

Study on synthesis and flocculation property of cation-polyacrylamide*

NIE Rong-chun(聂容春), GUO Li-ying(郭立颖), XU Chu-yang(徐初阳)
(Anhui University of Science and Technology, Huainan 232001, China)

Abstract On the basis of flocculating settling experimentation on flotation waste coal in Wangfenggang coal preparation plant, influence of medical dosage and cationization (CD) of CPAM samples on coal slurry's flocculating effect was studied, difference of flocculating effect on coal slurry among different categories of polyacrylamide was discussed. Experimental results show that when the dosage of flocculant reaches 2~4 g/m³ flotation waste, and the CD of CPAM is 5%, flocculating effect is the best, light transmittance of supernatant liquor reaches 93%. Taking 3types of sample CPAM, PAM and PHP, which formula weight vary a little, to deal with the same concn of coal slurry, when medicine dosage is 3 g/m³, flocculating effect of CPAM is the best, light transmittance of supernatant liquor reaches 92%.

Keywords cation-polyacrylamide, cationization, coal slurry, flocculant

Introduction

Polyacrylamide, which is a linear macromolecule polymer(CPAM), is a general designation of acrylamide homopolymer and copolymer obtained from acrylamide and other monomers. Based on constitution, it is classified to cationoid(CPAM), anionic (HPAM), non-ion (PAM), etc. CPAM is widely used to treat with industrial slush or city wastewater as a high efficient flocculant. Cationoid copolymer products are greatly valued for controllability and distributing uniformity of electric charge, as well as the simple manufacturing process^[1].

Dispose of coal slurry is an important production step to realize closed water cycle in coal preparation plant. Using flocculant intelligently is one of the main methods to clarify coal slurry. Cationoid organic macromolecule flocculant, which attaches cations at its big chain element, adsorbs negative electric charge min-

eral particles and other impurity particles strongly, besides, it has the power of charge neutralization and absorption bridging action in treating with waste water that dissolves large number of negative electric particles. Also, cationoid flocculant has the function of condensation. Add some opposite electric charge electrolyte to the system of sporadic colloid, suspended particles will dilate and settle down because of the electric charge collision that aroused by electric charge neutralization.

Although wide study has been carried on the synthetic method of CPAM, industrialization production is still at the very beginning. Therefore, it is of long term potential to develop CPAM industrially^[2]. Compare with traditional technique of thermal polymerization with redox system, CPAM that is synthesized by ultraviolet photo-initiation method, is of high quality, good water-solubility, low cost, property of pollution free, and is easy to realize industrialized produc-

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Tel: 86-551-2929353, E-mail: lyguo1981@163.com

tion. Acrylamide (AM) and dimethyl dially ammonium chloride (DMDAAC) are used as monomers to synthesize a new flocculant of Cation-polyacrylamide (CPAM) by ultraviolet photo-initiation method. Constitution of CPAM samples is identified by infrared (IR) spectrum. Effect of flocculating settling is discussed by experimenting on coal slurry of Wangfenggang coal preparation plant.

1 Experimental section

1.1 Experiment material

Acrylamide (AM), industrial product, content $\geq 98\%$, made in China medicine (Combine), Shanghai Company of chemical reagent; Dimethyl dially ammonium chloride (DMDAAC), content $\geq 60\%$, made in Yinhu chemical plant of Hangzhou; Photoinitiator, content $\geq 98\%$, made in laboratory; Sample of coal slurry, collected from Wangfenggang coal preparation plant, concn: 40 g/L.

1.2 Synthesis and characters examining

Photo-initiation method^[3,4] is taken to synthesize CPAM. Intrinsic viscosity number^[5] (η) (can be converted to molecular weight) and cationization (CD) are examined to characterize the resulting copolymer. Specificity of anion is characterized taking hydrolysis degree^[6] (HD).

1.3 Flocculating property examining

To simplify experiment, light transmittance of supernatant liquor (that is collected after flocculating settling) is used to reflect flocculating effect. The stronger transmittance is, the better effect for flocculant^[4] obtained. This is a single factor experiment, whose operation steps as follow. Put 500 mL of coal slurry sample into graduated cylinder; add quantitative pharmaceuticals, overturn graduated cylinder for 10 times bilaterally, then keep the graduated cylinder steady for 8 minutes, take some supernatant liquor into photometer with pipet, measure light transmittance (clarity) with photometer.

1.4 Structure identification

After getting specimen by coating process, molecular structure of HPAM and P (DMDAAC-AM) are analyzed using FTIR infra-red spectrometer. Results of infrared spectra are shown in Fig.1.

2 Results and discussion

2.1 Examination of flocculation property

2.1.1 Influence of medical dosage and cationization (CD) on flocculation

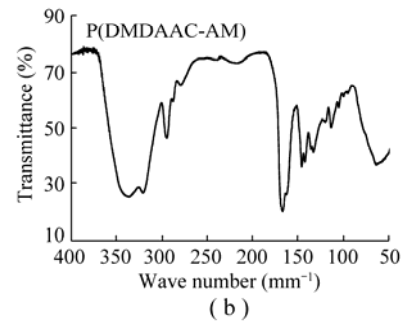
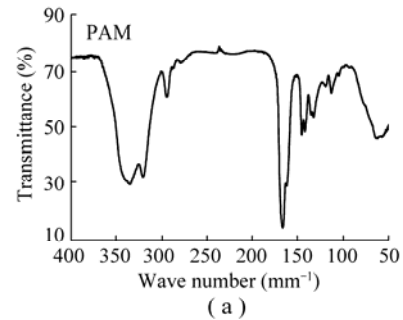


Fig.1 Infrared of PAM and P(DMDAAC-AM)

To study the influence of medical dosage and cationization (CD) on flocculation, 5 CPAM samples are taken to examine intrinsic viscosity number and cationization. The results are shown in Table 1.

Table 1 Measurement results of CPAM performance

No.	η (mL/g)	CD(%)
1	841	0
2	803	3
3	785	5
4	752	8
5	729	10

Treat coal slurry with the 5 samples gotten above, relationship between light transmittance of supernatant liquor and medical dosage cationization (CD) and is shown in Fig.2.

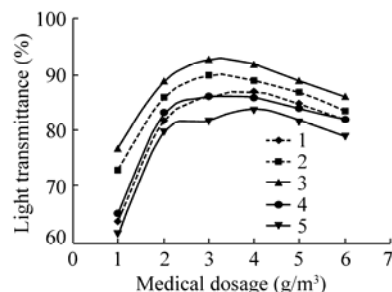


Fig.2 Influence of dosage of CPAM on transmittance

As shown in Fig.2, cationoid macromolecule flocculant is sensitive to medical dosage. Because

electrical neutralization of CPAM occupies the leading effects, there exists a best range for medical dosage. After negative charges in treating system are neutralized by cationoid flocculant, electrokinetic potential decreases. However, negative charges treating system changes to positive charges one when electrokinetic potential exceeds zero, that is, medicine dosage of flocculant is superfluous. Exclusion action makes flocs unit disperse again, so the effect of flocculating get worse. As shown in the curve, light transmittance increases along with the increment of dosage. When the dosage of flocculant reaches 2~4 g/m³, the volume of light transmittance reaches the maximum. If medical dosage increases continuously, the volume of light transmittance will decrease. That is coincident with theory of flocculation.

CD of CPAM affects flocculation greatly. Both light transmittance of supernatant liquor and flocculation are better if CD of CPAM is suitable. If CD of CPAM goes down, cation radical on the molecular chain diminishes, so not all the negative charges on surface of particle can be neutralized. In that case, particles are not easy to collide, so flocculating settling is not easy to generate. If CD of CPAM goes up, cation radical on the molecular chain increases, so after all the negative charges on surface of particle are neutralized, surplus cation radical remained. Reverse of electrical property makes repulsion between particles strengthen, which is adverse to flocculate. From experimental result, flocculating effect of No.3 sample of CPAM (the CD is 5%) is best of all.

2.1.2 Influence of different categories of PAM on flocculating effect

In order to identify the influence of different categories of PAM(non-ionic, anionic, cationic) on flocculation of coal slurry, taking 3 types of sample, whose formula weight varies a little and are examined. Results of character examination are shown in Table 2 (sample number: 1—PAM, 2—HPAM, 3—CPAM).

Table 2 Results of character examination

No.	η (mL/g)	HD(%)	CD(%)
1	829	/	/
2	843	30	/
3	785	/	5

Using 3 types of PAM mentioned above to treat with coal slurry. Relationship between medical dosage

and light transmittance of supernatant liquor is shown in Fig.3.

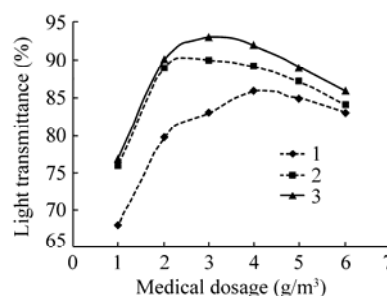


Fig.3 Influence of different flocculation on transmittance

As can be seen from Fig.3, flocculating effect of CPAM is best. Light transmittance of supernatant liquor reaches 92%, when medicine dosage is 3 g/m³. Flocculating effect of HPAM is in the middle, and flocculating effect of PAM is the worst. The reason is that surface of coal particles are occupied by negative charges, after adding CPAM, positive charge of CPAM neutralize negative charge of coal particles, electrokinetic potential decreased, repulsion weakened. That makes coal particles settle because collision happened. Also CPAM has the function of coalization, which makes flocculating effect better.

2.2 IR analysis

Structural spectrograms are shown in Fig.1. Infra-red spectrometer is used to analyze molecular structure of HPAM and P(DMDAAC-AM).

As shown in Fig.1(a), the peak value of vibrating absorption of amidogen(—NH₂) is 3 350 cm⁻¹, and methylene radical(—CH₂—) 2 943 cm⁻¹, and carbonyl of acylamino group 1 662 cm⁻¹.

As shown in Fig.1(b), the peak value of vibrating absorption of amidogen (—NH₂) is 3 349 cm⁻¹, and methylene radical(—CH₂—) 2 935 cm⁻¹, and acylamino group 1 661 cm⁻¹. 1 510~1 160 cm⁻¹ is peak range of δ vibrating absorption of double-methyl and methylene radical, which are connect with N⁺ in structural unit of DMDAAC, but there is no obvious difference in Fig.1.

In order to explain further, difference can be seen from quantitative analysis. 1 510~1 160 cm⁻¹ is peak range of δ vibrating absorption of double-methyl and methylene radical, which are connect with N⁺ in structural unit of DMDAAC. 2 955~2 825 cm⁻¹ is peak range of vibrating absorption of methylene radical (—CH₂—) of acrylamide (AM). After integrating area and calculating ratio for two wave numbers respec-

tively, result is shown in Table 3.

Table 3 Calculation of absorption value between homologous wave number area

No.	Peak range (cm ⁻¹)	Absorption value of PAM	Absorption value of CPAM
1	1 510~1 160	21.90	30.46
2	2 955~2 825	3.411	5.928
Radio(1/2)		6.420	5.138

From Table 3, calculating ratio of Fig.1(b) is minor than that of Fig.1(a), meaning that methylene radical(—CH₂—) of acrylamide (AM) in P (DMDAAC-AM) decreased. That is to say structural unit of DMDAAC entered polymeric unit. So P (DMDAAC-AM) is copolymer.

3 Conclusions

Conclusions are reached by experimenting on synthesis and flocculation property of cation-polyacrylamide (CPAM).

(1) CD affects flocculating effect of CPAM. When CD is 5% and medical dosage is 2~4 g/m³, flocculating effect of coal slurry is the best.

(2) Different types of polyacrylamide have different flocculating effect. Result in experiment indicates that, compared with HPAM and PAM, treating coal slurry with CPAM gets the best flocculating effect, when medical dosage is 3 g/m³.

(3) Structure analysis of CPAM with infrared spectrum testifies that CPAM is copolymer, meaning that DMDAAC and AM react into copolymer.

Flocculating effect of cation-polyacrylamide (CPAM) on coal slurry not only relates to CD and

medicine dosage, but also relates to concn, slickens content of coal slurry, etc. Because time and condition are limit, much work is remained to do in future.

References

- [1] 鞠耐霜, 曾文江. 阳离子型聚丙烯酰胺合成与絮凝特性研究[J]. 广州化工, 2000, 28(1): 65-68.
Ju Naishuang, Zeng Wenjiang. A study of synthesis and flocculant properties of cation-polyacrylamide[J]. Chemical Industry of Guangzhou, 2000, 28(1): 65-68.
- [2] 张元成, 刘树强, 高宝玉, 污水处理用阳离子 PAM 的开发现状和前景[J]. 工业水处理, 2002, 22(7): 15-17.
Zhang Yuancheng, Liu Shuqiang, Gao Baoyu. Development and prospects of cationic polyacrylamide for waste water treatment[J]. Industrial Water Treatment, 2002, 22(7): 15-17.
- [3] 徐初阳, 聂容春. 光引发合成聚丙烯酰胺的研究[J]. 安徽理工大学学报, 2003, 23(2): 49-52.
Xu Chuyang, Nie Rongchun. Study of photo-initiation for polyacrylamide synthesis[J]. Journal of Anhui University of Science and Technology, 2003, 23(2): 49-52.
- [4] 徐初阳, 聂容春, 张明旭, 等. 光引发合成聚丙烯酰胺在选煤中的应用[J]. 选煤技术, 2003, 23(2): 49-52.
Xu Chuyang, Nie Rongchun, Zhang Mingxu, et al. Application of polyacrylamide synthesized by photo-initiation in coal preparation plant [J]. Coal Preparation Technology, 2003, 5(5): 11-12.
- [5] 聚丙烯酰胺特性粘数测定方法[S]. GB12005. 1-89.
Measuring Method of Intrinsic Viscosity Number of Polyacrylamide[S]. GB12005. 1-89.
- [6] 聚丙烯酰胺水解度测定方法[S]. GB12005. 6-89.
Measuring Method of Hydrolysis Degree of Polyacrylamide[S]. GB12005. 6-89.