NH₂ antisymmetric stretching vibration in -CONH₂ was found at 3351 cm⁻¹; the characteristic absorption peaks of -CH₂ and -CH were at 2926 cm⁻¹ and 2855 cm⁻¹, respectively. The characteristic absorption peak of amide -C=O appears at 1654 cm⁻¹, the bending vibration peak of methylene in the structure of -CH₂-N⁺(CH₃)₂(C₁₂H₂₅) was at 1456 cm⁻¹, the vibration peak of -CH₃ was found at 1419 cm⁻¹, the characteristic absorption peak of -CN was found at 1319 cm⁻¹, and the characteristic peak of long chain alkyl group was at 1189 cm⁻¹. Therefore, AM, DM, and DMC12 were all involved in the copolymerization, and the copolymer P(AM-DM-DMC12) was formed. In summary, FT-IR analysis showed that there were -CH₂-, C-N, N-H, -CH₃, and -C=O in the structure of the copolymer, while the characteristic peak of C=CH₂ disappears, indicating that the carbon-carbon double bonds (C=C) have been opened.

Therefore, the results of FT-IR and ¹H-NMR spectrum analysis showed that AM, DM, and DMC12 were all

TABLE 1: $^1\text{H-NMR}$ spectrum analysis of P(AM-DM-DMC12).

Structure unit	δ (ppm)	n (H)	Groups
AM	1.52	2H	$-\text{CH}_2-$
	2.19	1H	$-\text{CH}-$
DM	1.165	3H	$-\text{CH}_3$
	1.74	2H	$-\text{CH}_2-$
	3.274	9H	$-\text{N}^+(\text{CH}_3)_3$
	3.73	2H	$-\text{CH}_2-\text{N}^+$
	4.68	2H	$-\text{O}-\text{CH}_2-$
DMC12	0.9	3H	$-\text{CH}_3$
	1.27	20H	$-(\text{CH}_2)_{10}-$
	1.78	2H	$-\text{CH}_2-$
	3.56	6H	$-\text{N}^+(\text{CH}_3)_2$
	3.64	2H	$-\text{N}^+-\text{CH}_2-$
	4.21	2H	$-\text{CH}_2-\text{N}^+$
	4.68	2H	$-\text{O}-\text{CH}_2-$

involved in the copolymerization, and the copolymers synthesized accorded with the structure of the target product P(AM-DM-DMC12).

3.3. Effects of Synthesis Conditions on Polymers. The effect of monomer mass fraction on the molecular weight of the polymer is shown in Figure 5(a), the effect of initiator dosage is shown in Figure 5(b), and the effect of monomer reaction ratio on the viscosity and solubility of polymers is shown in Figure 5(c).

It can be seen from Figure 5(a) that the molecular weight of the polymer increases first and then decreases slightly with the increase of monomer mass fraction. When the initiation dose is fixed and the monomer concentration (monomer quantity) is relatively low, many free radicals generated by the initiator can quickly consume the monomer, so the molecular weight cannot be increased effectively. With the increase of the monomer concentration (monomer quantity), the increment of monomer is mainly used for the extension of molecular chain, which shows that the rate of polymerization is accelerated, and the molecular weight of the polymer is also gradually increased [5, 8]. The polymer with higher molecular weight can be synthesized theoretically with the increase of the total mass fraction of monomer. However, when the monomer mass fraction is too high, a lot of heat will be released from the reaction system, and the high viscosity of the system makes heat dissipation difficult, which will eventually cause the system temperature to be too high and accelerate the termination rate of the chain, resulting in a slight decrease in the molecular weight. Therefore, it is advisable to select the monomer mass fraction at 36%.

Figure 5(b) shows that the amount of initiator also plays an important role in the polymerization process, and the molecular weight of the polymer decreases gradually with the increase of the initiator dosage. The main reason is that the larger the amount of initiator added, the more free radicals will be generated, resulting in a faster reaction rate and easier explosion polymerization, which is not conducive to

the formation of high molecular weight polymers. However, if the amount of initiator is infinitely reduced, it will result in too little free radical generation, difficulty in chain initiation, and slow initiation rate, which will lead to the disadvantages of long reaction time, low conversion rate and residual monomer content, and poor solubility of polymer. Therefore, 0.06 wt% was the optimum dosage of initiator for the polymerization experiment.

Figure 5(c) shows that the intrinsic viscosity of copolymer increases first and then decreases with the increase of the molar ratio of AM, DM, and DMC12, while the dissolution time is just the opposite. When the molar ratio of AM, DM, and DMC12 is 4 : 1 : 1, the intrinsic viscosity of the polymer can reach $10.04 \text{ dL}\cdot\text{g}^{-1}$ and the dissolution time is 120 min. The addition of hydrophobic groups brings the unique properties of hydrophobically associating polymers to the synthesized polymers, which not only improves the viscosity of the polymers but also reduces the dissolution time. It can improve the adsorption capacity of flocculants to organic substances in sewage, reduce the hydrophilic capacity of flocs, and improve the strength and settling speed of flocs. Because the hydrophobic groups have strong lipophilicity, when encountering organic substances such as oil droplets in sewage, hydrophobic groups on polymer molecular chains will adsorb them and flocculate rapidly through bridging and neutralization of other chains [21]. In the process of flocculation growth, other particles in sewage are often adsorbed, captured, and wrapped. As a result, the flocculation effect is improved.

Under the optimized reaction conditions determined by experiments, the molecular weight of the obtained polymer P(AM-DM-DMC12) can reach 1.02×10^7 , which can meet the molecular weight requirements of flocculants.

3.4. Effect of PAC on Flocculation Property. Figure 6 shows that the flocculation effect of wastewater increases with the increase of PAC dosage. It is no longer explained in detail because the PAC flocculation method is mature and widely used in industry [22]. Considering the sewage treatment cost and standard, $70 \text{ mg}\cdot\text{L}^{-1}$ is the optimal dosage of PAC, the turbidity removal rate is 92.81%, and the residual turbidity is 15.7 NTU.

3.5. Effect of PAM on Flocculation Property. As shown in Figure 7(a), the turbidity and COD removal rate of water samples first increased and then decreased with the constant change of PAC & PAM dosage and reached the maximum when the dosage was $50 + 2 \text{ mg}\cdot\text{L}^{-1}$. The main reason is that when the dosage is too large, on the one hand, it may cause recontamination of water samples; on the other hand, too much flocculant will surround the colloidal particles and achieve equilibrium in water; thus, it is difficult to further remove the colloidal particles. When the dosage is too small, it will lead to poor effects such as compression of the double electron layer, adsorption, and rebridging, which will lead to difficulties in flocculant flocculation with colloidal particles to form alum, resulting in poor flocculation effect.

Figure 7(b) shows that flocculation effect is the best when the time of settling time is 2 h. When the time of

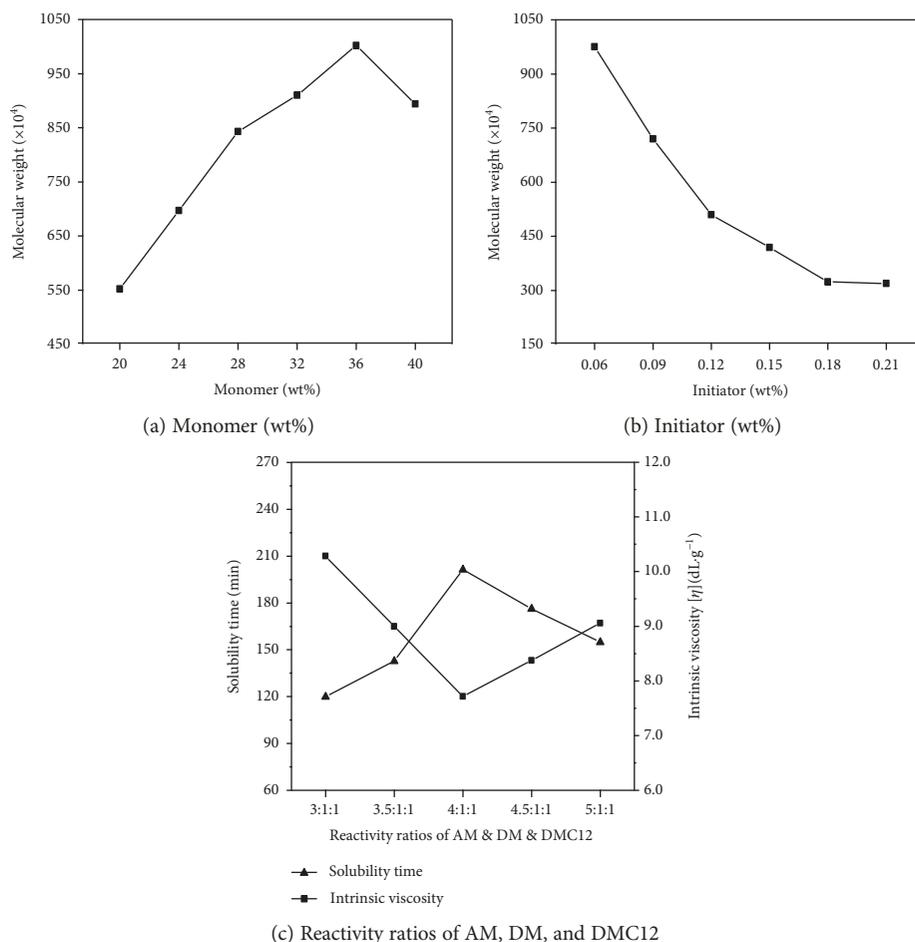


FIGURE 5: Effect of reaction conditions of P(AM-DM-DMC12).

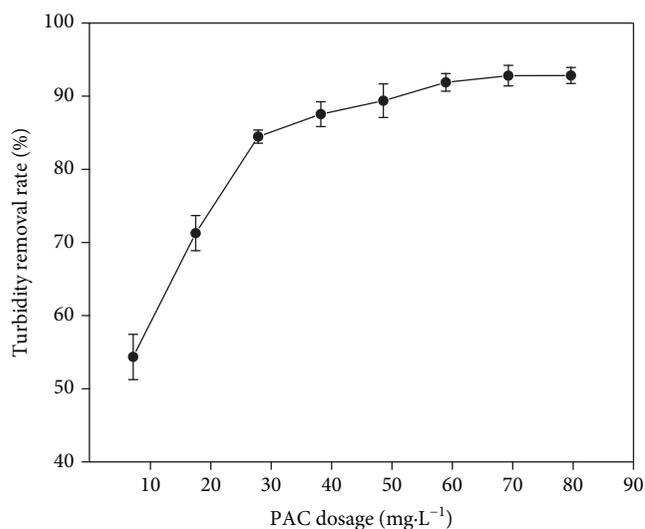


FIGURE 6: Effect of PAC on flocculation property.

settling time is too short, many alum flowers have not formed or have not completely settled, and when the time of static settling is too long, the settled alums will disperse and sus-

pend in water continuously, which leads to the increase of organic matter content.

Figure 7(c) shows that pH has a significant effect on the flocculation of the PAC & PAM system. The turbidity and COD removal rate of the water samples both increased obviously with the increase of pH, but when it was greater than 7.0, the effect of both began to decline gradually. When the pH is low, the flocculant mainly exists in the form of complex ions with a high charge and low degree of polymerization in the sewage, which cannot play the role of adhering and bridging. With the increase of pH, the hydrolysate of flocculant mainly exists in the form of a complex with high degree of polymerization, which can play a better role in complexing adsorption [23, 24]. However, when the pH is too high, the form of high charge body (e.g., Al^{3+}) will change and the flocculating effect will be weakened due to the increase of the concentration of OH^- . Therefore, the optimum pH on the flocculation of PAC & PAM is 7.0. At the optimal conditions of the PAC & PAM flocculation system, the turbidity removal rate is 93.84% and the residual turbidity is 13.4 NTU.

3.6. Effect of P(AM-DM-DMC12) on Flocculation Property. From Figures 8(a) and 8(b), the optimum dosage of PAC & P(AM-DM-DMC12) is $50 + 2 \text{ mg}\cdot\text{L}^{-1}$, the optimum settling

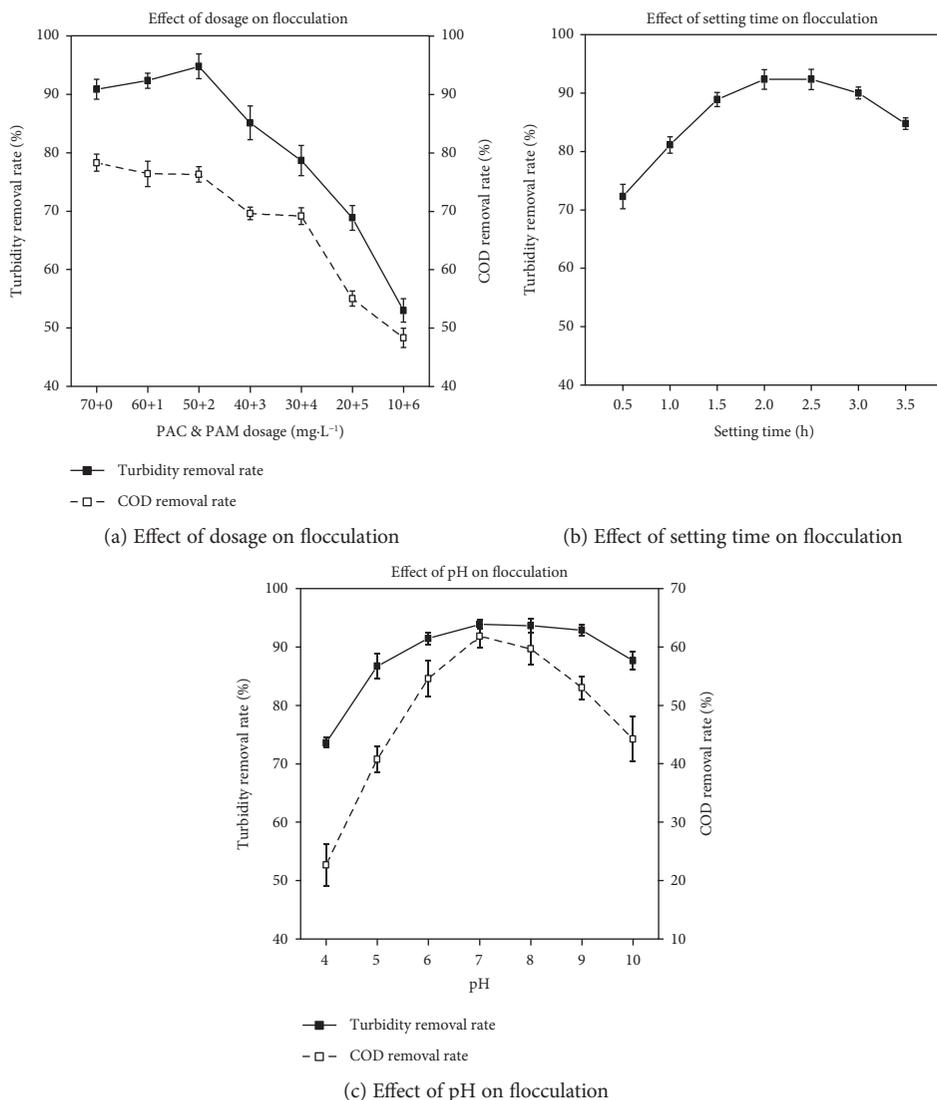


FIGURE 7: Effect of PAC & PAM on flocculation property.

time is 1.5 h, and the flocculation performance is significantly better than PAC & PAM. The main reason is that the molecular structure of P(AM-DM-DMC12) has a stable charge and carries a long chain of cationic and DM hydrophobic chains compared with PAM, which have rich structures and more functional groups. As P(AM-DM-DMC12) is added to sewage, the linear structure of large molecules in water constantly bend and stretch; once it contacts with sewage of colloid particles, the main link of the AM will adsorb and aggregate them, and the rest of the link is constantly stretching, more and more with the adsorption of colloid particles, and the flocs are formed bigger and bigger, namely, “bridging role” [25, 26]. In addition, when colloidal particles are negatively charged, cationic chains in macromolecules will neutralize them, namely, “electric neutralization.” As a result, P(AM-DM-DMC12) exhibited better flocculation performance under the synergism of these effects.

Figure 8(c) shows that PAC & P(AM-DM-DMC12) has better flocculation effect when the pH is between 5.0 and

9.0, which has a wider application range of pH compared with PAC & PAM. In the PAC & P(AM-DM-DMC12) flocculation system, PAC plays a major role in flocculation, while P(AM-DM-DMC12) acts as a coagulant aid. When the pH value is between 5.0 and 9.0, PAC is easy to hydrolyze and generates many high-polymer aluminum salts, which have strong flocculating ability. The electrostatic interaction in different structural units of P(AM-DM-DMC12) can improve the interaction between polymer molecules and further enhances the adsorption and bridging of the flocculation system. However, when the pH is less than 5, PAC mainly exists in the form of highly charged body (Al^{3+}) and low degree of polymerization in acidic conditions, which cannot play the role of adsorption bridge. The flocculation process is mainly completed by adsorption electric neutralization. When pH is greater than 9, with the increasing concentration of OH^- , a large amount of $Al(OH)_3$ will be produced by the hydrolysis of highly charged body (Al^{3+}); the wastewater is mainly treated by sedimentation net trapping [22, 23]. In addition,

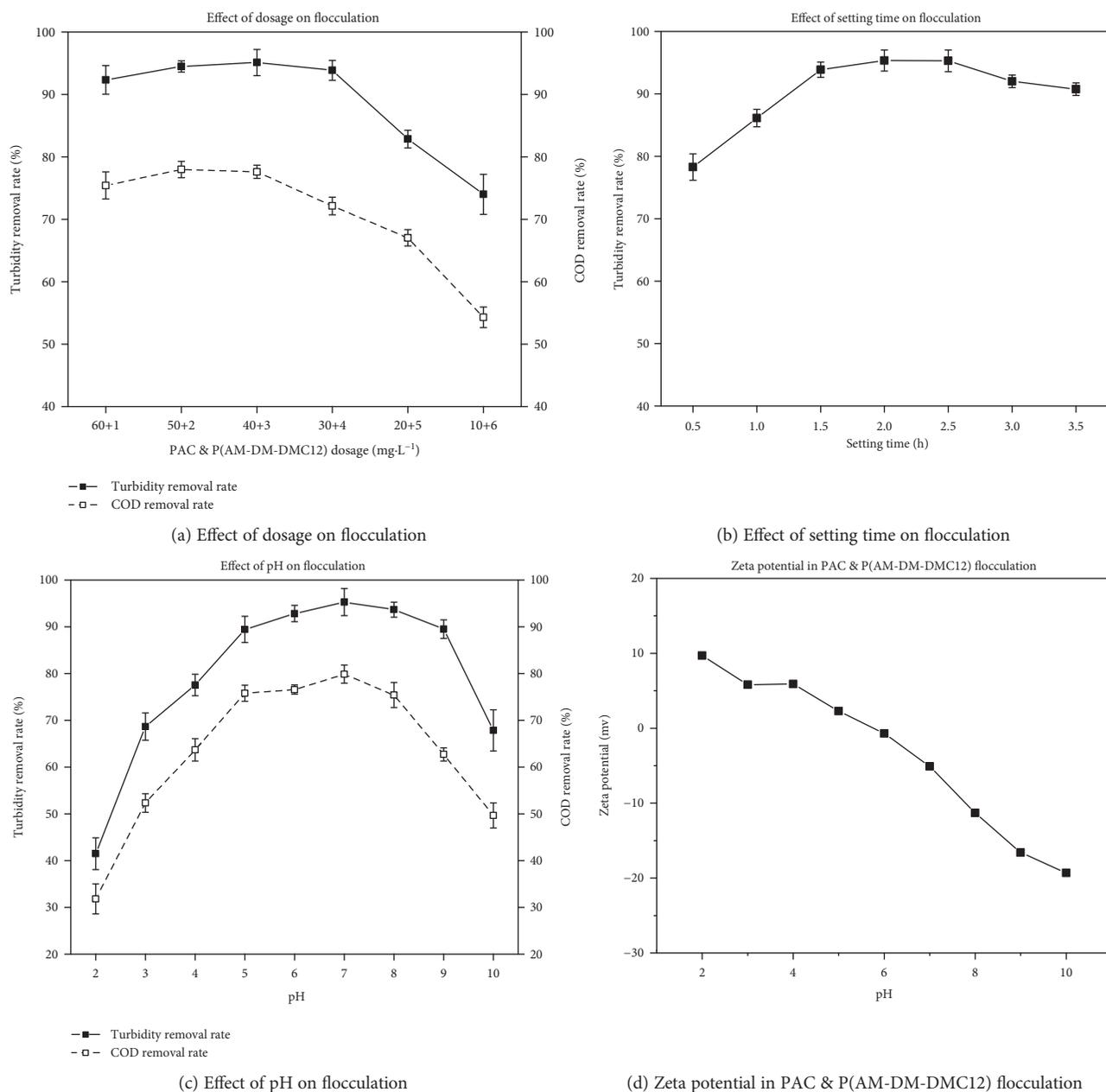


FIGURE 8: Effect of PAC & P(AM-DM-DMC12) on flocculation property.

when the pH value is too high or too low, the charged property of the whole flocculation will change dramatically, which will cause the polymer itself to curl up together, unable to effectively capture the colloidal particles in the water, and finally weaken the flocculation effect of PAC & P(AM-DM-DMC12). From Figure 8(d), the zeta potential of PAC & P(AM-DM-DMC12) is shown from positive to negative with the increase of pH, and pH_{pzc} is 5.8. When $pH < pH_{pzc}$, the surface of flocculant has a positive charge. On the contrary, when $pH > pH_{pzc}$, the surface of flocculant has a negative charge. It can be inferred that, in the flocculation process, there are both adsorption bridging effect and electrical neutralization effect. At the optimal conditions of the PAC &

P(AM-DM-DMC12) system, the turbidity removal rate is 95.27% and the residual turbidity is 10.3 NTU.

4. Conclusions

A new type of organic polymer modified hydrophobic monomer coagulant P(AM-DM-DMC12) was synthesized by water dispersion polymerization with AM, DM, and DMC12 as comonomers in the REDOX initiator system. The optimal synthesis conditions were as follows: total mass fraction of monomer is 35 wt%, molar ratio of AM, DM, and DMC12 is 4:1:1, total mass fraction of monomer is 35%, and dosage of initiator is 0.6 wt% ($(NH_4)_2S_2O_8$:NaHSO₃ is

1:1). Under the optimized reaction conditions, the molecular weight of P(AM-DM-DMC12) is 1.02×10^7 . Furthermore, P(AM-DM-DMC12) shows better flocculation performance in wastewater treatment and has lower dosage, wider pH range, and shorter flocculation time compared with PAM, which has better economic benefit and higher application value and prospect for improving the traditional flocculation process.

Data Availability

The data used to support the findings of this study have been deposited in the [Wei Guo] 286 repository (10.6084/m9.figshare.8044814.v1).

Conflicts of Interest

There are no conflicts of interest to declare.

Acknowledgments

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