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Grafting lignite with sulformethal phenoldehy resin and their performance in controlling rheological and filtration properties of water–bentonite suspensions at high temperatures

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ABSTRACT

A lignite graft polycondensate with water retention and dispersion properties in a water–bentonite slurry was synthesized by grafting sulformethal phenoldehy (SMP) resin onto a scaffold of humic acid (main component of lignite) through aqueous condensation polymerization. Characteristic results of the chemical structure and molecular weight of the polycondensate showed a successful grafting of the SMP. The resulting lignite graft polycondensate used as drilling fluid additive effectively controlled the filtrate volume and exhibited rheological stability at aging temperatures as high as 200 °C. The behavior of the polycondensate distinguishes it from most common synthetic high-temperature fluid loss additives, such as SMP, which excessively viscosify slurries. Colloidal properties of the water–bentonite admixture were investigated by adsorption and zeta potential experiments, and a possible mechanism was proposed.

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1. Introduction

Drilling fluids are important for oil well drilling because of their various functions, such as transporting rock cuttings to the surface, lubricating drill bits, applying hydrostatic pressure to well bores to ensure well safety, and minimizing fluid loss across permeable formations by forming a filter cake on the walls of well bores (Gray et al., 1980; Chiligarian and Vorabutr, 1983). As such, drilling fluids must have appropriate viscosity and low water loss from slurry, particularly for deep wells where high temperature and pressure are encountered. Drilling fluids require chemical additives to optimize the properties of water–bentonite slurry. Otherwise, bentonite gels are developed, which can result in the loss of filtration control and cause formation damage that can be detrimental to the drilling activity and later production (Ghalambor and Economides, 2002; Meeten and Sherwood, 1994).

Fluid loss additives (FLAs) are one of the most important drilling fluid chemicals used to prevent the dehydration of slurries. Currently, synthetic sulfonated polymers are extensively used as high-temperature FLAs. Such polymers include sulformethal phenoldehy (SMP) resin or polymers based on 2-acrylamido-2-methylpropane sulfonic acid (AMPS), *N*-vinylacetamide and

acrylamide or AMPS, and *N,N*-dimethylacrylamide (NNDMA) (Persinski and Cool, 1977; Engelhardt et al., 1982; Rao and Burkhalter, 1985; Stephens, 1994; Udarbe and Hancock-Grossi, 2000). The effectiveness of these polymers relies on the adsorption on the cement surface (Plank et al., 2006). A major drawback of these high-molecular-weight copolymers is their excessive viscosifying effect. Lignite has been used successfully as a thinner at temperatures lower than 180 °C (Kelessidis et al., 2007). However, lignite (generally causticized) itself is insufficient to control fluid loss. Complexes of lignite and lignosulfonates with specific metals, particularly Cr³⁺ and Cr⁶⁺, have been used as additives for fluid loss control. The suspensions have been proven to be highly thermally stable (Gray et al., 1980; Clark, 1994). However, complexes with Cr⁶⁺ can seriously contaminate the environment.

Lignite cannot sufficiently control fluid loss at high temperature. However, it has excellent dispersing and thinning properties (Kelessidis et al., 2009) in slurry, properties that have attracted the attention of researchers. Recently, Salami and Plank (2013) prepared a dispersing graft copolymer by grafting AMPS, NNDMA and acrylic acid onto humic acid using free radical polymerization. They reported that the prepared copolymers can effectively control the fluid loss of cement slurry with low viscosity up to 200 °C. Humic acid is the main component of lignite. It is a polycyclic aromatic hydrocarbon with many active groups, such as hydroxyl, carboxylic and phenolic groups (Gerasimowicz and Byler, 1985; Inbar et al., 1989). Although humic acid has numerous active

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groups, grafting humic acid through free radical polymerization is still difficult unless an exquisite method is designed. However, based on the chemical structure of humic acid, obtaining grafted lignite by using formaldehyde and phenol by the polycondensation method is easier. The preparation process is relatively simple and economical.

This paper reports the use of lignite graft polycondensate as drilling fluid additive in water–bentonite suspensions to control filtration and rheological properties at high temperature. The highly anionic SMP blocks were grafted onto the humic acid scaffold with a large amount of sulformethyl groups that ionize completely in aqueous solution. The phenol monomer renders the polymer chain more stable, resulting in high temperature resistance. Our tests showed that this polymer has excellent temperature stability up to 200 °C. The influence of the polymer on the flow behavior of water–bentonite slurry was established. In addition, the working mechanism of the polymer was verified by environmental scanning electron microscopy (ESEM) characterization and adsorption and zeta potential measurements.

2. Experimental

2.1. Materials

Phenol, formaldehyde, sodium hydroxide, sodium pyrosulfite and sodium carbonate were of analytical grade. Sodium bentonite and SMP were commercial products from Drilling Mud Ltd. Co. of Shengli Oil Field.

Lignite was obtained from Wuhai, Inner Mongolia, China. The moisture of lignite was determined after heating at 105 °C for 2 h. Ash content was determined after heating at 850 °C for 2 h. The humic acid, fulvic acid and humin contents of lignites were determined using the methods described by Swift (1996). The results are listed in Table 1.

2.2. Preparation and characterization of the lignite graft polycondensate

The pretreatment of lignite samples involved breaking up the lignite particles to sizes less than 150 μm and drying at 105 °C. The lignite graft polycondensate was prepared by grafting SMP onto the humic acid scaffold via polycondensation method. In a typical experiment, 50 g of lignite and 200 mL of 0.3 M NaOH solution were placed in a 500 mL three-necked flask equipped with a stirrer and a thermometer. The lignite was causticized at 80 °C for 2 h, and the temperature was cooled down to 60 °C. Then, 20 g of phenol, 20 mL of formaldehyde solution (37 wt%), and 12 g of sodium pyrosulfite were added to the causticized lignite solution. The weight ratio between the lignite and the graft chain was approximately 50:40. After 2 h of reaction time at 60 °C, the temperature was increased to 90 °C for another 2 h to complete the reaction. Finally, the product was obtained by drying at 105 °C for 12 h.

Prior to obtaining the Fourier transform infrared (FTIR) spectra, the samples were treated by the dissolution-acid out process to remove ungrafted impurities. The FTIR spectra (4000–400 cm⁻¹)

were obtained by using the IRPrestige-21-type FTIR spectrophotometer (Shimadzu Corporation, Japan). Molecular weight distribution of the causticized lignite and grafted lignite were determined by the DAWN HELEOS-II GPC (WyATT Technologies, USA) equipped with the Shodex P85140803 column (Japan). Aqueous 0.1 M NaNO₃ solution was used as eluent (flow rate of 0.5 mL/min). A d_n/d_c value of 0.133 mL/g was obtained based on the refractive index of various contents of lignite graft polycondensate aqueous solution, and then used to calculate M_w and M_n .

2.3. Fluid preparation and aging tests

The freshwater-based fluid was prepared as followed: solve 32 g sodium bentonite and 0.8 g Na₂CO₃ into 400 ml of deionized water, and stir for 20 min at a high speed of 10,000 rpm. The suspension was left in a sealed container for 16 h at room temperature (RT) for following use. Samples were added to this suspension at various concentrations of ~3.0% w/v. In a typical experiment, 7.2 g lignite graft polycondensate was added into 400 ml of freshwater-based fluid, then the suspension was stirred for 20 min at a high speed of 10,000 rpm, producing the graft lignite fluid with the dosage of 1.8% w/v. Static thermal aging experiments were conducted in a sealed container at different temperatures (150, 180, 200, and 220 °C) for 16 h and the dosage of sample is 1.8% w/v. After 16 h of aging, the slurries were stirred for 5 min at a high speed of 10,000 rpm prior to the fluid loss property tests.

2.4. Fluid loss property tests

Fluid loss property tests include the American Petroleum Institute (API) filtrate volume (FL_{API}) and high-temperature and high-pressure filtrate volume (FL_{HTHP}) measurements. FL_{API} and FL_{HTHP} were measured based on the API specifications 13 A. FL_{API} of the slurry was determined by using the ZNZ-D3-type filtration apparatus (Qingdao Haitongda Special Instrument Co. Ltd., China) under a pressure of 0.7 MPa. FL_{HTHP} was determined by using the GGS71-B-type high-temperature and high-pressure filtration apparatus (Qingdao Haitongda Special Instrument Co. Ltd., China) with a pressure of 3.5 MPa. FL_{API} and FL_{HTHP} were recorded for over 30 min.

Rheological data were determined by using the ZNN-D6-type six-speed rotating viscometer (Qingdao Haitongda Special Instrument Co. Ltd., China) at RT. The rotational speeds were 3, 6, 100, 200, 300 and 600 rpm for the shear rates of 5, 10, 170, 340, 511 and 1022 s⁻¹, respectively. The rheological parameters, such as shear stress (τ), apparent viscosity (AV), plastic viscosity (PV) and yield point (YP) were described as follows:

$$\begin{aligned} \tau &= 0.511\theta; \text{ AV} = \theta_{600}/2(\text{mPa s}); \text{ PV} = \theta_{600} - \theta_{300}(\text{mPa s}); \text{ YP} \\ &= 0.511(\theta_{300} - \text{PV})(\text{Pa}) \end{aligned}$$

where θ is the dial reading of the instrument (3.68×10^{-5} N m per degree) at the indicated rotational speeds (rpm).

We collected the filter cakes from the FL_{API} test of filter cake permeability. The filter cakes were immediately freeze dried, and then investigated by environmental scanning electron microscope (Quanta 200, FEI, Holland).

2.5. Adsorption and zeta potential measurements

The adsorbed amount was calculated from the difference of additives in the equilibrium concentrations in the liquid phase before and after adsorption. The concentration of additives in the filtrate from FL_{API} measurement was determined by using the TOC-LCPH analyzer (Shimadzu Corporation, Japan). The TOC-LCPH analyzer is equipped with a CO₂ detector for the quantification of

Table 1
Organic and inorganic components of lignite samples.

Sample	Moisture (%)	Ash (% dry lignite) ^a	Humin (% dry lignite) ^a	Humic and fulvic acids (% dry lignite) ^a
Lignite	39.2	32.4	25.1	42.5

^a Dry lignite is obtained by heating lignite at 105 °C for 2 h.

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