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Research Article

Nanoplugging Performance of Hyperbranched Polyamine as Nanoplugging Agent in Oil-Based Drilling Fluid

Gang Xie,[1] Pingya Luo,[1] Mingyi Deng,[1] and Zheng Wang[1,2]

[1] *State Key Laboratory of Oil & Gas Reservoir Geology and Exploitation, Southwest Petroleum University, Chengdu, Sichuan 610500, China*
[2] *Oil and Gas Engineering Institute, Southwest Petroleum University, Chengdu, Sichuan 610500, China*

Correspondence should be addressed to Pingya Luo; luopy@swpu.edu.cn

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A hyperbranched polyamine was synthesized by self-condensing vinyl polymerization with divinyl sulfone, N-phenyl-p-phenylenediamine, by $A_2 + BB_2'$ approach. The hyperbranched polyamine was characterized by FT-IR, TGA, and phase analysis light scanning. Average grain diameter of hyperbranched polyamine was 36.7 nm. Hyperbranched polyamine has good thermal stability. Hyperbranched polyamine (HBPA) was employed successfully as nanoplugging agent in oil-based drilling fluid system, which could plug nanopore formation in shale formation. HBPA has a little effect on rheological properties of oil-based drilling fluid and the FL_{API} and FL_{HTHP} decreased dramatically with an increase of hyperbranched polyamine. Emulsion-breaking voltage has a slight increase, which is beneficial to maintain stability of oil-based drilling fluid. When the HBPA concentration is greater than 1 wt%, plugging rate of oil-based drilling fluid for artificial core is close to 100% and the permeability recovery value can reach 99.7% after adding 1 wt% HBPA, which prove that HBPA has an excellent plugging performance.

1. Introduction

Drilling is linked to drilling fluid that can often cause borehole instability. Borehole instability often leads to various kinds of problems such as hole collapse, tight hole, stuck pipe, bit balling, high torque, and drag, which can increase drilling time and costs [1]. Serious problems mainly occur in shale (principally clay), particularly in water-sensitive shale formations, which account for 75% of all formations drilled by the oil and gas industry [2, 3]. Horizontal well drilling has become the main development of drilling shale formation. In order to prevent the borehole wall collapse of shale gas horizontal well, strong inhibitory oil-based drilling fluid was used. However, oil-based drilling fluid cannot solve the problem of borehole wall instability very well. The essential reason is hydraulic fracturing effect. When oil phase filtrate invades into the cracks, makes the crack open, and greatly reduces the friction of the joint surface at the same time, the collapse pressure rose significantly. If the filtrate cannot be blocked effectively into the cracks, the collapse pressure cannot be prevented. Therefore, whether water-based drilling fluid or oil-based drilling fluid strengthen the blocking that prevent filtrate into the formation of micro-cracks and reduce the formation collapse pressure is the key of drilling shale gas horizontal well, which can improve the pressure-bearing capability of borehole wall and expand the safe density window of drilling fluid. With the combination of nanotechnology and drilling fluid technology, nanomaterials as plugging agent can enhance its plugging effectiveness [4–6]. Nanoplugging particles enter the cracks a certain distance to form internal partition wall and the cracks can be effectively sealed, which prevent drilling fluid filtrate permeate into microcracks of shale wellbore, block the contact of mud filtrate and formation, reduce the formation collapsed pressure, improve wellbore pressure-bearing ability, and expand the safe density window of drilling fluid. In the recent study, fewer studies have focused on 1~100 nm of microfracture plugging. Effective plug for nanopore is the key of wellbore stability in shale formation that has a large number of nanoscale microfracture. Therefore, research of nanosealing materials

can help to study the sealing mechanism and the borehole instability in the process of drilling.

Nanoplugging materials have proven to be an important effect on decreasing the permeability of shale formation, helping to resist fluid invasion and dying out of the shale walls [7, 8]. Compared to the influence of shale permeability before and after nanoparticles were added in the drilling fluid to evaluate the plugging efficiency of nanoparticles, it is found that the blocking performance of nanoparticles is better than conventional polymer [9, 10]. Recently, various types of materials, that is, nanosilica [11], nanographene [12], and cellulose nanoparticle [13], have been applied as high performance nanosealing materials in drilling fluids. For example, it was reported that the cellulose nanoparticles, including cellulose nanocrystals and cellulose nanofibers, showed good rheological properties [14]. When nanoparticles are passed through a low permeability media with nanopores, the nanoparticles have no effective straining [15, 16]. Baker Hughes Corp. had prepared a novel style of nanosized insoluble deformable polymer. This nanometer polymer could infiltrate nanosized pores in shale formation, sedimentate on the surface, and shape low permeable filter cake. In addition, salt resistance was still elevated and was environmentally friendly drilling fluid. As a consequence, the deformable polymer could seal nanosized microfractures and pore throats in shale formation, decrease the permeability of filtrate penetrate into the formations, lower pressure transmission, and block borehole collapse [17]. Blocking materials have a variety of grain sizes for oil-base drilling fluid that need to be optimized. A variety of sealing mechanism can be utilized to block microcracks and make the drilling fluid form a layer of isolation film near borehole wall, enhancing the plugging effect of oil-based drilling fluid and maintaining borehole wall stability, which is expected to solve borehole wall instability problem that use oil-based drilling fluid to drill shale gas horizontal well.

Hyperbranched polymers exhibit three-dimensional dentritic architecture, very low viscosity, high solubility, and plenty of functional groups at the terminal units [18]. An increasing number of studies have a focus on the potential value of hyperbranched polymers in the oil and gas industry [19]. Owing to the three-dimensional dentritic architecture of hyperbranched polymers, hyperbranched polymers were used as nanometer plugging agent such as hyperbranched polyamine which can enhance its plugging effectiveness and hyperbranched polymers had no effect on the drilling fluid rheology. Therefore, hyperbranched polyamine would become a progressing nanometer plugging agent for plugging nanopore in oil-based drilling fluid system. In this paper, the synthesis and properties of hyperbranched polyamine and its nanoplugging properties are described in detail.

2. Materials and Methods

2.1. Materials. Divinyl sulfone come from Chengdu Micxy Chemical Co., Ltd., was used after purification by vacuum distillation. N-Phenyl-p-phenylenediamine come from Chengdu Changzheng Co., Ltd., was used. N,N-Dimethylformamide (DMF) as organic solvents was used. White oil, emulsifier, calcium oxide, fluid loss additive, wetting agent, shear strength improving agent, calcium chloride, and barite are industrial products.

2.2. Synthesis and Characterization of Hyperbranched Polyamine. The Fourier transform infrared (FTIR) spectra of the polymer were determined with Beijing Rayleigh Analytical Instrument Co., Ltd. Thermogravimetric analysis (TGA) was carried out in METTLER TGA/DSC at a nitrogen flow rate of 60 mL/min. The polymer was heated at a heating rate of 10°C/min from 30 to 800°C. The particle size analysis was carried out in Brookhaven ZetaPALS.

2.3. Fluid Preparation and Thermal Aging Tests. The based mud (white oil + 3.0 wt% emulsifier + 2.0 wt% calcium oxide + 2.0 wt% fluid loss additive + 1.0 wt% wetting agent + 1.0 wt% shear strength improving agent + 25 wt% calcium chloride + barite) was made by stirring at a high speed of 10,000 rpm for 20 min to generate homogeneous dispersions. HBPA was added into the based mud, stirring for 20 min at a high speed of 10,000 rpm at room temperature. Thermal aging experiments of based mud HBPA fluid were carried out by GW300 Roller furnace from Qingdao Jiaonan Tongchun Machinery Plant, China, through hot rolling at the appointed temperature 180°C for 16 h. Oil-based drilling fluids property tests were measured before and after thermal aging experiments.

2.4. Fluid Property Tests. The rheological properties were tested with a ZNN-D6 rotating viscometer from Qingdao Jiaonan Tongchun Machinery Petroleum Instrument Ltd., China, at room temperature. Geometry of viscometer spinning rod is shown in Figure 1. The dimensions of viscometer spinning rod are designed strictly according to the standard of American Petroleum Institute (API). Rotating viscometer has six speed changes of 3, 6, 100, 200, 300, and 600 rpm, which correspond to six different shear rates of 5, 10, 170, 340, 511, and 1022 s^{-1}, respectively. Therefore, the range of shear rate is 5~1022 s^{-1}. The probe of rotating viscometer consists of an inner cylinder and an outer cylinder of concentric rotating. When drilling fluid is in the annular space between inner cylinder and outer cylinder, outer cylinder rotates with a constant speed. The outer cylinder produces a torque for inner cylinder through drilling fluid, which causes the inner cylinder connected with torsion spring to rotate a corresponding angle. According to Newton's law, the angle is proportional to the viscosity of liquid. Therefore, the measurement of liquid viscosity becomes the measurement of angle. The angle is reflected in the dial reading. These rheological parameters such as apparent viscosity (AV), plastic viscosity (PV), and yield point (YP) were calculated from 600 to 3 rpm readings through the following formulas from American Petroleum Institute (API) that recommended practice of standard procedure for field testing drilling fluids [20]:

$$\text{apparent viscosity (AV)} = \varphi 600/2 \ (\text{MPa·s});$$
$$\text{plastic viscosity (PV)} = \varphi 600 - \varphi 300 \ (\text{MPa·s});$$
$$\text{yield point (YP)} = (\varphi 300 - \text{PV})/2 \ (\text{N/m}^2).$$

2.5. Synthesis. Ten mmol of N-phenyl-p-phenylenediamine was placed in a 100 mL three-necked round bottom flask.

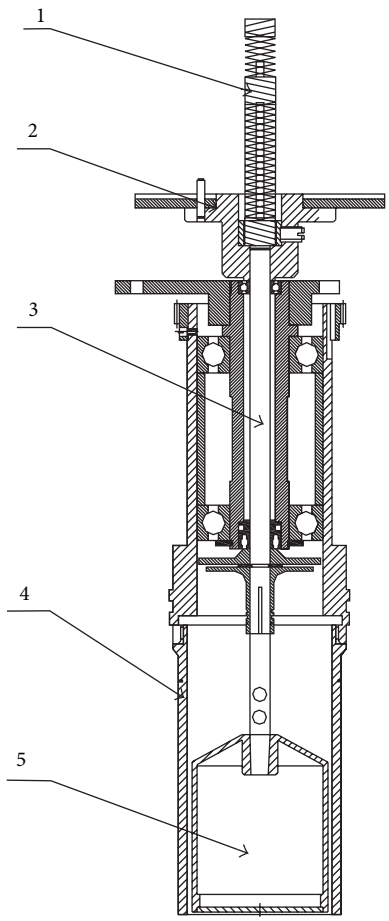

Figure 1: The geometry of viscometer spinning rod. Note: 1: spring assembly; 2: dial components; 3: inner tank shaft; 4: outer cylinder; 5: inner cylinder.

When the reaction mixture was totally dissolved in 20 mL of N,N-dimethylformamide, 20 mmol of divinyl sulfone was slowly added under N_2 purge and then bubbled into nitrogen for 10 min. The mixture was heated to 50°C for 3 h. The reaction mixture was slowly poured into 500 mL of methanol and then the precipitate was collected. The precipitate was purified by reprecipitation with 500 mL of methanol. The product was dried under vacuum at 65°C for 24 h. The powdery product was obtained.

3. Results and Discussion

3.1. Synthesis of Hyperbranched Polyamine. Hyperbranched polyamine (HBPA) as nanoplugging agent was synthesized by using an A_2 + BB_2' method (Figure 2). A_2 monomer is divinyl sulfone. BB_2' monomer is N-phenyl-p-phenylenediamine for this reaction. Vinyl groups of divinyl sulfone react rapidly with secondary amino groups of N-phenyl-p-phenylenediamine compared to vinyl groups of divinyl sulfone with primary amino groups of N-phenyl-p-phenylenediamine, generating AB_2' monomers. AB_2' type monomers as the intermediate were polymerized further, which generate branched polyamine [21]. This excellent choice of reaction conditions such as proper ratio, appropriate concentration, and slowly adding A_2 monomers could successfully avoid gelation. The mole ratio of A_2 and BB_2' monomers was 2 : 1 and the A_2 monomer was added slowly into a dilute solution (<10%, w/v) of BB_2' the monomer in DMF solvent. The yield of hyperbranched polyamine was greater than 84%.

3.2. Characterization of Hyperbranched Polyamine. The FT-IR spectrum (Figure 3) of the polymer shows various peaks to the ideal structure of the copolymer. 3034 cm^{-1} is benzene ring=CH stretching vibration absorption peak. 1597 cm^{-1} is benzene ring C=C stretching vibration absorption peak. 838 cm^{-1} is characteristic absorption peak of 1, 4-substituted benzene. 692 cm^{-1} is characteristic absorption peak of single substituted benzene, which proves that nanometer blocking agent polymer has benzene ring. 1327 cm^{-1} is O=S=O asymmetric stretching vibration peak and 1121 cm^{-1} is O=S=O symmetrical stretching vibration peak [22, 23]. 3465 cm^{-1} and 3378 cm^{-1} are stretching vibration absorption peaks of aromatic primary amine. 1298 cm^{-1} is stretching vibration absorption peak of aromatic amine C-N and 1597 cm^{-1} is the deformation vibration peak of primary amines. These prove that plugging agent was synthesized by N-phenyl-p-phenylenediamine and divinyl sulfone. Thermogravimetric analysis curve shows that benzene ring enhances the rigidity of polymer chains and weakens the thermal motion of polymer chains. So polymer has good thermal stability.

3.3. Thermostability of Hyperbranched Polyamine. Hyperbranched polyamine has a high solubility in oil. Figure 4 is the thermogravimetric curve of hyperbranched polyamine. The weight loss of hyperbranched polyamine about 0.95% at 209°C was owing to the loss of absorbed water or crystalliferous water. The degradation of hyperbranched polyamine started at about 299.13°C and happened in the first step. There was no obvious thermal decomposition when the temperature was under 299.13°C. Benzene ring has a good thermal stability due to its domain structure. Compared to common alkyl carbon chain of prepared polymers, the c-c bond of benzene ring has an extreme rigidity and thermal motion of the molecular chain will be blocked under an environment of high temperature, which would improve the high-temperature resistance of polymer. With the increase of temperature, the weight of hyperbranched polyamine started to decrease due to the decomposition of the polymer. Almost 72.31% weight losses have occurred at 489.7°C. It was demonstrated that hyperbranched polyamine has a strong temperature-resistance and its decomposition temperature is higher than 299°C.

3.4. Grain Size Distribution. Recently, a more sensitive technique, phase analysis light scattering (PALS), has been developed with particular benefits for application to nonpolar systems [24]. The technique, phase analysis light scattering (PALS), is based upon classical laser-Doppler electrophoresis but employs signal processing of the time domain phase information within the scattered light signal, rather than analysis of its frequency spectrum [25]. Compared with the

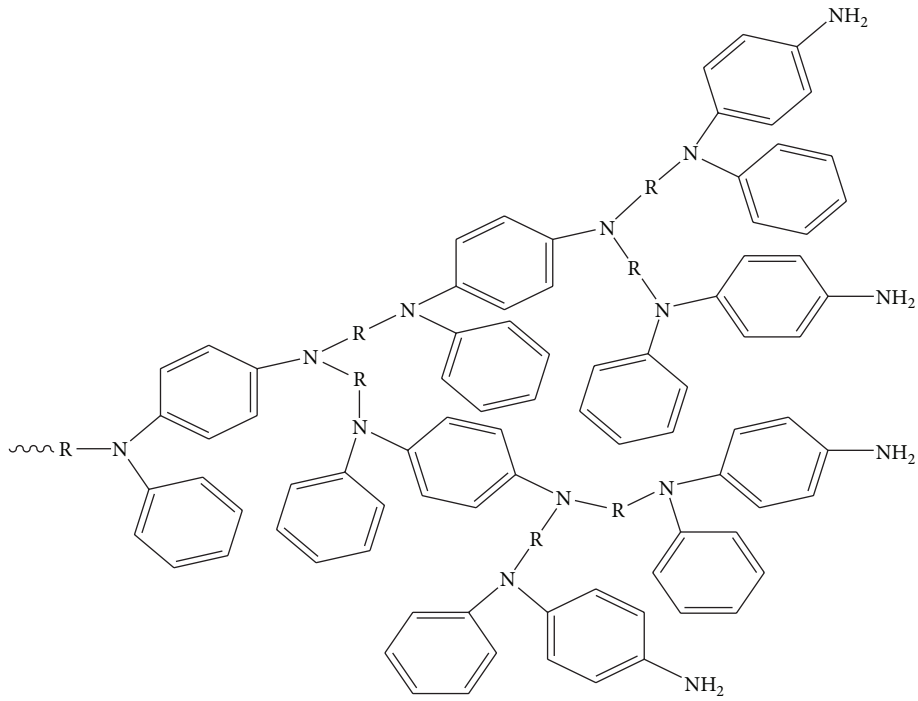

Figure 2: Chemical structure of hyperbranched polyamine.

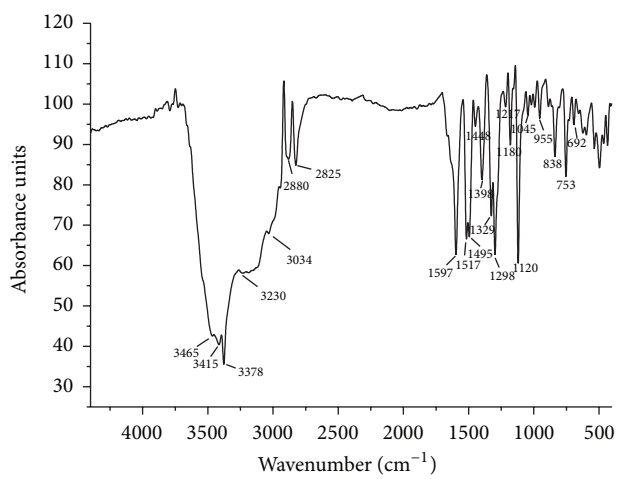

Figure 3: Fourier transform infrared (FTIR) spectroscopy of hyperbranched polyamine.

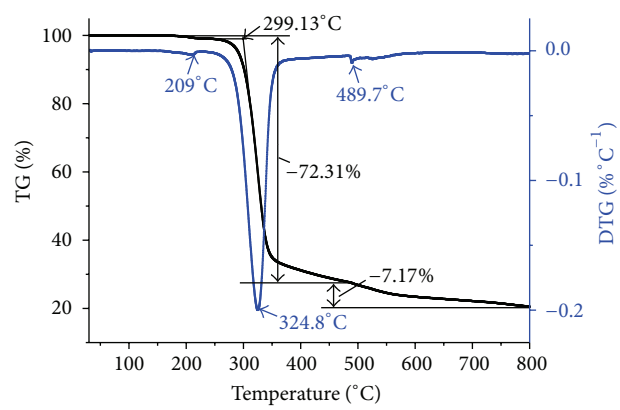

Figure 4: Thermogravimetric analysis of hyperbranched polyamine.

traditional light scattering method based on the technology of frequency shift, the sensitivity of phase analysis light scattering can be enhanced by 1000 times. Firstly, 1 wt% HBPA is dissolved in normal hexane. Then HBPA solution is collected in the cuvette through 450 nm filters. Next, the cuvette is placed in the pool of the measurement. Finally, choose normal hexane as measuring media and start measuring. The particle size of nanoblocking polymer is determined by the ZetaPALS laser particle size instrument of Brookhaven, which is shown in Figure 5. Figure 5 shows that the grain size distribution of the polymer is more concentrated and the curve of grain size distribution presents parabolic type. The grain size distribution is between 3~350 nm and the average grain diameter is 36.7 nm. The grain size of polymer particles smaller than 100 nm accounted for 80%, which can block the microfracture of different nanoscale.

3.5. Plugging Effect Evaluation

3.5.1. Rheological and Filtrate Properties of HBPA in the Oil-Based Drilling Fluid. The change of dial readings with different concentration of HBPA in different shear rate before and

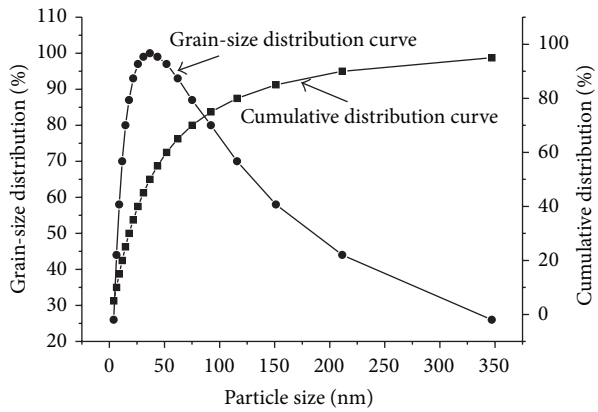

Figure 5: The grain size distribution and cumulative distribution of HBPA as nanoplugging polymer.

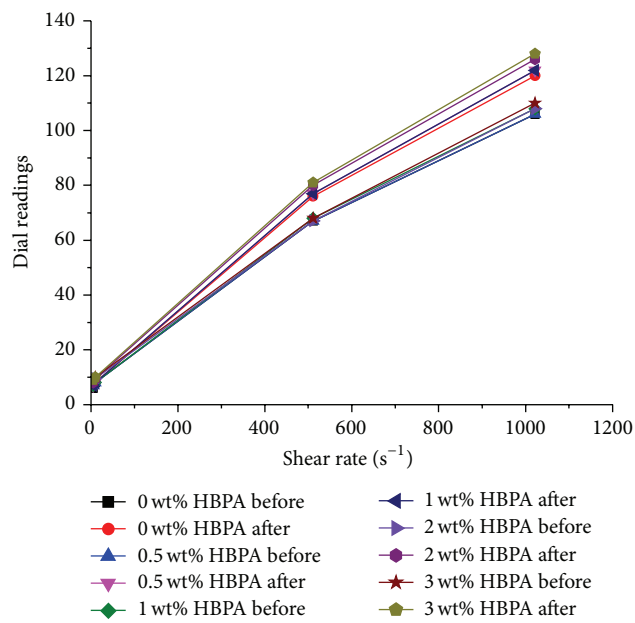

Figure 6: The change of dial readings with different concentration of HBPA in different shear rate before and after the thermal aging tests at 180°C for 16 h.

after the thermal aging tests at 180°C for 16 h was shown in Figure 6. With the increase of HBPA, dial reading of drilling fluid is only increased a little before the thermal aging tests at 180°C for 16 h; that is, drilling fluid viscosity has a little change, which indicated that the HBPA has a little impact on the rheological properties of drilling fluid. Compared with the dial readings of drilling fluid without HBPA after the thermal aging tests at 180°C for 16 h, when HBPA is less than 1 wt%, dial reading has only a little increase with the increase of HBPA; that is, drilling fluid viscosity has almost no change. When HBPA is greater than 1 wt%, the dial reading increases slightly; that is, drilling fluid viscosity increased slightly. The results fully embody the physical chemistry properties of low viscosity of hyperbranched polymer and have a little impact on the rheological properties of drilling fluid before and after the thermal aging tests at 180°C for 16 h.

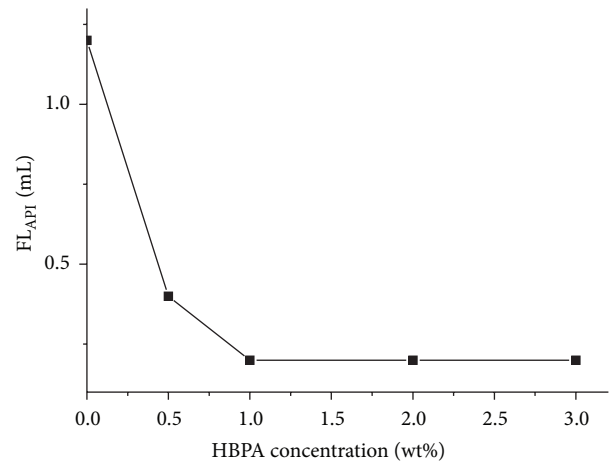

Figure 7: The effect of HBPA concentration on the API filtrate volume (FL_{API}) after thermal aging tests at 180°C for 16 h.

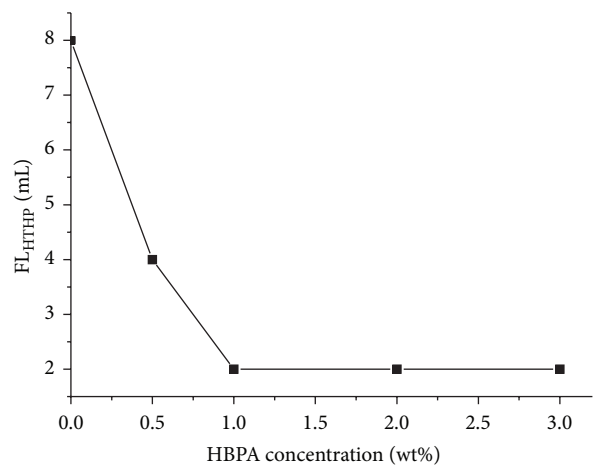

Figure 8: The effect of HBPA concentration on the FL_{HTHP} after thermal aging tests at 180°C for 16 h.

Rheological and filtrate properties of oil-based drilling fluid with different concentrations of HBPA before and after hot rolling test at 180°C for 16 h are shown in Table 1. Before and after hot rolling test, all the rheological data, including AV, PV, and YP, have almost a very small increase. Emulsion-breaking voltage has a little increase, which is beneficial to maintain stability of oil-based drilling fluid. It can be inferred that HBPA has little effect on the rheological properties of oil-based drilling fluid because of hyperbranched polymers having very low viscosity. The filtrate volume of FL_{API} and FL_{HTHP} after the thermal aging tests at 180°C for 16 h was shown in Figures 7 and 8, respectively. It showed that HBPA can decrease dramatically the FL_{API} and FL_{HTHP} with the increase of HBPA concentration after thermal aging tests. When the dosage of HBPA is greater than 1 wt%, the filtrate volume of FL_{HTHP} has not changed. Therefore, the optimum added amount of HBPA is 1 wt%.

3.5.2. Plugging Effect Evaluation. Kumar uses kind of gradually narrow wedge grooves to simulate the formation fracture.

Table 1: Rheological behaviors of oil-based drilling fluid under different HBPA concentrations (before and after the thermal aging tests at 180°C for 16 h).

HBPA concentration (wt%)	AV (MPa·s)		PV (MPa·s)		YP (Pa)		GEL (Pa/Pa)		ES (V)	
	Before	After	Before	After	Before	After	Before	After	Before	After
0	53	60	39	44	14	16	4/8	4/9	1255	1345
0.5	53	61	39	45	14	16	4/9	5/10	1258	1355
1	54	61	40	45	14	16	4/9	5/10	1262	1364
2	54	63	41	46	13	17	4/10	5/11	1268	1372
3	55	64	42	47	13	17	4/10	5/11	1275	1380

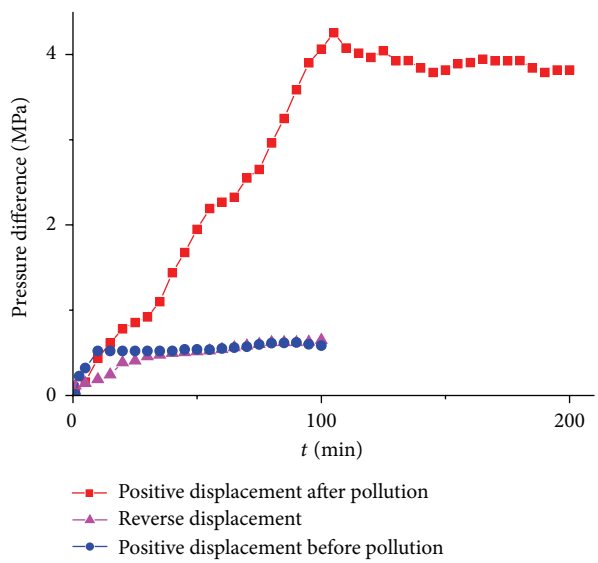

Figure 9: The displacement pressure curve of oil-based drilling fluid for artificial core without HBPA.

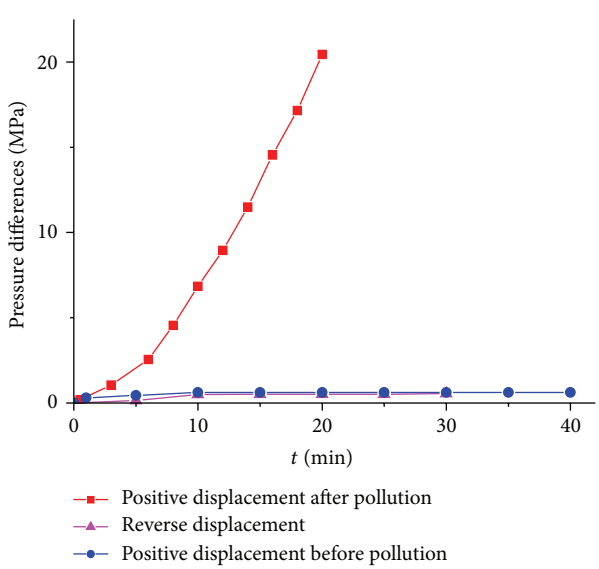

Figure 10: The displacement pressure curve of oil-based drilling fluid for artificial core with 1 wt% HBPA.

Pressure conditions and plugging performance of the fiber and solid particles for fracture were evaluated [26, 27]. The groove diameter is 1000~2500 μm and the width of another metal cutting board is tens of microns. However, microcracks and pore in shale formation are usually nanoscale. The above two kinds of models are not suitable for the simulation of shale microcracks. Therefore, artificial core with a low permeability of $10 \times 10^{-3}\ \mu m^2$ was utilized to simulate the formation fracture. Permeability and displacement pressure curve of artificial core were determined by permeability test equipment and pressure sensor, respectively. The displacement pressure curve was illustrated in Figures 9 and 10. Figure 9 is the displacement pressure curve of oil-based drilling fluid without HBPA. Figure 9 shows that the pressure peak of positive displacement is only 0.62 MPa before pollution by oil-based drilling fluid without HBPA. The pressure peak of positive displacement is about 4.05 MPa and the pressure peak of reverse displacement is about 0.65 MPa after pollution by oil-based drilling fluid without HBPA. Figure 10 is the displacement pressure curve of oil-based drilling fluid with 1 wt% HBPA. Figure 10 shows that the pressure peak of positive displacement is only 0.62 MPa before pollution by oil-based drilling fluid with 1 wt% HBPA. The pressure peak of positive displacement is about 20.45 MPa and the pressure peak of reverse displacement is about 0.55 MPa after pollution by oil-based drilling fluid with 1 wt% HBPA. Plugging rate of oil-based drilling fluid through formulas (1) and (2) is close to 100% after adding 1 wt% HBPA. The permeability recovery value can reach 99.7%, which prove that HBPA has an excellent plugging performance:

$$K = \frac{h\mu Q_t}{A\Delta P}. \tag{1}$$

K is the permeability of artificial core, $10^{-5}\ \mu m^2$. A is the cross-sectional area of artificial core, cm^2. ΔP is the differential pressure, MPa. h is the core length, cm. μ is the viscosity of oil-based drilling fluid, MPa·s. Q_t is the average flow rate, $cm^3 \cdot s^{-1}$. Consider the following:

$$W = \frac{(K_1 - K_2)}{K_1} \times 100\%. \tag{2}$$

W is the plugging rate of oil-based drilling fluid, 1. K_1 is the permeability of artificial core after pollution by oil-based drilling fluid without HBPA. K_2 is the permeability of

artificial core after pollution by oil-based drilling fluid with 1 wt% HBPA.

4. Conclusion

The hyperbranched polyamine was synthesized by self-condensing vinyl polymerization with divinyl sulfone, N-phenyl-p-phenylenediamine, by an $A_2 + BB_2'$ method. The hyperbranched polyamine was characterized by FT-IR, phase analysis light scanning, and TGA methods. Hyperbranched polyamine as nanoplugging agent was successfully applied in the oil-based drilling fluid system.

Grain size distribution of HBPA is in the range of 3~350 nm and the average grain diameter is 36.7 nm by phase analysis light scattering. The grain size of polymer particles smaller than 100 nm accounted for 80% and the grain size distribution is wide, which can block the microfracture of different nanoscale. HBPA has a good thermal stability and the decomposition temperature is greater than 299.

Artificial core with a low permeability was utilized to simulate the shale formation. When hyperbranched polyamine was added into the oil-based drilling fluid system, the permeability of artificial core declines sharply with the increase of concentration. When the HBPA concentration is greater than 1 wt%, the plugging effect of HBPA is the best and the emulsion-breaking voltage has a slight increase. HBPA has a negligible effect on rheological properties of oil-based drilling fluid and decreases dramatically the FL_{API} and FL_{HTHP}. In conclusion, hyperbranched polyamine was an excellent nanoplugging agent, which was expected to solve the problem of shale borehole wall instability.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

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