

# Properties of High-Temperature Drilling Fluids Incorporating Disodium Itaconate/Acrylamide/Sodium 2-Acrylamido-2-methylpropanesulfonate Terpolymers as Fluid-Loss Reducers

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**ABSTRACT:** The terpolymer of disodium itaconate (DIA), acrylamide (AM) and sodium 2-acrylamido-2-methyl-1-propane sulfonate (SAMPS) was synthesized through free-radical polymerization, and characterized using FTIR and TGA methods. The IR spectra of DIA-AM-SAMPS terpolymer confirmed that there was no olefinic band at 1635–1620  $\text{cm}^{-1}$ , while the TGA results revealed that the terpolymer was of higher thermal stability than the SAMPS homopolymer. The filtrate volume reduced with increase of the terpolymer concentration before or after the aging test. The rheology properties of both fresh-water mud and salt-water mud were improved by DIA-AM-SAMPS terpolymer, and apparent viscosity ( $\eta_a$ ); plastic viscosity ( $\eta_b$ ) and yield point ( $\tau_0$ ) of salt-water mud reached the smallest values at 1.2% of the terpolymer concentration after the aging test. The particle size data demonstrated that only a small change of the clay particle size occurred before and after the aging test at 220°C. This further confirmed the thermal stability of the terpolymer–clay dispersion from another point of view. © 2002 John Wiley & Sons, Inc. *J Appl Polym Sci* 83: 3068–3075, 2002; DOI 10.1002/app.2335

**Key words:** disodium itaconate; acrylamide; sodium 2-acrylamido-2-methyl-1-propane sulfonate; fluid-loss reducer; drilling fluid

## INTRODUCTION

When drilling an oil well, drilling fluid (i.e., mud) is circulated downwards through the drillpipe, then upwards through the annular space between drillpipe and borehole at sufficiently high speed to cool the drilling bit effectively, to exert hydro-

static pressure, to carry the drill cuttings up to surface, and to form a filter cake on the walls of the borehole, which can prevent water leak-off from the drilling fluid into the geologic formation.<sup>1–4</sup> For the drilling fluid, adequate filtration control is essential to prevent drilling problems such as excessive torque and drag; differential pressure sticking; borehole instability; and formation damage.<sup>5–8</sup> In the past 30 years, there have been a number of challenges for drilling fluids to keep pace with advancing operational drilling technology. One of these challenges is high-temperature (and by extension, high-pressure) wells.<sup>9</sup>

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Oil-based mud has been the fluid of choice in hostile drilling environments for many years because of its thermal stability up to 500°F, high rate of penetration, and easy mud maintenance. Since the 1980s, environmental legislation has increased restrictions on the use of oil-based fluids, and progressively new water-based formulations presenting improved properties for specific applications are required.<sup>10–12</sup>

A water-based drilling fluid (especially at high temperature) consists of water, salt, polymers, inert solids, and clays such as montmorillonites (commercially known as bentonites). Polymers are intentionally added to drilling fluids to perform very specific functions, such as fluid-loss control, rheology modification, shale stabilization, etc. It is the task of the mud engineer to let a drilling-fluid system perform at its best within the given limitations of oil drilling industry. Such limitations are imposed by the degradation of polymers at high temperatures, which leads to loss of rheology and fluid-loss control.<sup>13–19</sup> Therefore, the thermal stability of the polymers is of great significance.<sup>20</sup> However, conventional polymers such as partially hydrolyzed polyacrylamide and polyanionic cellulose cannot meet high-temperature limitations due to their rapid hydrolysis, strongly thermal degradation, and undesirable properties of the corresponding drilling fluid.<sup>21–23</sup>

Fortunately, 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 (HTHP) drilling,<sup>10</sup> and the copolymer or terpolymer containing sodium 2-acrylamido-2-methyl-1-propane sulfonate (SAMPS) plays an important role in these synthetic polymers. McCormick,<sup>24–26</sup> Travas-Sejdic,<sup>27–28</sup> Aggour<sup>29–30</sup> etc., have conducted extensive research on syntheses, characterizations, and thermal properties of SAMPS copolymers. Audibert,<sup>31</sup> Parker,<sup>23</sup> and Collette<sup>4</sup> have investigated hydrolysis and thermal degradation of sulfonated polymers with an SAMPS segment used in high-temperature drilling fluids. They have also studied fluid loss and rheology properties of the corresponding muds at elevated temperatures. All the former literature represents that sulfonated groups may be resistant to high temperature and tolerant to the action of high salt, and the muds containing sulfonated polymers can be used in some hostile environments. Until now, very little research has been reported on the terpolymer of disodium itaconate

(DIA), acrylamide (AM), and SAMPS as well as its mud formulation.

The purpose of this work was to investigate the synthetic and thermal characters of DIA-AM-SAMPS terpolymer, and find some useful information on particle size, the fluid loss, and rheology properties of water-based drilling fluids before and after aging tests at 220°C for 16 h.

## EXPERIMENTAL

### Materials

Sodium persulfate (SPS) and the vinyl monomers such as IA, AM, and AMPS were all chemical agents, used without further purification, and provided by Beijing Chemical Company in China, and were 99.5% pure. The raw bentonite sample was 96% montmorillonite, provided by Shengli Oilfield, Dongying, China.

The bentonites used in the measurements of 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. When the bentonites sedimented downwards to 2/3 of the total suspension volume, the sodium chloride supernatant and the gravel at the bottom were removed. Such operations were repeated several times until the clay suspension formed a stable dispersion and became difficult to sedimentate naturally. Then the suspension was centrifuged (3500 rpm for 10 min), the sodium chloride supernatant was removed, and this operation was repeated four times to ensure that the totality of exchangeable cations were substituted by sodium ions. The sodium bentonites obtained above were washed in deionized water and centrifuged for 30 min at 5000 rpm, afterwards the supernatant water containing chloride ions was removed. This final procedure was repeated until the system formed a stable dispersion and became difficult to centrifuge. The final bentonite contained very few chloride ions, shown with a silver nitrate test, its solid content was approximately 6.5%, and this purified clay suspension could be directly used in the preparation of drilling mud.

### Preparation and Characterization of the Terpolymer

The polymerization of DIA-AM-SAMPS terpolymer was carried out in a 1000-mL glass reactor

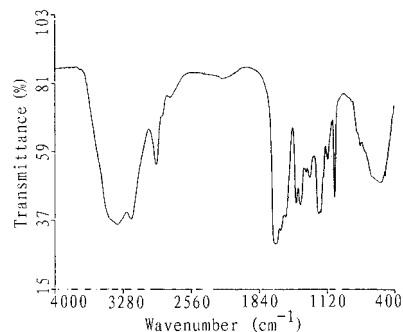
fitted with a stirrer, condenser, thermometer, and nitrogen inlet. The reactor was repeatedly degassed and purged with the nitrogen, and 435 g of deionized water, 62.5 g of a 40% solution of DIA, 250 g of a 50% AM solution, and 200 g of a 50% solution of SAMPS were added. The mixture was stirred, and heated to 75°C by means of an external heating jacket. Then 52.5 g of a 5% solution of SPS was drop-fed into the reactor uniformly over a period of 2 h, maintaining a temperature of 75 ± 2°C. At the end of the SPS addition, 75°C was maintained for an additional 2 h. While cooled to 40°C, the reaction mixture was slowly poured into 3 L of acetone, and the precipitated polymer was isolated and dried under vacuum at 40°C. The prepared terpolymer was dissolved in distilled water, precipitated in acetone, and dried under vacuum again. Finally, a white terpolymer product was obtained.

The weight-average molecular weight of the terpolymer was about 500,000, as measured by Waters 510/410 gel permeation chromatography (GPC), and the magnitude of the probable error was approximately 5%. The infrared spectra (IR) (4000–400 cm<sup>-1</sup>) of the purified DIA-AM-SAMPS terpolymer was measured by a Nicolet 20SX FTIR spectrophotometer by the KBr disc technique. Thermogravimetry analysis (TGA) of the terpolymer was made using a Rheometric Scientific TGA-1500. Samples (10 mg) were heated at 10°C min<sup>-1</sup> in a dynamic nitrogen atmosphere.

### Mud Preparation and Aging Tests

The fresh-water base mud containing 4% of sodium bentonite and 0.2% of Na<sub>2</sub>CO<sub>3</sub> was prepared by mixing the raw bentonite, Na<sub>2</sub>CO<sub>3</sub>, 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<sub>2</sub>CO<sub>3</sub>, was prepared by a similar procedure to that of the fresh-water base mud preparation. The terpolymer mud was obtained with an addition of DIA-AM-SAMPS terpolymer into the corresponding base mud, stirring for 20 min at a high speed of 10,000 rpm, and aging for 24 h at room temperature.

Aging experiments of bentonite–terpolymer 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 aging experiments.



**Figure 1** IR spectra of DIA-AM-SAMPS terpolymer.

### Mud Property Tests

Mud property tests were measured according to American Petroleum Institute (API) specifications. API filtrate volume of mud was determined with an SD-type medium-pressure filtration apparatus made in Qingdao Photograph Camera Factory, China. The rheological parameters, such as apparent viscosity ( $\eta_a$ ), plastic viscosity ( $\eta_b$ ), and yield point ( $\tau_0$ ), were determined through measurements of the viscosity at two rotating rates of 600 and 300 rpm by a ZNN-D6 type rotating viscometer.

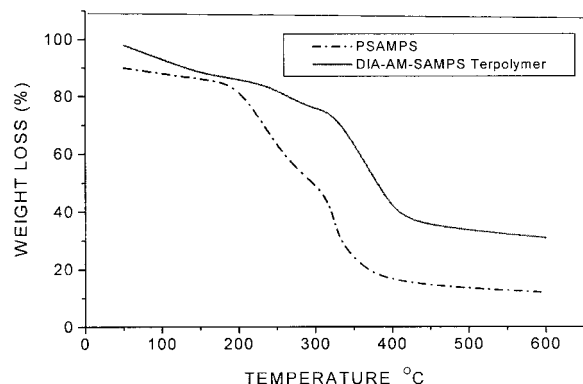
### Particle Size Measurements of Muds

The average size of clay particles and their size distributions were determined by use of a Zeta-sizer 3000. The samples were usually dispersed by ultrasonic agitation before size measurements, and the maximum concentration for measurements was set at 0.1 g/L. The samples were diluted to a certain solid content by demineralized water with a small amount of salt added, for example, 1 mmol L<sup>-1</sup> 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 a range of 50–200 Kcp (1000s per second). The clay used here was the purified bentonite.

## RESULTS AND DISCUSSION

### IR Characterization

In the IR spectra of DIA-AM-SAMPS terpolymer (see Fig. 1), there was a strong band at 1665 cm<sup>-1</sup> due to carbonyl in amide groups and a band at 1406 cm<sup>-1</sup> due to C—O stretching of COO<sup>-</sup> groups. The SAMPS units were revealed by a CO



**Figure 2** TGA curves for DIA-AM-SAMPS terpolymer and PSAMPS.

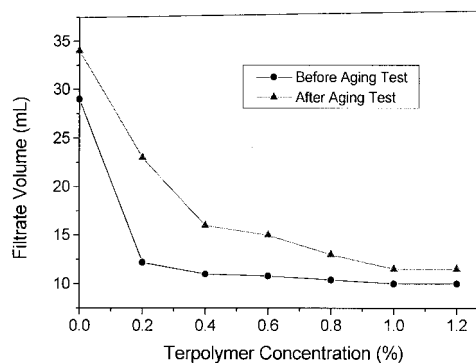
band at  $1558\text{ cm}^{-1}$ ,  $\text{SO}_2$  at  $1217\text{ cm}^{-1}$ , CH at  $1451\text{ cm}^{-1}$ , and NH at  $3211\text{ cm}^{-1}$ . The AM units were characterized by a CO band at  $1665\text{ cm}^{-1}$ , CH at  $579\text{ cm}^{-1}$ , and  $\text{NH}_2$  at  $3339\text{ cm}^{-1}$ . There was no olefinic band at  $1635\text{--}1620\text{ cm}^{-1}$ ,

#### Thermal Analysis of the Terpolymer

Figure 2 shows the TGA curves for SAMPS homopolymer (PSAMPS) and DIA-AM-SAMPS terpolymer. Obviously, the terpolymer was of higher thermal stability than SAMPS homopolymer. PSAMPS began to degrade at  $182^\circ\text{C}$ . When the temperature was increased to  $303^\circ\text{C}$ , a second decomposition process occurred. The thermal degradation of the terpolymer became appreciable at approximately  $240^\circ\text{C}$ . The terpolymer sample turned yellow at first, and later fused and the rate of degradation gradually decreased. The terpolymer exhibited three stages of the decomposition. The first decomposition stage began at  $240\text{--}270^\circ\text{C}$  and ended at  $300\text{--}330^\circ\text{C}$ . The second degradation stage occurred in the range of  $340\text{--}420^\circ\text{C}$ . The third stage became smooth at temperatures higher than  $450^\circ\text{C}$ . Here, the first stage corresponded to the decomposition of amide and carboxyl groups, the second stage to the degradation of sulfonic groups, and the third stage to the decomposition of the terpolymer backbone.

#### Effect of Terpolymer Concentration on Mud Properties

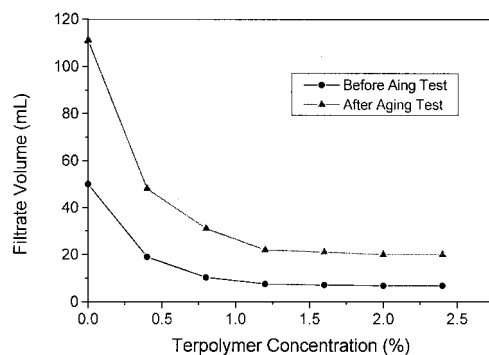
As a fluid-loss reducer, DIA-AM-SAMPS terpolymer could improve mud properties of high-temperature drilling fluid. A series of fresh-water or salt-water mud formulations with different terpolymer concentrations were formulated, and the



**Figure 3** The effect of DIA-AM-SAMPS terpolymer concentration on the filtrate volume of fresh-water mud (before and after the aging test).

appropriate mud properties such as filtrate volume and rheology properties were measured before and after aging tests at  $220^\circ\text{C}$ . The clay sample used in this part was raw bentonite. Therefore, the effect of terpolymer concentration on fluid-loss or rheology properties could be determined.

Figures 3 and 4 show the effect of terpolymer concentrations on the filtrate volume of fresh-water mud and salt-water mud, respectively. It can be seen that the filtrate volume decreased with the increase of the terpolymer 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 permeability of the filter cake and spoil a part of the mud gel structure. However, if aged at elevated temperatures of  $220^\circ\text{C}$ , even if some clay aggregation occurred, the residual clay network was kept,<sup>14</sup> so the filtrate volumes of fresh-water mud and salt-water mud



**Figure 4** The effect of DIA-AM-SAMPS terpolymer concentration on the filtrate volume of salt-water mud (before and after the aging test).

**Table I Rheology Behaviors of Fresh-Water Muds under Different Terpolymer Concentrations (before and after Aging Tests)**

Terp. Conc. (%)	$\eta_a$ (mPa s)		$\eta_b$ (mPa s)		$\tau_0$ (Pa)	
	Before	After	Before	After	Before	After
0 <sup>a</sup>	5.2	10.0	3.0	7.5	1.8	2.5
0.2	14.8	4.2	8.0	4.5	6.8	0
0.4	17.5	4.5	10.0	4.5	7.5	0
0.6	19.5	5.0	11.0	5.0	8.5	0
0.8	22.8	7.5	13.5	8.0	9.2	0
1.0	27.0	8.0	17.0	8.5	10.0	0
1.2	37.0	11.8	20.0	11.0	11.0	0.8

All rheology data listed above were measured at  $30 \pm 0.5^\circ\text{C}$ . 0<sup>a</sup> represented base mud.

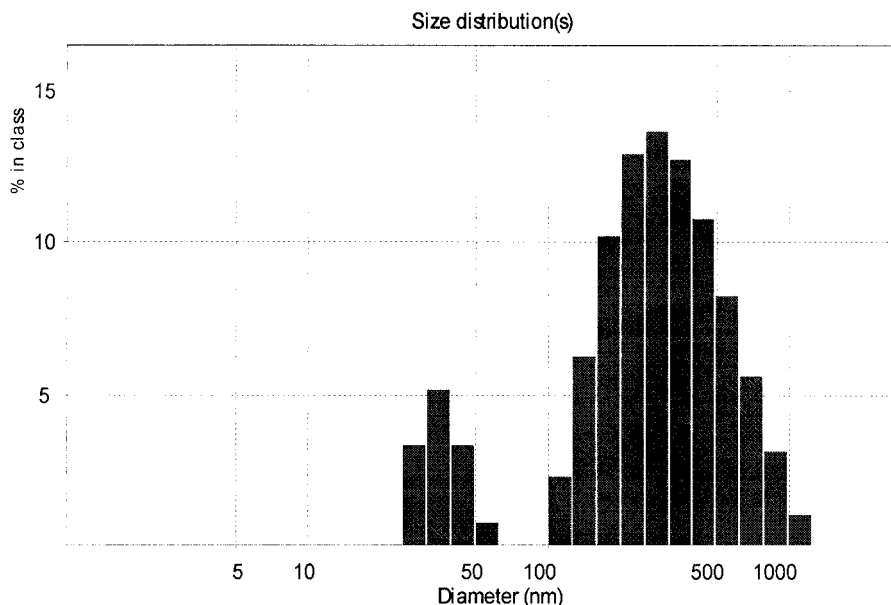
were still smaller than 20 mL (in accordance with Plank<sup>10</sup>) and 50 mL, respectively. When the terpolymer concentration was high enough (e.g., 1.2% for salt-water mud), the filtrate properties changed very slowly. This illustrated that DIA-AM-SAMPS terpolymer was a very efficient fluid-loss reducer. In accordance with Zhuang's view,<sup>3</sup> the terpolymer might be adsorbed on the clay surface to raise  $\zeta$ -potential of the clay particle, plug the filter cake holes, improve the quality of the filter cake, and reduce the coefficient of penetration and filtration; therefore, the mud filtrate volume was reduced. In salt-water mud treated by DIA-AM-SAMPS terpolymer, negative charge density on the molecular chain was reduced, the molecular chain contracted, and the  $\zeta$ -potential on the clay surface decreased, attributable to the salt screening effect of NaCl on the electric double layer of the clay particles, so fluid-loss controlling properties dropped.

The effects of terpolymer concentrations (Terp. Conc.) on the rheology behaviors of fresh-water mud and salt-water mud are listed in Tables I and II. For fresh-water mud (Table I), apparent viscosity ( $\eta_a$ ), plastic viscosity ( $\eta_b$ ), and yield point ( $\tau_0$ ) increased with the increase of terpolymer concentration except for the base mud after the aging test, and the DIA-AM-SAMPS terpolymer could decrease high-temperature mud viscosity and yield power in the concentration range of less than 1.2%. According to Zhang,<sup>17</sup> clay particles in water are attracted to each other to build up a gel structure with viscosity and strength, some anion-polymers adsorbed on clay particles may reduce or inhibit the attractions between particles by electrostatic repulsion, and the mud viscosity and yield power may be reduced. Therefore, DIA-AM-SAMPS terpolymer apparently improved the rheology performance of the fresh-water mud.

**Table II Rheology Behaviors of Salt-Water Muds under Different Terpolymer Concentrations (before and after Aging Tests)**

Terp. Conc. (%)	$\eta_a$ (mPa s)		$\eta_b$ (mPa s)		$\tau_0$ (Pa)	
	Before	After	Before	After	Before	After
0 <sup>a</sup>	5.2	6.0	3.0	3.0	2.2	3.0
0.4	5.5	5.0	3.0	1.0	2.5	3.5
0.8	6.0	6.0	4.0	1.0	2.0	4.5
1.2	5.5	3.8	5.0	1.0	0.5	2.2
1.6	8.0	5.0	6.0	1.0	2.0	3.5
2.0	11.5	6.5	9.0	1.8	2.5	3.8
2.4	12.5	6.5	12.0	2.0	0.5	3.5

All rheology data listed above were measured at  $30 \pm 0.5^\circ\text{C}$ . 0<sup>a</sup> represented base mud.



**Figure 5** The particle size distribution of the purified bentonite-terpolymer mud before the aging test.

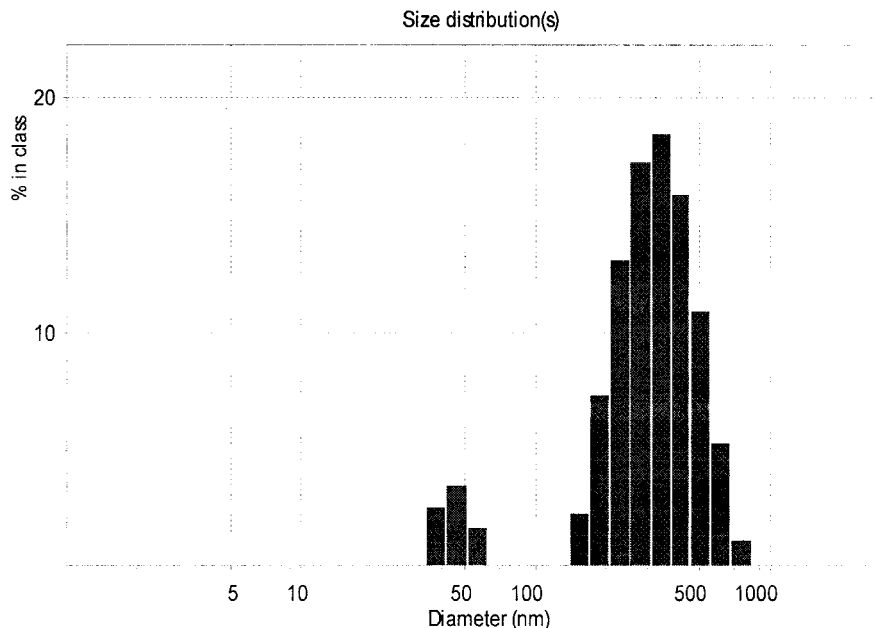
However, the rheology properties of the salt-water mud were not uniform (Table II). It was found that  $\eta_a$  and  $\tau_0$  of salt-water mud varied very little before and after the aging test, meaning that the mud formulation had an excellent tolerance to salt.  $\eta_a$ ,  $\eta_b$ , and  $\tau_0$  reached the smallest values at 1.2% of terpolymer concentration, and the mud exhibited the best rheology performance. This smallest value dosage of DIA-AM-SAMPS terpolymer was explained as follows. In the salt-water clay suspension, there is present a competition between aggregation and dispersion of the clay particles.<sup>1</sup> Before the smallest value point, the terpolymer adsorbed on the edges (with positive charge sites) of sodium bentonite through carboxyl groups of DIA segments. The steric stabilization between hydrophobic segments of adsorbed terpolymers elevated the gel strength of clay particles, and the aggregation of the clay particles increased the mud viscosity due to the salt screening effect of NaCl on the electric double layer of the clay particles. Hence  $\tau_0$  attained the highest value 4.5 Pa at 0.8% of terpolymer concentration. At the smallest value point, there was enough adsorbed terpolymers with carboxyl groups to disperse clay particles by electrostatic repulsion in the light of DLVO theory, and the rheology properties were improved. After the smallest value point, the larger amount of terpolymer increased the mud viscosity for the reason of the greater opportunity for aggregation

between clay particles. In addition, the salt screening effect decreased with the increase of terpolymer concentration, leading to enhancement of electrostatic stability for the clay particles.<sup>16</sup> Therefore,  $\eta_a$ ,  $\eta_b$ , and  $\tau_0$  increased again.

From Plank's point of view,<sup>10</sup> 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 rheology performance, so thinners might be required for mud formulations. The mud property results confirmed that DIA-AM-SAMPS terpolymer with an average molecular weight of 500,000 was an excellent fluid-loss reducer, and thinner (rheology modifier) was not necessary to add into the mud formulation. The corresponding mud had an excellent tolerance to salt and a high stability to temperature.

#### Particle Size of Mud

The particle size of fresh-water mud containing 4% of the purified sodium bentonite and 0.5% of DIA-AM-SAMPS terpolymer was measured with a Zetasizer 3000. The clay used here was purified bentonite, and the sample concentration was set at 0.1 g/L. Figures 5 and 6 show the measured results of the fresh-water mud before and after the aging test individually. The average size of the mud particle before the aging test was 397.9 nm, the polydispersity index was 0.517, and the



**Figure 6** The particle size distribution of the purified bentonite-terpolymer mud after the aging test.

particle size distribution was wide. The average size of the mud particle after the aging test was 380.6 nm, the polydispersity index was 0.382, and the particle size distribution was relatively narrow compared with that before the aging test.

It is clear that only a small change of the clay particle size occurred before and after the aging test at 220°C, meaning that the clay structure and the interaction of clay particles with terpolymer molecules varied very little under the hostile condition of high temperature. This was described as follows: first, the sulfonic groups in SAMPS segments had strong hydration abilities, the hydrated shell of clay particles was not easy to damage, and this increased the stability of the water phase at high temperature. Second, the carboxyl groups in DIA segments and the sulfonic groups carried negative charges. These negative charges greatly increased the width of the electric double layer around the clay particle according to the DLVO theory, and the electrostatic stability of the mud system was improved.

## CONCLUSIONS

A DIA-AM-SAMPS terpolymer was synthesized through a free-radical polymerization, and characterized using FTIR and TGA methods. The IR spectra of DIA-AM-SAMPS terpolymer confirmed

that there was no olefinic band at 1635–1620  $\text{cm}^{-1}$ . The TGA results revealed that the terpolymer was of higher thermal stability than SAMPS homopolymer.

The terpolymer had strong effects on mud properties. It can be seen that the filtrate volume decreased with the increase of the terpolymer concentration before or after the aging test, and the filtrate volume after the aging test was larger than that before the aging test, but still smaller than 20 mL of fresh-water mud and 50 mL of salt-water mud. In addition, the rheology properties of both fresh-water mud and salt-water mud were improved by DIA-AM-SAMPS terpolymer after the aging test, and  $\eta_a$ ,  $\eta_b$ , and  $\tau_0$  for salt-water mud reached the smallest values at 1.2% of the terpolymer concentration.

The particle size data demonstrated that only a small change in the mud particles occurred before and after the aging test at 220°C, meaning that the clay structure and the interaction of clay particles with terpolymer molecules varied very little under the hostile condition of high temperature. This further confirmed the thermal stability of the terpolymer–clay dispersion from another point of view.

In summary, the DIA-AM-SAMPS terpolymer, with average molecular weight of 500,000, was an excellent fluid-loss reducer under high temperature, and a thinner (rheology modifier) was not

necessary to add into the mud formulation. The corresponding drilling fluid had an excellent tolerance to salt stability to high temperature.

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