



Photo-Initiated Fabrication of Zwitterionic Polyacrylamide with Enhanced Flocculation Activity†

CHUYANG XU^{1,*}, YANFEN WANG¹, RONGCHUN NIE², MENGTING LI¹ and BENXIA LI¹

¹School of Materials Science and Engineering, Anhui University of Science & Technology, Huainan 232001, Anhui Province, P.R. China

²School of Chemical Engineering, Anhui University of Science & Technology, Huainan 232001, Anhui Province, P.R. China

*Corresponding author: Fax: +86 554 6668540; Tel: +86 554 66348649; E-mail: rchnie@aust.edu.cn

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Zwitterionic polyacrylamide was synthesized by photo-initiated polymerization method. The structure characteristic of the AmpPAM was confirmed through intrinsic viscosity study and FTIR spectroscopy. Further, flocculation efficacy of the zwitterionic polyacrylamide was studied in coal slime water through jar test procedure. The improved flocculation activity indicated its potential application as superior flocculant compared with the commercial zwitterionic polyacrylamide and cationic polyacrylamide.

Keywords: Zwitterionic polyacrylamide, Flocculant, Photo-initiation, Coal slime water.

INTRODUCTION

Flocculation is an important industrial process for solid-liquid separation during the primary purification of wastewater. As one of the most extensive organic flocculants, polyacrylamide with unique physicochemical properties is extensively used to many wastewater treatment applications, such as electroplating wastewater, metallurgical wastewater, coal washing water, *etc.*¹⁻³.

So far, attention has been increasingly paid to zwitterionic polyacrylamide (AmpPAM). This polymer presents both anionic and cationic benefits that have a much wider pH range and smaller dosage, which shows prominent flocculation performance compared with those conventional flocculants^{4,5}. However, most of the existing synthesis involve the complicated process or low relative molecular mass. It remains a limitation in its further practical application. Herein, we report a facile photo-initiated polymerized method for the AmpPAM flocculant at low temperature. The effect of different experimental conditions on the synthesized grades of the AmpPAM were examined. The flocculation performance test showed the AmpPAM flocculant have an excellent flocculation activity for coal slime water.

EXPERIMENTAL

A specific amount of NaOH was dissolved in deionized water and added into acrylics to a neutral sodium acrylate

solution. Acrylamide (AM) and methacryloyloxyethyl trimethyl ammonium chloride (DMC) with a certain proportion were added dropwise into sodium acrylate solution at stirring condition, forming a reaction system with 25 g, followed by the dropwise addition of the photoinitiator into the bottle to initiate action. After 4 min of reaction under N₂, the resulting solution was sealed and exposed to sunlight for 24 h. After the reaction finished, the obtained samples were dried at room temperature. The final product was AmpPAM.

The coal fine used in the experiment obtained from Qianyingzi coal preparation plant. The intrinsic viscosity was measured by one point method according to GB 12005.1-89. The measurement was conducted under ambient conditions with a non-diluted Ubbelohde viscometer (d = 0.54 mm). The transmittance was recorded on JH-721-100 spectrophotometer.

RESULTS AND DISCUSSION

The intrinsic viscosity was evaluated for the synthesized grades of AmpPAM. Various grades of the copolymer were synthesized by varying the illumination time, photoinitiator dosage, temperature (T) and pH value. As shown in Fig. 1(a-d), similar clear trends were observed in the systems examined. Under the experimental conditions, the yield of intrinsic viscosity increased with these experimental parameters, approaching an asymptotic value and then unchanged or decreased. It indicates that these factors have an optimal range for intrinsic

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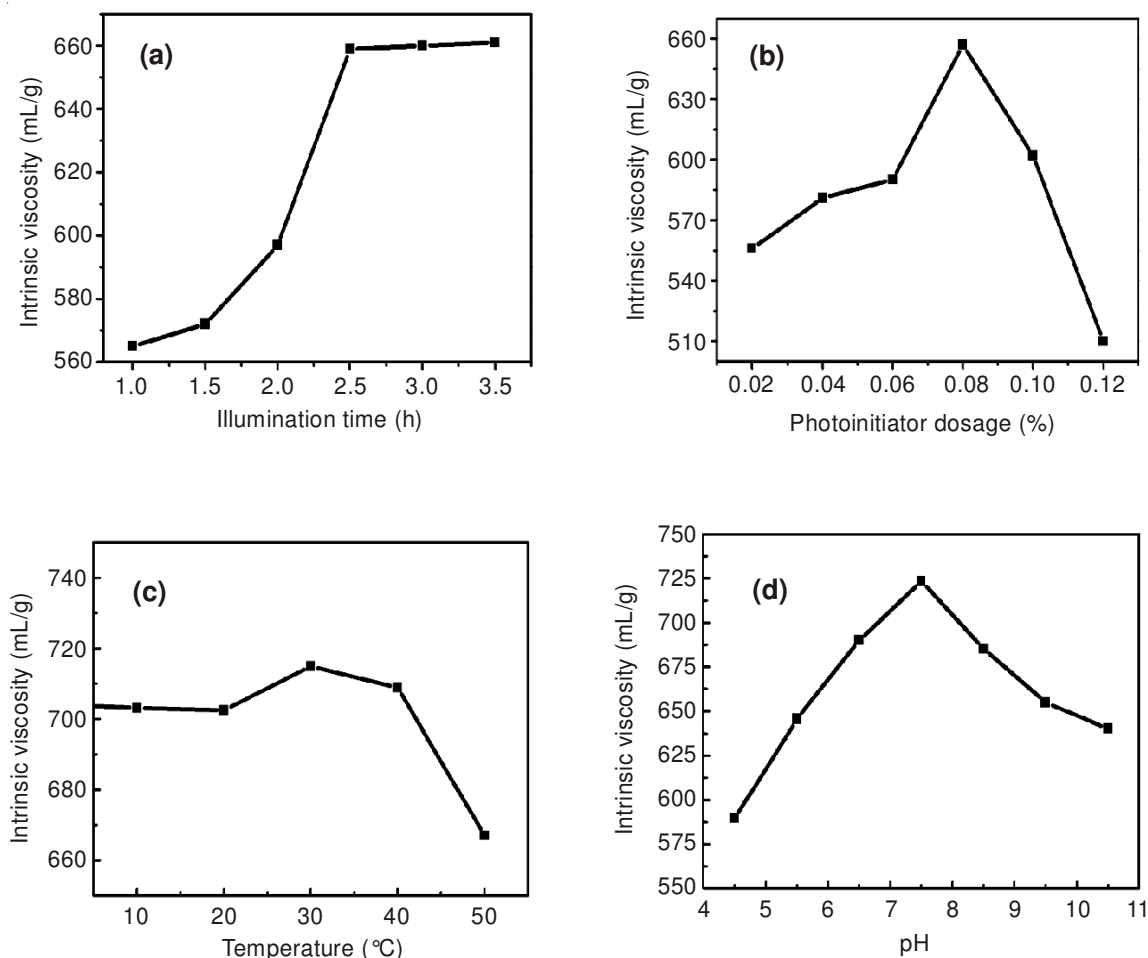


Fig. 1. Effects of experimental parameters on the intrinsic viscosity of AmPAM

viscosity. The intrinsic viscosity does not exceed the top value even at higher temperature, because the reactive temperature higher than 30 °C could cause AmPAM depolymerization and chain transfer.

In order to further optimize test, we design the orthogonal test of $L_9(3^4)$. Table-1 shows the results of the orthogonal test. The results showed that when the experimental conditions are 40 % monomer mass concentration, temperature of 20 °C, 20 μ L photoinitiator dosage and pH value of 6.5, the intrinsic viscosity of AmPAM is optimal. It indicated that it is possible

to obtain better flotation results by these preliminary results and the optimization program.

Infrared spectra of AmPAM is shown in Fig. 2. The stretching vibration peaks of C=O and N-H of AM were at 1637 and 3438 cm^{-1} , respectively. COO-vibration peak of DMC and sodium acrylate was at 1402 cm^{-1} . The stretching vibration peaks of $-\text{CH}_3$ could be observed at 2920 cm^{-1} . Moreover, the stretching vibration peaks of $-\text{CH}_2-\text{N}^+(\text{CH}_3)_3$ were at 1637 and 3438 cm^{-1} , respectively. All of these groups belonged to the characteristic absorption peaks of zwitterionic polyacrylamide.

TABLE-1
RESULT AND ANALYSIS OF THE ORTHOGONAL TEST

Run No.	Monomer mass concentration (%) (A)	Temperature (°C) (B)	Photo-initiator dosage (μ L) (C)	pH value (D)	Intrinsic viscosity (η)
1	1 (30)	1 (20)	1 (10)	1 (6.5)	572.25
2	1 (30)	2 (30)	2 (20)	2 (7.5)	505.35
3	1 (30)	3 (40)	3 (30)	3 (8.5)	473.70
4	2 (35)	1 (20)	2 (20)	3 (8.5)	666.66
5	2 (35)	2 (30)	3 (30)	1 (6.5)	495.32
6	2 (35)	3 (40)	1 (10)	2 (7.5)	575.94
7	3 (40)	1 (20)	3 (30)	2 (7.5)	634.17
8	3 (40)	2 (30)	1 (10)	3 (8.5)	621.11
9	3 (40)	3 (40)	2 (20)	1 (6.5)	775.31
K_1	1551.3	1873.08	1769.3	1842.88	T = 5319.81
K_2	1737.92	1621.78	1947.32	1715.46	
K_3	2030.59	1824.65	1603.19	1761.47	
R	479.29	251.3	344.13	127.42	

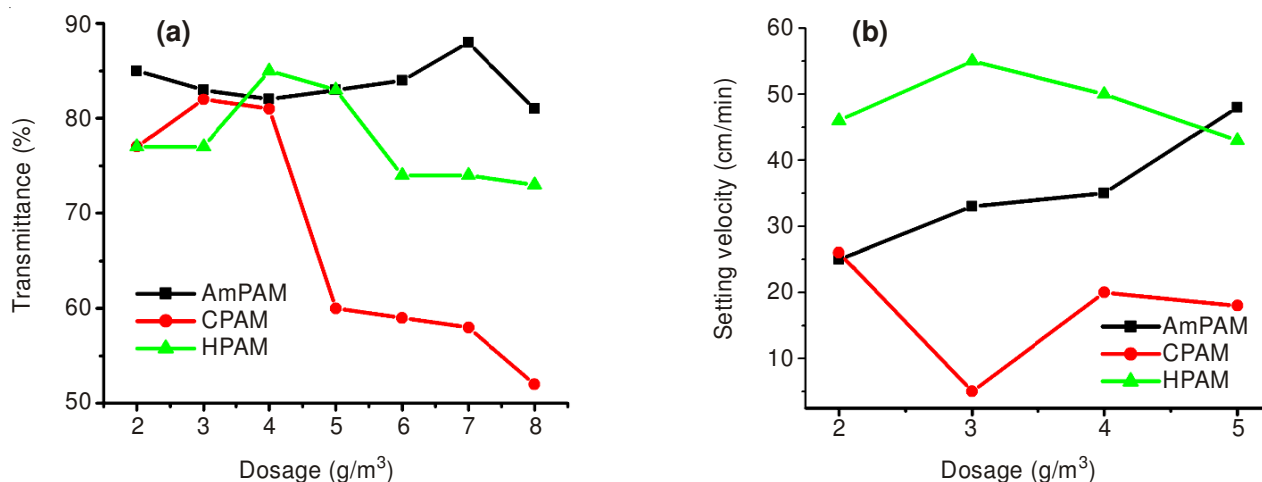


Fig. 3. Effects of different flocculants on settling velocity (a) and transmittance (b) for coal slime water

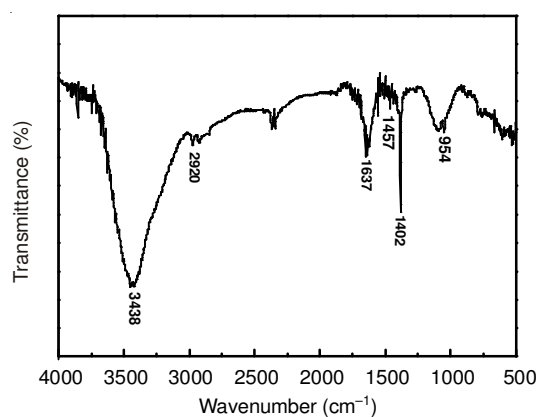


Fig. 2. FTIR spectra of AmPAM

It could be indicated that structure characteristic of the resulting AmPAM sample is in accordance with zwitterionic polyacrylamide.

Zwitterionic polyacrylamide with 500 million molecular weight, commercial zwitterionic polyacrylamide (CPAM) and cationic polyacrylamide (HPAM) were used for slime water flotation to evaluate flocculation activity. The settling velocity and transmittance are adopted to describe their flocculating abilities. In present work, the concentration of slime water was 80 g/L from Qianyingzi coal preparation plant. The molecular weight of AmPAM flocculants is calculated by the intrinsic viscosity. As shown in Fig. 3, the supernatant exhibits an enhanced transmittance compared with other flocculants (CPAM and HPAM). A faster settling velocity was also observed from coal slime water treated with AmPAM. It indicated that the synthesized AmPAM has an excellent flocculating sedimentation performance for coal slime water as a flocculant. Fig.4 shows the sedimentation efficacy of coal slime water before and after treatment with 500 million AmPAM.

Conclusion

A novel amphoteric chemically bonded AmPAM flocculant was designed and successfully prepared by the visible-light-initiated polymerized route. Using intrinsic viscosity as

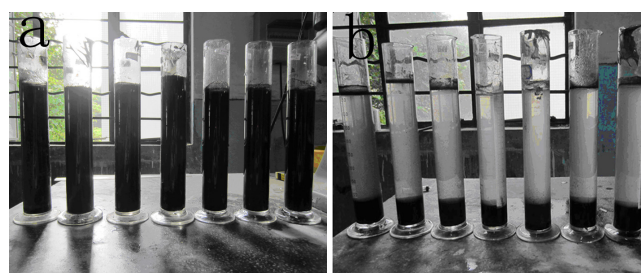


Fig. 4. Sedimentation efficacy of coal slime water before (a) and after (b) treated with 500 million AmPAM

performance index, the optimal experimental conditions were confirmed: 40 % monomer mass concentration, temperature of 20 °C, 20 μL photoinitiator dosage and pH value of 6.5. Flocculation tests at various dosage levels proved that compared with other flocculants (CPAM and HPAM), the designed AmPAM flocculant displays an enhanced flocculating effect for coal slime water.

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