

Preparation of Nano-Modified Polyacrylamide and Its Application on Solid-Liquid Separation in Waste Drilling Mud

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Abstract

To satisfy the requirement on solid-liquid separation in high-density waste drilling mud, prepare the nano-modified polyacrylamide (PAM) flocculant for high density waste drilling mud by in-situ dispersion method, direct dispersion method and simultaneous formation method. The result showed the flocculent effect of nano-modified polyacrylamide prepared by simultaneous formation method was the best. When the content of water glass and acrylamide (AM) were respectively 3% and 15%, reaction temperature was 60°C and reaction time was 3 h, the performance of product was the best. The water content in filter cake was 24.32% after the waste drilling mud disposed by the optimization flocculant. The flocculent effect of optimization flocculant was superior to that of other flocculant in market.

Keywords: In-Situ Dispersion Method, Direct Dispersion Method, Simultaneous Formation Method, Nano-Modified Polyacrylamide, High Density Waste Drilling Mud

1. Introduction

The waste drilling fluids was the inevitable industrial waste while Oil and Gas exploration drilling. It had become one of the most severe pollution sources, whose effect on environment has been concerned gradually [1-3]. There were many methods [4-6] to dispose the waste drilling mud and drilling wastewater. The solid-liquid separation [7] was the most important and widely application method. At deep well drilling, the component of waste drilling mud was complexity, the density of waste drilling mud was higher and higher, and the waste drilling mud treatment was more and more hard [8]. The flocculant in market had not deal with the high density waste drilling mud. So a new nano-modified polyacrylamide flocculant for high density waste drilling mud was prepared.

2. Experiment

2.1. Agent

Cation polyacrylamide (GP), anion polyacrylamide (GP),

silane coupling agent KH570 (GP), homemade nano-SiO₂ (40 - 60 nm), water glass (modulus = 3.2~2.3), hydrochloric acid (AR), absolute ethyl alcohol (AR), acrylamide (AR), Potassium peroxydisulfate (AR), Sodium sulfite (AR), oxalic acid (AR), Nitrogen.

High density waste drilling mud, the density is 1.639 g/mL and the solid content is 56.72%.

2.2. Preparation of Nano-Modified Polyacrylamide

2.2.1. In-Situ Dispersion Method

(1) Preparation of nano-modified Cation polyacrylamide (CPAM)

Dissolve respectively 4.2 g Na₂SiO₃ in 10 ml deionized water and 8.0 g CPAM in 380 ml deionized water, then drop the Na₂SiO₃ solution into CPAM solution and mix equably. Make HCl to adjust the pH of above mixture to about 7 and keep the system react for 3 h at room temperature.

(2) Preparation of nano-modified anion polyacrylamide (APAM)

Dissolve respectively 4.2 g Na₂SiO₃ in 10 ml deio-

nized water and 8.0 g APAM in 380 ml deionized water, then drop the Na_2SiO_3 solution into APAM solution and mix equably. Make HCl to adjust the pH of above mixture to about 7 and keep the system react for 3 h at room temperature.

2.2.2. Direct Dispersion Method

Disperse 8.0 g homemade nano SiO_2 in 50 ml absolute ethyl alcohol, add 2 ml KH570 into above mixture, then keep stirring and reacting for 48h at room temperature. Drying above mixture in the end at 60°C to gain the nano SiO_2 modified by silane coupling agent. Disperse 4.0 g nano SiO_2 modified by silane coupling agent in 100 ml deionized water, add 5 g acrylamide into above mixture and removal of oxygen dissolved in the solution by blowing continuously nitrogen. Then add 0.1 g potassium persulfate to initiate polymerization and keep the system react for 4 h to gain product.

2.2.3. Simultaneous Formation Method

Dissolve 4 ml water class into 80 ml deionized water, adjust the pH of the water class solution to 3 - 4 by HCl and react for 1 h at room temperature. Dissolve 1 ml KH570 into 10 ml absolute ethyl alcohol, adjust the pH of the KH570 solution to 3 by oxalic acid and hydrolyze for 1 h at room temperature. Add KH570 solution into water class solution, then add 20 acrylamide into above mixture and removal of oxygen dissolved in the solution by blowing continuously nitrogen. Finally add 0.1 g potassium persulfate and 0.1 g sodium sulfite into above system to initiate polymerization. Keep the system react for 4 h to gain product.

2.2.4. Flocculent Treatment

Add 4ml MgCl_2 solution (0.2 g/ml) as gel breaker to 200 g waste drilling mud (1:1-fold diluted with water), then add some flocculant to above system and stir, centrifugal separation (4000 r/min) for 10 min. Weight the quality of filter cake (m1) and soil phase after baking (m2). Gain the water content in filter cake by the formula as follows:

$$\text{The water content in filter cake} = \frac{(m1 - m2)}{m1}.$$

3. Result and Discussion

3.1. Flocculent Effect of Three Kinds of Preparation Method

(1) Nano-modified CPAM flocculant prepared by in-situ dispersion method

(2) nano-modified APAM flocculant prepared by in-situ dispersion method

(3) nano-modified PAM flocculant prepared by direct dispersion method

(4) nano-modified PAM flocculant prepared by simul-

taneous formation method

From **Tables 1-4**, we can know that flocculent effect of nano-modified polyacrylamide prepared by simultaneous formation method was the best Among three preparation methods. The optimum conditions for preparing nano-modified polyacrylamide prepared by simultaneous formation method was obtained through orthogonal experiments.

3.2. Optimization of the Simultaneous Formation Method

Content of water class and acrylamide, react temperature and react time were selected and tested by four-factors-three-level orthogonal processing test. The test result is shown in **Table 5**.

From **Table 5**, we can know the test number 2 was the best.

3.3. Analysis of FTIR

Figure 1 was The FTIR spectrum of the sample (test number 2 in **Table 5**). As can be seen from the **Figure 1**, the peak of IR spectra of N-H stretching vibration appeared at 3419 cm^{-1} and 3197 cm^{-1} ; the peak of IR spectra of $-\text{CH}_2-$ asymmetry stretching vibration and $-\text{CH}_2-$ symmetric stretching vibration appeared respectively at 2922 cm^{-1} and 2850 cm^{-1} ; the absorption peak appeared at 1652 cm^{-1} and 1417 cm^{-1} was caused respectively by the stretching vibration of C=O in acylamide group and vibration of saturated C-H. The absorption peak of Si-O-C and Si-O-Si appeared at 1120 cm^{-1} .

Table 1. The effect of Content of nano-modified CPAM on the water content in filter cake.

Content of flocculant (%)	0.2	0.4	0.6	0.8
The water content in filter cake (%)	51.05	52.30	46.04	47.62

Table 2. The effect of Content of nano-modified APAM on the water content in filter cake.

Content of flocculant (%)	0.2	0.4	0.6	0.8
The water content in filter cake (%)	56.37	54.47	48.76	50.07

Table 3. The effect of Content of flocculant on the water content in filter cake.

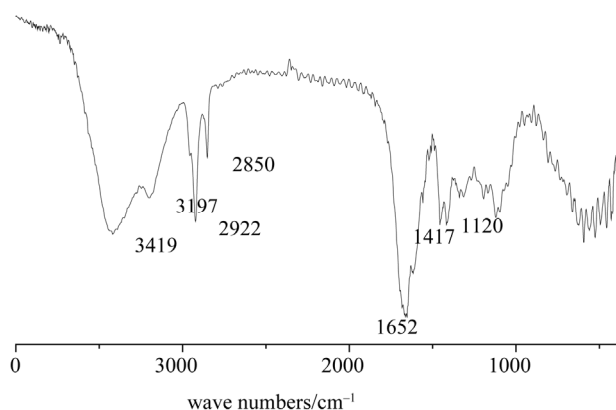
Content of flocculant (%)	0.2	0.4	0.6	0.8
The water content in filter cake (%)	35.98	35.24	34.87	35.13

Table 4. The effect of Content of flocculant on the water content in filter cake.

Content of flocculant (%)	0.2	0.4	0.6	0.8
The water content in filter cake (%)	33.01	32.55	30.68	31.73

Table 5. The test result of orthogonal experiment.

Test number	Content of water class/%	Content of acrylamide/%	React temperature/°C	React time/h	the water content in filter cake/%
1	3	10	50	2	30.23
2	3	15	60	3	24.32
3	3	20	70	4	25.98
4	5	10	60	4	26.67
5	5	15	70	2	25.49
6	5	20	50	3	26.35
7	7	10	70	3	26.22
8	7	15	50	4	28.38
9	7	20	60	2	29.89

**Figure 1. The FTIR spectrum of the nano-modified PAM flocculant.**

So, we can think that there must have some structural units of AM and SiO₂ in the nano-modified flocculant.

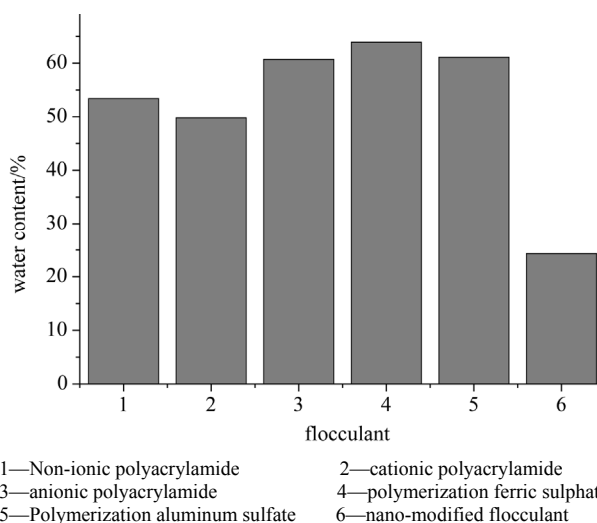
3.4. Contrast of Flocculent Effect

We contrast the flocculent effect of nano-modified flocculant prepared by simultaneous formation method with that of the other coagulant in market, such as, Non-ionic polyacrylamide, cationic polyacrylamide, anionic polyacrylamide, polymerization ferric sulphate, Polymerization aluminum sulfate, nano-modified flocculant.

From **Figure 2** we can see that adding the same amount of flocculant, the flocculent effect of organic flocculants was better than that of inorganic coagulant, but the results were not satisfactory, the water content in filter cake $\geq 50\%$. The flocculent effect of the nano-modified flocculant is significantly better than that of the other coagulant in market, water content of waste drilling mud after treated is 24.32%.

4. Conclusions

Water class and acrylamide used as raw material and potassium persulfate as initiator to prepare nano-

**Figure 2. Flocculent effect of some flocculant.**

1—Non-ionic polyacrylamide 2—cationic polyacrylamide
3—anionic polyacrylamide 4—polymerization ferric sulphate
5—Polymerization aluminum sulfate 6—nano-modified flocculant

modified PAM flocculant by simultaneous formation method. The optimum conditions were obtained through orthogonal experiments. When making the nano-modified PAM flocculant prepared under the optimum conditions to treat the high-density waste drilling mud, the water content in filter cake is 24.32% which was significantly better than that of the other coagulant in market.

5. Acknowledgements

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6. References

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