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Study on Preparation of Fluid Loss Reducer for High Temperature and High Salinity Water-based Drilling Fluid

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Abstract

In the ultra-deep well drilling project, the bottom hole temperature exceeds 200℃, the formation pressure is high, the bottom hole situation is complex, and drilling is difficult, which puts forward higher requirements for drilling fluid and drilling fluid additives. As one of the most important additives for water-based drilling fluid, fluid loss additive plays an important role in ensuring safe, fast and efficient drilling. Firstly, according to the performance requirements of high temperature and high salt resistance fluid loss additive for drilling fluid, AM/AMPS/SSS/NVP quadripolymer water-based fluid loss additive was synthesized by copolymerization for the first time. Performance evaluation shows that the fluid loss reducer can be used in 200℃/220℃ saturated brine drilling fluid, and can keep good rheology and low fluid loss.

Keywords

High Temperature and High Salt Resistance; Water-based Drilling; Fluid Loss Reducer; Prepare.

1. Introduction

In the process of drilling, because the formation temperature is getting higher and higher with the increase of drilling depth, in the high temperature and high pressure environment of deep well drilling formation, the chemical additives in wellbore drilling fluid will undergo high temperature destruction such as high temperature degradation, high temperature crosslinking, high temperature desorption and high temperature dehydration, which will make the drilling fluid performance worse or even worse, and cannot meet the requirements of drilling engineering [1]. Therefore, the anti-high temperature additives in drilling fluid, especially the core additives-anti-high temperature fluid loss additives, have become the "bottleneck" that restricts the development of anti-high temperature drilling fluid technology in deep wells. Developing new fluid loss additives and breaking the "bottleneck" of this technology is an important subject that China's deep well drilling fluid technology is facing urgently.

The effect of fluid loss additive on drilling fluid is self-evident: forming a thin and tough filter cake to stabilize the borehole wall and reduce the fluid loss of drilling fluid to protect oil and gas reservoirs from pollution. However, with the development of drilling engineering, the formation conditions for drilling fluid application are more severe, and the disadvantages of the existing fluid loss additives are also obvious. Therefore, more and more researchers are studying the fluid loss additives against high-temperature and salt-resistant drilling fluids [2-3]. Literature [4] vinyl alcohol sodium acrylate copolymer was prepared by copolymerization with vinyl acetate as raw materials. Compared with 4% saturated brine drilling fluid without polymer, the filtration loss of 4% saturated brine drilling fluid containing 2% polymerization product is reduced by more than 90% and the filtration reduction performance is remarkable [5]. Reference [6] a fluid loss reducer pkc-3 was prepared from

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polyacrylonitrile hydrolysate through a series of reactions. The filtration loss of saturated brine drilling fluid at 100 ℃ is controlled to 14.4ml. Its maximum application temperature in compound brine drilling fluid is 180 ℃, with strong resistance to salt and calcium pollution [7-8]. Literature [9] Using AMPS and others as synthetic monomers, a multi-polymer fluid loss additive with high temperature resistance and salt resistance was prepared by reaction. After adding this polymer fluid loss additive to bentonite slurry with 3% content, it was hot rolled at 210℃ for 16h, and the fluid loss was low. The corresponding sodium salt and calcium salt were added, and the experimental results showed that the concentration of salt resistance was saturated. The maximum concentration of anti-CaCl2 is 5% [10].

Based on polymer chemistry and high temperature and high salt environment, a new type of fluid loss reducer for water-based drilling fluid was developed in this paper. Using the optimized synthetic monomer materials, the initiation system and polymerization method were determined by further experimental study. At the same time, the influence of various key factors in the synthesis experiment on the polymer properties was studied, and finally the best material ratio and the best reaction conditions were determined.

2. Key technology analysis of high temperature and high density water-based drilling fluid

The temperature and pressure in deep and ultra-deep wells are relatively high. For water-based drilling fluid, because of the low compressibility of water, pressure has little influence on the performance of drilling fluid, while temperature has great influence on the performance of drilling fluid system, and the higher the temperature, the more serious the water-based drilling fluid is affected by temperature. Under high temperature, various components in drilling fluid will undergo degradation, thickening, fermentation, failure and other changes due to high temperature, resulting in dramatic changes in the performance of drilling fluid, which is difficult to adjust and control.

The function of anti-high temperature treatment agent is to ensure that drilling fluid has good thermal stability and good performance under high temperature and high pressure. In essence, this effect is still achieved by absorbing hydration, increasing the thickness of hydration film on the surface of particles and increasing the ξ potential of particles.

The obvious thickening and gelation of drilling fluid at high temperature and after high temperature are closely related to the hydration of clay particles at high temperature. Therefore, it is required that the anti-high temperature viscosity reducer can not only effectively inhibit the high temperature dispersion of clay, but also adsorb on the end face of clay, break up or prevent the formation of network structure, and play a role in viscosity reduction. An effective way to solve this problem is to complex high valence cations with viscosity reducer to form complexes, such as FCLS.

It can be seen from the formula (1) of drilling fluid filtration that the main factors affecting the filtration of drilling fluid are filter cake quality and filtrate viscosity.

$$V_f = A\sqrt{\frac{K\left(\frac{C_c}{C_m}-1\right)\Delta p t\times 10}{\mu}} \tag{1}$$

In the formula, V_f——The fluid loss of drilling fluid, ml;

A——Filter area, cm^2;

K——Permeability of filter cake;

C_c——Percentage content of solid particles in filter cake;

C_m——Percentage content of solid particles in mud;

Δp——Differential pressure, MPa;

t——The filtration time, min;

μ——Viscosity drilling fluid, mPa·s.

It can be seen from the above formula that the water loss is proportional to the filtration area, filter cake permeability, filtration time and the quadratic root of pressure difference. It is inversely proportional to the square root of mud viscosity. Therefore, there are usually two ways to control fluid loss, one is to increase the viscosity of liquid phase, the other is to improve the quality of mud cake and reduce the permeability of mud cake. For deep wells, the viscosity of liquid phase is reduced due to high temperature, and the effect of fluid loss reduction by increasing the viscosity of filtrate is not significant, but it is of more practical significance to improve the quality and compressibility of mud cake.

3. Preparation of filtrate reducer for high temperature and high salt water-based drilling fluid

3.1 Main raw materials and instruments

Main raw materials: AM, AMPS, NVP, NIPAM, NaOH, NaCl and are all industrial products.

Main instruments: constant temperature water bath pot, mechanical stirrer, five-axis high-speed stirrer, ZNS type fluid loss meter, ZNN-D6S type rotary viscometer, high temperature roller oven, OFI171-01-C type high temperature and high pressure filter loss meter, Fourier transform infrared spectrometer, thermogravimetric analyzer (TGA) and differential scanning calorimeter (DSC).

3.2 Reaction flow

The copolymer was synthesized by free radical solution polymerization with deionized water as solvent and ammonium persulfate as initiator. Specific synthetic experimental steps are as follows:

(1) Dissolving a certain amount of acrylamide, 2- acrylamido -2- methylpropanesulfonic acid, sodium p-styrene sulfonate and maleic anhydride monomer in deionized water;

(2) Adding a proper amount of sodium hydroxide aqueous solution to adjust the pH value to be neutral;

(3) Use a glass rod to stir the mixed solution so that the monomer is uniformly dispersed and completely dissolved, then transfer the monomer solution into a three-necked flask, fix the three-necked flask at the same time, connect the heating jacket, stirrer and thermometer, and adjust the heating jacket to slowly raise the temperature of the three-necked flask;

(4) When that set reaction temperature is reach, an initiator is added into the reaction solution, stir and heating are continued, the reaction time is timed for 2h, and the polymer solution is pour out after the reaction is finished;

(5) Measure the viscosity of the polymer solution at 50℃, pour the polymer solution onto a tray and put it into an oven, set the temperature at 35℃, and then dry it and crush it; Or directly spraying the polymer solution through a spray drier to form powder.

3.3 Optimization of optimum synthesis conditions

The effect of copolymer fluid loss additive is closely related to the synthesis conditions. The main experimental conditions affecting the copolymer fluid loss additive are: pH value of polymerization system, type and amount of initiator, monomer concentration, monomer ratio, reaction temperature and reaction time. As the main content of the research is the fluid loss reducer for drilling fluid with high temperature and high salt resistance, this paper takes the intrinsic viscosity of polymer products and API fluid loss of British evaluation soil drilling fluid prepared with compound brine as evaluation criteria, and determines the best synthetic conditions of AM/AMPS/SSS/NVP quadripolymer by optimizing various experimental conditions.

3.4 Rating of merit

In the process of product synthesis, the factors such as the starting time of cold water, the interval of adding water, the reaction temperature and the ratio of monomers were taken into account, and a series of products were synthesized. The product performance was evaluated. When evaluating the performance of synthetic products, the high-temperature and high-pressure filtration loss and mud cake thickness are taken as the main performance indexes of the products, and the influence on the medium pressure filtration loss and viscosity is also referred.

4. Results and discussion

4.1 Copolymer characterization

4.1.1 Infrared spectroscopic analysis

The principle of infrared spectroscopy is to analyze the group structure according to the phenomenon that the molecular group of matter absorbs the radiation energy of infrared light with the same frequency and then transitions to a higher energy level. Infrared analysis is a common method in chemical product analysis.

The synthesized AM/AMPS/SSS/NVP sample was mixed with KBr for tabletting, and was tested by Fourier transform infrared spectrometer. The obtained infrared spectrogram is shown in Figure 1.

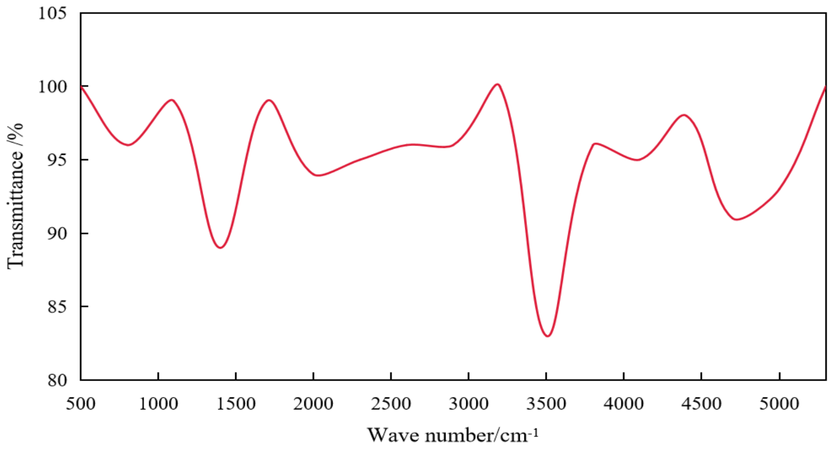

Figure 1. Ir spectrogram of AM/AMPS/SSS/NVP

The spectrum in Figure 1 shows that there are characteristic absorption peaks of hydroxyl groups near wave numbers 3 530 $cm^{-1}$and 3205cm^{-1}. There are characteristic absorption peaks of amido group near wave number 1 650 $cm^{-1}$and 1 552cm^{-1}, and characteristic absorption peaks of (methylene) group near wave number 2 901cm^{-1} and 766cm^{-1}. There are characteristic absorption peaks of sulfonic acid groups near 1 180cm^{-1} and 1 015cm^{-1}. There are characteristic absorption peaks of pyrrolidone ring near the wave numbers of 1 450cm^{-1} and 1 380cm^{-1}.

4.1.2 Molecular weight analysis

In the research of polymers, the relative molecular mass is an indispensable and important data. Because it not only reflects the size of polymer molecules, but also directly relates to the physical and chemical properties of polymers. There are several methods to determine the molecular weight of polymers: end group analysis, boiling point rising method, freezing point lowering method, gas phase osmotic pressure method, photorefractive method, membrane osmotic pressure method, ultracentrifugal sedimentation velocity method, ultracentrifugal sedimentation equilibrium method, viscosity method and gel chromatography.

Among them, viscosity method is one of the commonly used methods, which is simple in equipment, convenient to operate and has good experimental accuracy.

When the solvent, solution temperature and polymer molecular structure are the same, the intrinsic viscosity is only related to the molecular weight of polymer. The relationship between them can be determined by Mark-Houwink equation:

$$[\eta] = KM^{a} \tag{2}$$

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In the above formula, η is intrinsic viscosity in mL/g, M is polymer viscosity-average molecular weight, and K, a is empirical constant. After querying K, a value, the viscosity-average molecular weight can be calculated according to the measured intrinsic viscosity value.

In this paper, the viscosity-average molecular weight of AM/AMPS/SSS/NVP quadripolymer was measured by using ubbelohde viscometer. Test results are shown in Table 1.

Table 1. Measurement of molecular weight of AM/AMPS/SSS/NVP quadripolymer

Measured parameters	Detail
K	6.32×10^{-3} ml/g
a	0.83
$[\eta]$	96.14ml/g
M	1.66×10^5

Because polymers are generally a mixture of homologues with different molecular weights, their molecular weights have a certain distribution. The distribution of polymer molecular weight has great influence on the performance of polymer, so it is very important to study the distribution of polymer molecular weight.

4.1.3 Hermogravimetric analysis

Thermogravimetric analysis analyzes the thermal stability of polymers by measuring the mass of samples at different temperatures during the heating process. Rigaku TG8120 thermogravimetric analyzer was used in the experiment. The temperature range of the instrument is room temperature ~600℃. During the test, the protective gas N_2 is introduced, and the heating rate is set at 10℃ per minute to measure the thermal stability of the sample.

Through the thermogravimetric analysis curve of the polymer, the thermal decomposition of each functional group of the polymer at different temperatures can be analyzed, and the overall high temperature resistance of the polymer can be evaluated according to their thermal decomposition temperature. Figure 2 is the analytical curve of synthetic polymer.

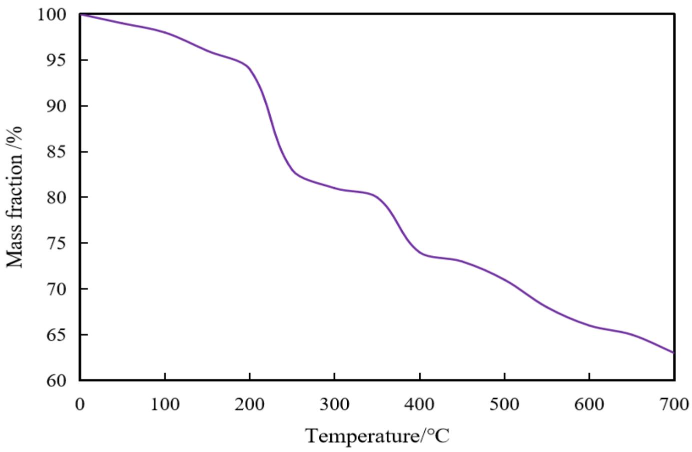

Figure 2. Synthetic polymer analysis curve

The fluid loss additive has good thermal stability at room temperature 0~270℃, and the loss rate of thermogravimetry decreases slowly. Between 270℃ and 330℃, the thermogravimetric curve decreased obviously, and the mass fraction decreased to about 75%, which may be caused by the thermal cracking of hydroxyl groups in the fluid loss additive. At 330~350℃, the thermogravimetric curve decreased slightly, which may be caused by the decomposition of carboxyl and amide groups in the fluid loss additive. At 350~650℃, the thermogravimetric curve decreased and leveled off, and

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finally the mass fraction stabilized at about 70%, which may be caused by the thermal cracking of carbon bonds in the main chain and benzene rings. Therefore, the fluid loss additive has strong thermal stability and can be applied in drilling fluid system at 220℃.

4.2 Fluid loss reduction performance

The synthesized fluid loss additives were divided into three groups for experiments, numbered as follows:

The molar ratio of the first group is:

mol(AM):mol(AMPS):mol(SSS):mol(NVP)= 2:2:3:1;

The molar ratio of the second group is:

mol(AM):mol(AMPS):mol(SSS):mol(NVP)= 1:2:4:1;

The molar ratio of the third group is:

mol(AM):mol(AMPS):mol(SSS):mol(NVP)=2:2:3:1;

The experimental conditions are as follows: the concentration of saturated brine drilling fluid is 30%, the amount of synthetic fluid loss additive is 3% of the solvent mass, and proper n-octanol defoamer. The experimental hot rolling temperature of each sample is 180℃, 200℃ and 220℃, and the hot rolling time of each group is 16 hours. The six-speed viscosity and high-temperature and high-pressure fluid loss after hot rolling are measured. The experimental results of each group are shown in the following tables 2, 3 and 4:

Table 2. Group 1 performance test of fluid loss additive

Hot rolling temperature/℃	Apparent viscosity/mPa·s	Plastic viscosity/mPa·s	Yield point /Pa	Filtrate losss /mL
180℃	40	32	7	8.6
200℃	42	35	4	9.8
220℃	46	43	3	11.3

Table 3. Group 2 performance test of fluid loss additive.

Hot rolling temperature/℃	Apparent viscosity/mPa·s	Plastic viscosity/mPa·s	Yield point /Pa	Filtrate losss /mL
180℃	36	28	6	9.3
200℃	38	33	5	10.5
220℃	40.9	38	4.4	12.7

Table 4. Group 3 performance test of fluid loss additive

Hot rolling temperature/℃	Apparent viscosity/mPa·s	Plastic viscosity/mPa·s	Yield point /Pa	Filtrate losss /mL
180℃	44.1	34	10.6	9.6
200℃	48.6	39	9.8	10.3
220℃	51.7	43	8.4	12.2

From the data obtained in the above three tables, it can be seen that the rheological parameters and fluid loss of synthetic fluid loss additives with different monomer ratios in saturated brine drilling fluid change. At the experimental temperature of each group, the polymer of the first group, i.e., the monomer molar ratio of 2:2:3:1, has the best overall fluid loss reduction performance. The filtration loss in saturated brine drilling fluid after hot rolling at 220℃ for 16 hours is only 11.3mL. This shows that the fluid loss additive is an efficient fluid loss additive.

4.3 Salt resistance

When designing AM/AMPS/SSS/NVP quadripolymer fluid loss additive, the design goal is that the copolymer not only has high temperature fluid loss resistance, but also has good salt resistance. In this section, the resistance to high temperature and high salt of AM/AMPS/SSS/NVP quadripolymer filtrate reducer is tested through indoor evaluation experiment.

The specific steps of the evaluation experiment are as follows: 2.0% AM/AMPS/SSS/NVP quadripolymer fluid loss additive is added to 400m1 drilling fluid base slurry (bentonite mass fraction is 4.0%). After the copolymer fluid loss reducer is completely dissolved, add a certain amount of salt or calcium to test the rheology and API fluid loss of drilling fluid.

After the test, the drilling fluid is aged at 180℃ for 16 hours at high temperature, and the rheological properties, API filtration loss and high temperature and high pressure filtration loss of the aged drilling fluid are tested. The experimental results are shown in Table 5.

Table 5. Influence of rheological property and filtration property of quadripolymer brine-based pulp

NaC concentration /wt%	Experiment condition	AV/mPa·s	PV/mPa·s	YP/Pa	API/m1	HTHP/ml
5	Hot rolling front	27.3	20.1	6.6	5.1	-
	After hot rolling.	18.9	16.2	3	6.2	33.2
10	Hot rolling front	21.2	15.5	6	5.9	-
	After hot rolling.	14.5	13.2	2	7.5	40.1
10	Hot rolling front	18.1	14.6	3.3	6.2	-
	After hot rolling.	11.2	10.1	1	12.5	50.8
Saturate	Hot rolling front	14.9	13.8	1.6	6.9	-
	After hot rolling.	9.6	9.1	0.6	13.4	66.1

It can be seen from Table 5 that after the copolymer drilling fluid added with NaCI is hot rolled, the viscosity decreases at high temperature, and the higher the salt content, the greater the viscosity decreases. This is mainly because the existence of NaCI makes the coalescence between copolymer and clay particles more obvious at high temperature. It can be seen from the measurement of API fluid loss of drilling fluid system that AM/AMPS/SSS/NVP quadripolymer fluid loss reducer has excellent high temperature and salt resistance.

Before hot rolling, the copolymer fluid loss reducer reduced the API fluid loss of saturated brine drilling fluid to 6.9m1. After aging at 180℃ at high temperature, the copolymer fluid loss reducer still has a good fluid loss reduction effect. The API fluid loss of 10.0% brine drilling fluid containing copolymer is only 5.9m1, and the fluid loss at high temperature and high pressure is less than 40m1.

5. Summary

(1) Through the characterization of AM/AMPS/SSS/NVP quadripolymer filtrate reducer, the molecular structure, viscosity-average molecular weight and thermal stability of the copolymer filtrate reducer were analyzed. The results show that the chemical composition and molecular structure of the copolymer fluid loss additive meet the design requirements, the molecular weight and molecular weight distribution are reasonable, and the temperature resistance is superior.

(2) Evaluate the performance of AM/AMPS/SSS/NVP quadripolymer fluid loss additive. The evaluation results show that the copolymer fluid loss additive has good fluid loss reduction performance, and the temperature resistance and salt resistance of a single agent are up to 180 0C and 1 100000mg/L respectively.

(3) The polymer with the molar ratio of AM:AMPS:SSS:MAH=2:2:3:1 has the best fluid loss reduction effect in saturated brine, and the fluid loss in 30min is only 11.3mL.

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