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ORIGINAL CONTRIBUTION

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Properties of the forpolymer of *N*-vinylpyrrolidone with itaconic acid, acrylamide and 2-acrylamido-2-methyl-1-propane sulfonic acid as a fluid-loss reducer for drilling fluid at high temperatures

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Abstract The forpolymer of *N*-vinylpyrrolidone (NVP), itaconic acid (IA), acrylamide (AM) and 2-acrylamido-2-methyl-1-propane sulfonic acid (AMPS) was synthesized through free-radical polymerization and was characterized using Fourier transform IR spectroscopy. The colloidal properties of the drilling fluid were investigated in the form of fresh-water or salt-water mud. It was found that the NVP–IA–AM–AMPS forpolymer had strong effects on the properties of the mud. The filtrate volume decreased with the increase of the forpolymer concentration before or after the aging test at 220 °C, and the filtrate volume after the aging test was larger than that before the aging test, but was still very small compared with the corresponding base mud. In addition, the rheological properties of both fresh-water mud and salt-water mud were modified by the forpolymer. The particle size data demonstrated that the average size of the clay particle after the aging test was larger than that before the aging test and that the particle size of the fresh-water mud was narrower compared with that of the salt-water mud before and after the aging test, respectively. The drilling fluid containing the forpolymer had an excellent tolerance to salt and high temperature.

Key words *N*-Vinylpyrrolidone · Itaconic acid · Acrylamide · Fluid-loss reducer · Drilling fluid

Introduction

A very important application of clay suspensions is their use as drilling fluids in the oil industry [1]. One of the functions for the drilling fluid (i.e. mud) is to form a thin filter cake on the wall of the borehole, which can prevent water leak-off into the geologic formation, and maintain the stability of the wellbore [2–5]. In recent years, there have been a number of challenges for drilling fluids to keep pace with the advancing operational drilling technologies. One of these challenges is high-temperature (and by extension, high-pressure) wells [6]. Since the 1980s, environmental legislation has increased restrictions on the use of oil-based fluids, which possess high-thermal stability, and progressively new water-based formulations presenting improved properties for specific applications at high temperature are required [7–9]. A water-based drilling fluid consists of water, salt, polymers, inert solids and clays such as montmorillonite (commercially known as bentonite). The polymers are intentionally added into drilling fluids to perform very specific functions, such as fluid-loss control, rheology modification and shale stabilization, etc. [10–17].

The development of novel synthetic fluid-loss reducers and polymeric deflocculants has helped make water-based mud systems stable for high-temperature and high-pressure drillings, and the copolymer or terpolymer containing *N*-vinylpyrrolidone (NVP) or 2-acrylamido-2-methyl-1-propane sulphonic acid (AMPS) plays an important role in these synthetic polymers [5, 7, 18–25]. All the former literature indicates that pyrrolidone and sulfonated segments may be resistant to high temperatures and tolerant to the action of salt, and the muds with the polymers containing pyrrolidone and sulfonated groups can be used in some hostile environments. Till now, no research has been reported on the forpolymer of NVP, AMPS, itaconic acid (IA) and acrylamide (AM) as well as its mud formulation. The aim of this work was to find some useful information about the colloid behavior, such as the particle size, the fluid-loss and the rheological properties of water-based drilling fluids with the forpolymer before and after aging tests at 220 °C for 16 h.

Experimental

Materials

The vinyl monomers NVP, IA, AM and AMPS were of analytical grade. Potassium persulfate (KPS) and sodium hydroxide were also of analytical grade. The raw bentonite sample was 96% montmorillonite donated by Shengli Oilfield, Dongying, China. The NVP-AM-AMPS and IA-AM-AMPS terpolymers were gained from Zhongyuan Oilfield, Puyang, China. The molecular weights of both terpolymers were approximately 600,000.

The bentonites used in the measurements of the particle size were purified through a series of procedures as follows. Raw bentonites were dispersed (about 7%) in 1 N sodium chloride solution and shaken for 12 h, and the sodium chloride supernatant and the gravel at the bottom were removed. Then, the suspension was centrifuged (3500 rpm for 10 min), and the sodium chloride supernatant was removed. The sodium bentonites obtained were washed in deionized water and centrifuged at 5000 rpm for 30 min; afterwards, the supernatant water containing chloride ions was removed. A silver nitrate test indicated that the final bentonite contained very few chloride ions.

Preparation and characterization of the forpolymer

The polymerization of the NVP–IA–AM–AMPS forpolymer having an initial weight ratio of NVP:IA:AM:AMPS = 10:10:40:40 was carried out in a 1000-ml glass reactor fitted with a stirrer, a condenser, a thermometer and a nitrogen inlet. The reactor was repeatedly degassed and purged with nitrogen, and deionized water was added. Then, the water was heated to 60 °C and approximately one-tenth of the monomer solution (containing NVP, a 40% solution of disodium IA, a 50% AM solution and a 50% solution of sodium AMPS) was fed followed by an aqueous solution containing one-seventh of the total KPS dosage. After 10 min, the remaining monomer mixture was added dropwise over 1 h, and an aqueous solution containing one-seventh of the total KPS dosage was fed every 6 min. The reaction was maintained at 60 °C for an additional 3.5 h. While cooled to 40 °C, the reaction mixture was slowly poured into 3 l acetone, and the precipitated polymer was isolated and dried under vacuum at 40 °C. The forpolymer prepared was dissolved in distilled water, precipitated in acetone and dried under vacuum again. Finally, a white forpolymer product was obtained.

The molecular weight of the forpolymer was about 600,000 as measured by gel permeation chromatography. The IR spectra (4000–400 cm^{-1}) of the forpolymer was measured using a Nicolet 20SX Fourier transform (FT) IR spectrophotometer with the KBr disc technique.

Mud preparation and aging tests

The fresh-water base mud containing 4% of sodium bentonite and 0.2% of Na_2CO_3 was prepared by mixing the raw bentonite, Na_2CO_3 and fresh water at a certain ratio, stirring for 20 min at a high speed of 10,000 rpm and aging for 24 h at room temperature. The salt-water base mud containing 8% of sodium bentonite, 4% of NaCl and 0.4% of Na_2CO_3 was prepared by a similar procedure to that of the fresh-water base mud preparation. The forpolymer mud was obtained by addition of NVP–IA–AM–AMPS forpolymer into the corresponding base mud, stirring for 20 min at 10,000 rpm and aging for 24 h at room temperature.

Aging experiments of bentonite–forpolymer muds were carried out in a GH-3-type rolling oven through hot rolling at 220 °C for 16 h. Mud property tests or particle size measurements were performed before and after the aging experiments.

Mud property tests

Mud property tests were measured according to American Petroleum Institute (API) specifications. The API filtrate volume (FL) of the mud was determined with an SD-type medium-pressure filtration apparatus made by Qingdao Photograph Camera Factory, China. The rheological parameters, such as the apparent viscosity, the plastic viscosity and the yield point, were determined through measurements of the viscosities at two rotation rates of 600 and 300 rpm using a ZNN-D6-type rotating viscometer.

Particle size measurements of muds

The average size of the clay particles and their size distribution were determined by use of a ZetaSizer 3000. The samples were usually dispersed by ultrasonic agitation before the size measurements, and the maximum concentration was set at 0.1 g/l. The samples were diluted to a certain solid content with deionized water with a small amount of salt added, for example, 1 mmol/l NaCl, and this ensured that an extended electric double layer did not artificially increase the size by a few nanometers. The count rates were in the range 50–200 kcp (1000s per second). The clay sample used here was the purified bentonite.

Results and discussion

In the IR spectra of the NVP–IA–AM–AMPS forpolymer, there were a strong band at 1669 cm^{-1} due to the carbonyl in the amide groups, a band at 1219 cm^{-1} due to C–O stretching of COO– groups, an NH_2 stretching band at 3335 cm^{-1}, an NH stretching band at 3204 cm^{-1}, an NH bending band at 1545 cm^{-1}, a CH stretching band at 2930 cm^{-1}, a CH bending band at 1451 cm^{-1} and two SO_2 stretching bands at 1044 and 529 cm^{-1}. There was no olefinic band at 1635–1620 cm^{-1}. After the aging test at 220 °C, the IR spectra of the forpolymer were similar to that before the aging test, because the forpolymer contained too many functional groups although some of the groups were hydrolyzed and degraded.

A series of fresh-water and salt-water mud formulations with different forpolymer concentrations were prepared, and the mud properties (such as the filtrate volume and the rheological properties) were measured before and after aging tests at 220 °C. The clay samples used in the measurements of the mud properties were raw bentonites. Then, the effects of forpolymer concentrations on fluid-loss and rheological properties were investigated, in other words the colloid properties of the forpolymer–clay system could be disclosed.

The effects of the NVP–IA–AM–AMPS forpolymer concentrations on the filtrate volumes (FL) of fresh-water muds and salt-water muds are shown in Figs. 1 and 2, respectively. The filtrate volume showed an aspect of the colloid stability for the polymer mud. It could be seen that the filtrate volume decreased with the increase in the forpolymer concentration before or after the aging test. The filtrate volume after the aging test was larger than that before the aging test, meaning that the elevated temperature could raise the permeability of the filter cake and spoil a part of clay gel structure. However, when aged at 220 °C, even if some clay particles aggregated, the residual clay network was kept [11], so the filtrate volumes of fresh-water mud and salt-water mud were still small compared with the corresponding base muds (without the forpolymer added). When the forpolymer concentration was high enough (e.g. 0.6% for fresh-water mud and 1.2% for salt-water mud), the filtrate properties changed very slowly with the increase in the forpolymer concentration; this illustrated that the forpolymer was a very efficient fluid-loss reducer. In accordance with the view of Zhuang et al. [4], the forpolymer might be adsorbed on the clay surface to raise the ζ potential of the clay particle, plug the filter cake holes, improve the quality of the filter cake, reduce the coefficient of penetration and filtration and, therefore, the mud filtrate volume was reduced. In salt-water mud treated with the NVP–IA–AM–AMPS forpolymer, the negative charge density on the molecular chain was reduced, the molecular chain contracted and the ζ potential on the clay surface decreased owing to the salt screening effect of NaCl on the electric double layer of the clay particles. Hence, the fluid-loss controlling properties of the salt-water mud dropped in contrast with the fresh-water mud.

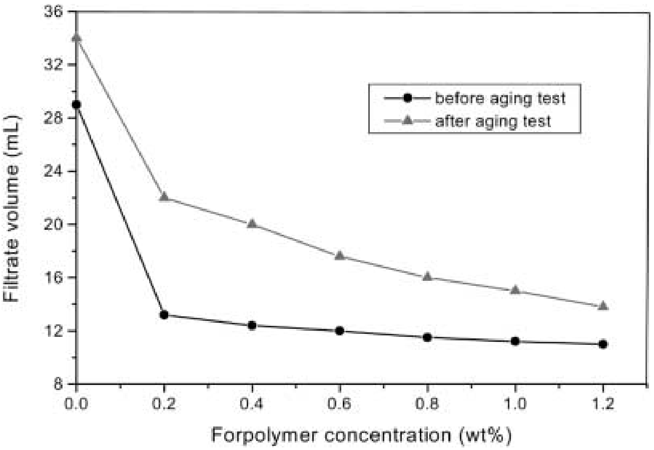

Fig. 1 The effect of the *N*-vinylpyrrolidone (*NVP*)–itaconic acid (*IA*)–acrylamide (*AM*)–2-acrylamido-2-methyl-1-propane sulfonic acid (*AMPS*) forpolymer concentration on the filtrate volume of the fresh-water mud before and after the aging test

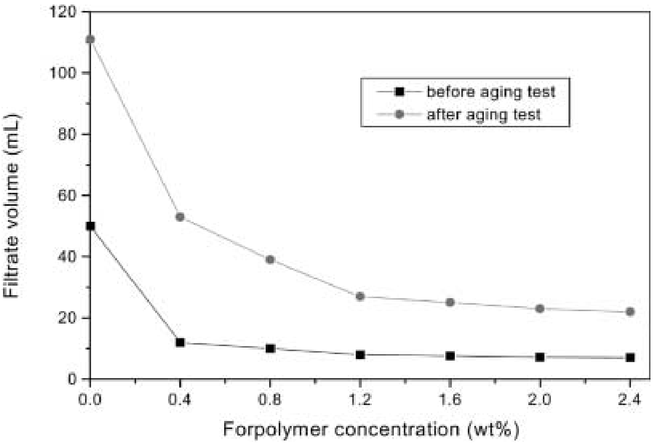

Fig. 2 The effect of the NVP–IA–AM–AMPS forpolymer concentration on the filtrate volume of the salt-water mud before and after the aging test

The effects of NVP–IA–AM–AMPS forpolymer concentrations on the rheological behavior of fresh-water muds and salt-water muds are listed in Tables 1 and 2. For the fresh-water mud (Table 1), the apparent viscosity (η_a) and the plastic viscosity (η_b) increased with the increase in the forpolymer concentration before the aging test, and the yield point (τ_0) was basically constant. After the aging test, the fresh-water mud lost a lot of viscosity, η_a and η_b changed very little with the variation of the forpolymer concentration, and τ_0 even

Table 1 Rheological behavior of fresh-water muds under different forpolymer concentrations (before and after the aging tests). All rheological data were measured at 30 ± 0.5 °C

Forpolymer concentration (%)	η_a (mPas)		η_b (mPas)		τ_0 (Pa)	
	Before	After	Before	After	Before	After
0[a]	5.2	10.0	3.0	7.5	1.8	2.5
0.2	9.0	4.5	5.0	5.0	4.0	0
0.4	11.0	4.5	7.0	5.0	4.0	0
0.6	13.5	5.2	9.0	5.5	4.5	0
0.8	14.0	5.5	10.0	5.5	4.0	0
1.0	15.8	5.5	11.5	5.5	4.2	0
1.2	19.0	6.5	14.0	6.0	5.0	0.5

[a] Fresh-water base mud

Table 2 Rheological behavior of salt-water muds under different forpolymer concentrations (before and after the aging tests). All rheological data were measured at 30 ± 0.5 °C

Forpolymer concentration (%)	η_a (mPas)		η_b (mPas)		τ_0 (Pa)	
	Before	After	Before	After	Before	After
0[a]	5.2	6.0	3.0	3.0	2.2	3.0
0.4	6.5	6.0	4.0	4.0	2.5	2.0
0.8	7.0	5.0	5.0	3.0	2.0	2.0
1.2	10.0	3.2	7.0	2.5	3.0	1.0
1.6	11.0	3.0	8.0	2.0	2.0	0.8
2.0	11.5	3.0	9.0	3.0	3.5	0
2.4	14.0	3.0	11.5	4.0	2.5	0

[a] Salt-water base mud

became zero. According to Zhang and Yin [14], clay particles in water are attracted to each other and build up a gel structure with viscosity and strength, some anion polymers adsorbed on clay particles may reduce or inhibit the attraction between particles by electrostatic repulsion, and the mud viscosity and the yield power may be reduced. Thus, the NVP–IA–AM–AMPS forpolymer apparently modified the rheological performance of the fresh-water mud on the condition that the mud had good filtrate properties.

From Table 2, it is found that η_a, η_b (before and after the aging test) and τ_0 (before aging test) of the salt-water mud had similar changing trends to that of the fresh-water mud, meaning that the mud formulation had an excellent tolerance to salt. However, τ_0 decreased with the increase in the forpolymer concentration after the aging test; this illustrated that the forpolymer possessed strong thinning abilities because the NVP–IA–AM–AMPS forpolymer contained many carboxyl and sulfonic groups which exhibited unique hydration and dispersion capacities. In the salt-water clay suspension, competition existed between the aggregation and the dispersion of the clay particles [2]. Adsorbed forpolymers with negative charges tended to disperse clay particles by electrostatic repulsion and break up the gel structure of the clay particles. In addition, the salt screening effect on the electric double layer of the clay particle decreased with the increase in the forpolymer concentration, which led to the enhancement of the electrostatic repulsion and the reduction of the aggregation [13]. Therefore, the rheological properties of the salt-water mud was modified.

The mud property comparisons of the NVP–IA–AM–AMPS forpolymer with the IA–AM–AMPS terpolymer and the NVP–AM–AMPS terpolymer are shown in Tables 3 and 4. Obviously, either in the fresh-water mud or in the salt-water mud, the filtrate volume of the forpolymer was smaller than that of the terpolymers before and after the aging test. At the same

Table 3 The fresh-water mud properties of the *N*-vinylpyrrolidone (NVP)–itaconic acid (IA)–acrylamide (AM)–2-acrylamido-2-methyl-1-propane sulfonic acid (AMPS) forpolymer compared with the terpolmers (before and after aging tests). All the polymer concentrations in the fresh-water mud were 0.5%

The mud samples	η_a (mPas)		η_b (mPas)		τ_0 (Pa)		FL(ml)	
	Before	After	Before	After	Before	After	Before	After
0[a]	5.2	10.0	3.0	7.5	1.8	2.5	29.0	34.2
1[b]	31.5	4.5	17.0	4.0	14.5	0	15.2	20.4
2[c]	28.5	6.8	12.5	7.5	16.0	0	13.0	19.6
3[d]	11.3	5.0	7.5	5.0	3.7	0	12.0	18.2

[a] Fresh-water base mud
[b] IA–AM–AMPS terpolymer mud
[c] NVP–AM–AMPS terpolymer mud
[d] NVP–IA–AM–AMPS forpolymer mud

Table 4 The salt-water mud properties of the NVP–IA–AM–AMPS forpolymer compared with the terpolmers (before and after aging tests). All the polymer concentrations in the salt-water mud were 1.5%

The mud samples	η_a (mPas)		η_b (mPas)		τ_0 (Pa)		FL(ml)	
	Before	After	Before	After	Before	After	Before	fter
0[a]	5.2	6.0	3.0	3.0	2.2	3.0	52.5	110.0
1[b]	14.0	2.8	12.5	2.5	1.5	0.2	20.8	39.8
2[c]	28.0	7.0	17.0	4.5	14.5	2.5	17.0	33.0
3[d]	12.5	5.5	9.0	3.5	3.5	2.0	15.8	26.3

[a] Salt-water base mud
[b] IA–AM–AMPS terpolymer mud
[c] NVP–AM–AMPS terpolymer mud
[d] NVP–IA–AM–AMPS forpolymer mud

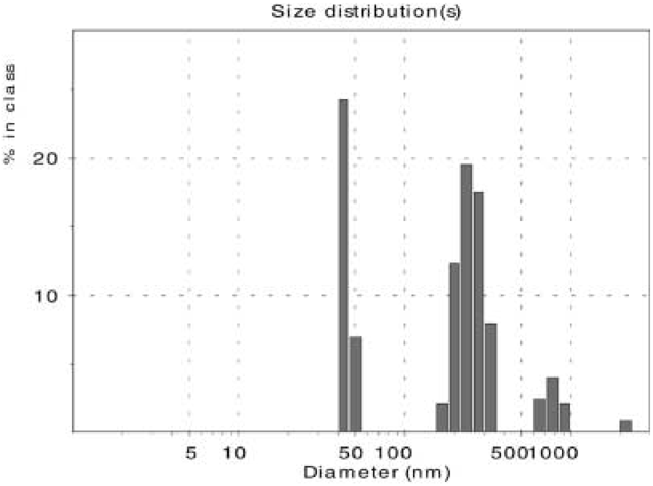

Fig. 3 The particle size distribution of the NVP–IA–AM–AMPS forpolymer fresh-water mud before the aging test

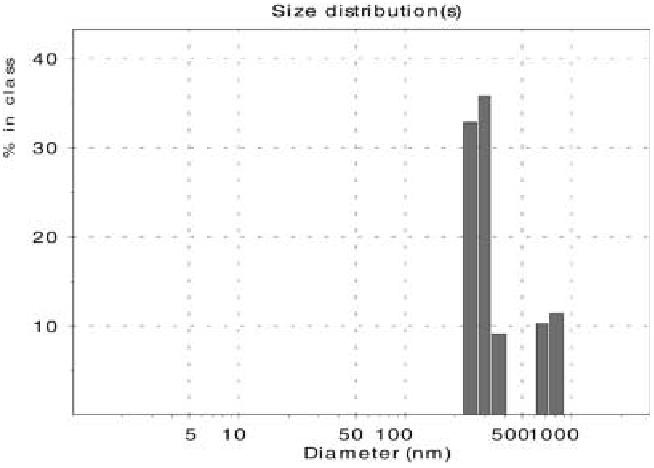

Fig. 4 The particle size distribution of the NVP–IA–AM–AMPS forpolymer fresh-water mud after the aging test

time, the apparent viscosity, the plastic viscosity and the yield point of the forpolymer were a little larger than those of the IA–AM–AMPS terpolymer, but far smaller than those of the NVP–AM–AMPS terpolymer, and the absolute values of the viscosities were very small. This means that the forpolymer muds had good rheological behavior. As a result, the NVP–IA–AM–AMPS forpolymer seemed to be the most effective fluid-loss reducer compared with its relevant terpolymers.

From Plank's point of view [7], the fluid-loss reducer for the polymers with higher molecular weight not only decreased the filtrate volume, but greatly increased the mud viscosity, resulting in bad rheological performance, so thinners might be required for the mud formulations. Nevertheless, the mud property results confirmed that the NVP–IA–AM–AMPS forpolymer with the average molecular weight of 600,000 was an excellent fluid-loss reducer as well as a good rheology modifier, and it was not necessary to add the thinner to the mud formulation. The corresponding mud had an excellent tolerance to salt and a high stability to temperature.

The particle size of the fresh-water mud containing 4% of the purified sodium bentonite and 0.5% of the NVP–IA–AM–AMPS forpolymer was measured using a ZetaSizer 3000. The clay used here was the purified bentonite, and the sample concentration was set at 0.1 g/l. The average particle size of the fresh-water base mud was 501.6 nm and the polydispersity index was 0.511 after the aging test, while they were 331.5 nm and 0.481, respectively, before the aging test. The particle size results of the forpolymer fresh-water mud before and after the aging test are shown in Figs. 3 and 4. The average size of the clay particle for the forpolymer fresh-water mud before the aging test was 307.2 nm, the polydispersity index was 0.597 and the particle size distribution was wide. The average size of the clay particle after the aging test was 410.4 nm, the polydispersity index was 0.537 and the particle size distribution was narrower. It was clear that the aggregation of the clay particles increased after the aging test at 220 °C, and the forpolymer fresh-water mud had excellent dispersion performance compared with the base mud. This result is described as follows. First, the hydrated shell of the clay particles was partly damaged at high temperature and the attraction between the particles enlarged. Second, the carboxyl groups in IA segments and the sulfonic groups in AMPS segments carried negative charges; these negative charges greatly increased the width of the electric double layer around the clay particle before the aging test; however, these anionic groups were partly hydrolyzed and thermally degraded at the high temperature of 220 °C, leading to the electric double layer contracted after the aging test. So the repulsion between the clay particles weakened and the aggregation of the particles was enhanced according to Derjaguin–Landau–Verwey–Overbeek (DLVO) theory. Of course, most pyrrolidone segments and sulfonic groups were stable at high temperature, the forpolymer–clay particles had not completely aggregated, the particle size was smaller than that of the fresh-water base mud and the forpolymer–clay suspension still exhibited desirable filtrate properties after the aging tests.

Moreover, the particles larger than 500 nm seemed to be slightly affected by aging (Figs. 3, 4). The possible explanation for this novel result may be as follows. On the one hand, the surface free energy of the large particle is less than that of the small particle in the view of the basic theory of colloid chemistry. This means that the large particle is more stable than the small particle, while the large particle displays thermodynamic stability to some extent before and after the aging test. On the other

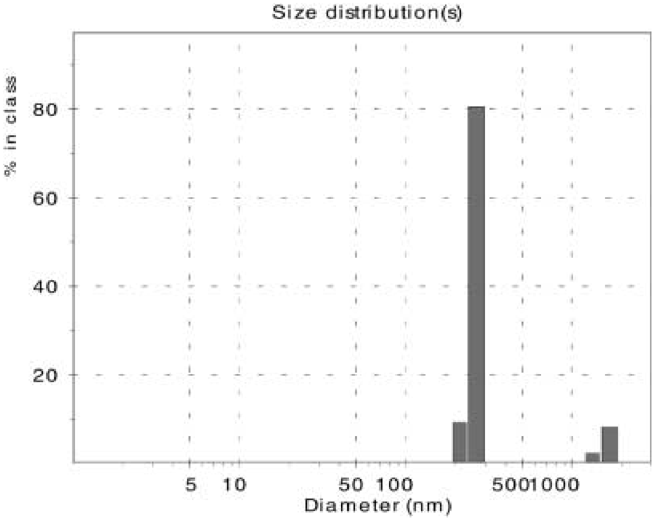

Fig. 5 The particle size distribution of the NVP–IA–AM–AMPS forpolymer salt-water mud before the aging test

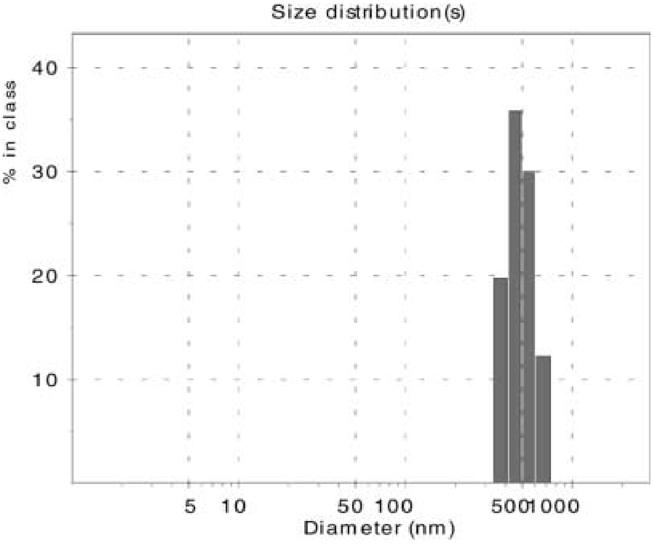

Fig. 6 The particle size distribution of the NVP–IA–AM–AMPS forpolymer salt-water mud after the aging test

hand, the clay particle has negative charges, and the electrostatic repulsion between the large particles was stronger than that between the small particles. Especially when the forpolymer with negative charges exists in the clay/water suspension, the electrostatic repulsion between the particles as well as the repulsion between the forpolymer molecule and the clay particle is stronger. As a result, the large particles retain electrostatic stability before and after the aging test. Besides, the forpolymer/clay/water suspension exhibits depletion stability owing to the unique hydration ability of the forpolymer. These stabilities ensure that the particle size of clay particles larger than 500 nm varies slightly before and after the aging test.

Simultaneously, the particle size of the salt-water mud containing 8% of the sodium bentonite was investigated. The average particle size of the salt-water base mud was 796.1 nm and the polydispersity index was 1.000 after the aging test, while they were 535.4 nm and 0.253, respectively, before the aging test. The particle size results of the forpolymer salt-water mud before and after the aging test are shown in Figs. 5 and 6. The average size of the clay particle for the forpolymer salt-water mud before the aging test was 482.6 nm and the polydispersity index was 0.072. The average size of the clay particle after the aging test was 761.9 nm, while the polydispersity index was 1.000. Just like for the fresh-water mud, these results also illustrate that the aggregation of the clay particles increased in the course of the aging test at 220 °C, and the forpolymer salt-water mud had better dispersion performance than the salt-water base mud. The explanation of this result is described in the same way as that of the forpolymer fresh-water mud, except for the salt screening effect on the mud system. The salt screening effect made the repulsion between the clay particles weaker, the aggregation of the particles increased in light of the DLVO theory and the average particle size was correspondingly larger compared with that of the fresh-water mud before and after the aging test.

Conclusions

The NVP–IA–AM–AMPS forpolymer was synthesized through free-radical polymerization and was characterized using the FTIR spectroscopy method. The IR spectra of the forpolymer confirmed that there was no olefinic band at 1635–1620 cm^{-1}.

The NVP–IA–AM–AMPS forpolymer had strong effects on the mud properties. It could be seen that the filtrate volume decreased with the increase in the forpolymer concentration before or after the aging test and that the filtrate volume after the aging test was larger than that before the aging test, but it was still very small compared with the corresponding base mud. In addition, the η_a and the η_b regularly increased before the aging test, while they changed very little with the increase in the forpolymer concentration after the aging test, both in the fresh-water mud and in the salt-water mud. τ_0 basically remained constant with the variation of the forpolymer concentration, and τ_0 even became zero after the aging test for the salt-water mud. Apparently, the rheological properties of both fresh-water mud and salt-water mud were modified by the NVP–IA–AM–AMPS forpolymer. The particle size data demonstrated that the average size of the clay particle after the aging test was larger than that before the aging test and that the particle size of the fresh-water mud was narrower compared with that of the salt-water mud before and after the aging test. It was clear that the aggregation of the clay particles increased owing to the high temperature and the salt screening effects.

In one sentence, the NVP–IA–AM–AMPS forpolymer with an average molecular weight of 600,000 was an excellent fluid-loss reducer at high temperature, and it was not necessary to add a thinner (rheology modifier) to the mud formulation. The corresponding drilling fluid had an excellent tolerance to salt and a high stability to temperature.

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