



Inhibiting shale hydration and dispersion with amine-terminated polyamidoamine dendrimers



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ABSTRACT

Dendrimers have been paid increasing attention in the area of drilling fluid recently due to their unique structure and properties. The inhibitive properties of amine terminated polyamidoamine (PAMAM) dendrimers ranging from G0 to G5 were evaluated by relative inhibition rate, shale cuttings hot-rolling dispersion test and particle size distribution measurement respectively. Meanwhile the inhibitive capacity of the polymers under various pH value conditions was studied. The interaction between sodium bentonite and PAMAM dendrimers was characterized by zeta potential measurement and X-ray diffraction (XRD). The results indicated that, G0 and G5 are superior to polyether diamine, while G1, G2, G3 and G4 perform better than KCl. PAMAM dendrimers from G0 to G5 can decrease the zeta potential of clay particles from -39 mV to about -22.6 mV at natural pH values and reverse the charge when the pH value is adjusted to 9. When intercalated into the clay interlayer in aqueous solution, the candidate PAMAM dendrimers can form monolayer arrangement at lower concentrations and reduce the interlayer spacing of hydrated clay from 1.95 nm to 1.38–1.63 nm, which is the direct evidence of inhibiting shale hydration. With the increase of concentration, mixed phase and bilayer orientation are formed in the interlayer. The multi-terminal amine groups of dendrimers can effectively bind the adjacent layers together with electrostatic attraction and hydrogen bonding, and expel the water molecules out of the interlayer, contributing to the suppression of clay swelling and dispersion. The pH value reduction of aqueous solution improves the protonation of terminal-amine group, which can intensify the interaction between the polymers and clay, inducing the decrease of hydrated interlayer spacing and the zeta potential of clay particles, thus enhancing the inhibitive properties.

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1. Introduction

It is always a worldwide challenge for well drilling in shale formations due to the wellbore instability problems induced by the interaction between shale and aqueous filtration including tight hole, sloughing, washout, caving and so on (Al-arfaj et al., 2014; Stelger and Leung, 1998). Oil-based drilling fluids are the first choice for most shale formations because of the outstanding shale stability, however, the drawback of high cost and stringent environmental legislation make them a much less attractive alternative than before (He et al., 2014; Stamatakis et al., 1995; Suter et al., 2011). Consequently, much research has been transferred to improve the inhibitive properties of water-based drilling fluids with the eventual goal of replacing oil-based drilling fluids, which

is also a hallmark of high performance water-based drilling fluid (Galindo et al., 2015).

Based on the understandings of shale hydration mechanisms and the physical–chemical interactions between shale and drilling fluid, lots of work has been carried out to evaluate and seek solutions to mitigate and control the reactive shale formations during the past decades (Van Oort et al., 1994; Van Oort, 1997). Numerous chemicals including inorganic salts, formates, organic amine compounds, polymers, silicates and polyglycerols have been used with different mechanisms such as hydration inhibition, reduction of pore pressure transmission, or cuttings encapsulation (Galindo et al., 2015; Patel et al., 2007; Reid et al., 1995; Van Oort, 2003; Xuan et al., 2013; Zhang et al., 2001). Based on these chemical inhibitors, several inhibitive water-based drilling fluids have been established and utilized in the field (Donham and Young, 2009; Patel and Gomez, 2013; Stamatakis et al., 1995; Young and Ramses, 2006). Nevertheless, it is not completely successful for these fluids to inhibit the hydration and dispersion of reactive

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shales with various performance limitations (Patel et al., 2002). Therefore, developing new highly inhibitive and environmentally friendly shale inhibitors is still an ongoing endeavor and research hotspot.

Dendrimers have been paid extensive attention since the first synthesis because of their unique characteristics such as highly ordered structure, narrow size distribution, and uniform dense terminal functional groups, which render the polymers as ideal candidates in a wide range of applications (Abdel Rehim et al., 2010; Mo et al., 2008). Recent studies have shown that dendrimers have potential application in the area of oilfield chemistry (Amanullah and Aramco, 2013; Kaiser, 2013; Lai et al., 2013; Li et al., 2012; Rivers et al., 2011; Stanciu et al., 2015). As far as drilling fluid is concerned, Amanullah and Aramco (2013) illustrated a promising potential of developing high performance drilling fluid in future challenging conditions with the use of dendrimers. Miller (2011) applied a patent that poly(propyleneimine) dendrimers and polyamine twin dendrimers can be used as shale hydration inhibition agent and high temperature resistant to 500 °F. Teixeira et al. (2014) synthesized hyperbranched polyglycerols and evaluated their inhibitive performance. The results showed that the polymer has a great potential to be environmentally friendly inhibitor in water-based drilling fluid. In a recent study, elaborated dendritic polymers were designed and synthesized to reduce the fluid loss with temperature resistance of 260 °C in water based drilling fluid (Zhang et al., 2015).

PAMAM dendrimers, first synthesized by the Tomalia et al. (1985), are one of the most widely used dendrimers (Gillies and Frechet, 2005). According to the molecular design rule of the clay swelling inhibitor (Suter et al., 2011), the unique characteristics including high density of terminal amine group and ellipsoidal or spheroidal structure of PAMAM dendrimers make them suitable for shale inhibition. The feasibility of PAMAM dendrimers as shale stabilizers has been investigated in our previous work (Zhong et al., 2015). The purpose of this paper is to further study the effect of PAMAM dendrimers on shale hydration and dispersion suppression under different pH values, and establish a relationship between the molecular structure of PAMAM dendrimers and stabilization of reactive shales.

2. Experimental

2.1. Materials

Different generations (from G0 to G5) of amine terminated polyamidoamine (PAMAM) dendrimers with ethylene diamine as initial core, were purchased from Sigma–Aldrich (Fig. 1). Polyether diamine (PEA) with molecular weight of 230 was provided by BASF Chemical Co. Potassium chloride was purchased from Sinopharm Chemical Reagent Co., Ltd, China with analytical purity. Sodium bentonite was provided by Boyou bentonite Group Co., Ltd, China, and the detailed mineralogical composition was presented in mass percentage as follows: montmorillonite, 57.6%, illite, 1.2%, kaolinite, 0.6%, chlorite, 0.6%, quartz, 32%, potassium feldspar, 8%. The cation exchange capacity (CEC) was determined to be 74 mmol/g. Drilling fluid bentonite was obtained from Weifang Huawei Bentonite Group Co., Ltd, China, following the American Petroleum Institute (API) standard. All of the chemicals were used as received without further purification.

The shale cuttings used for shale dispersion tests were provided by Zhudong Drilling Company from Well Yueman 6 of Tahe oilfield. The detailed mineralogical composition of the shale cuttings was identified using X-ray diffraction (XRD) and shown as follows, quartz, 41%, K-feldspar, 5%, plagioclase, 15%, calcite, 17%, iron dolomite, 4%, kaolinite, 1%, chlorite, 1%, illite, 7%, interlayered illite-

smectite, 9%.

2.2. Methods

2.2.1. Relative inhibition rate

Inhibitor solution containing 350 mL fresh water and 3.5 g inhibitor sample was prepared. Drilling fluid bentonite (35 g) was incorporated into the solution and stirred at 10,000 rpm for 20 min. Then the dispersion was hot-rolled at 120 °C for 16 h. After cooling to ambient temperature, the rheological properties of the dispersion were determined by ZNN-D6 rotational viscometer (Qingdao Haitongda Special Instrument Co., Ltd). The relative inhibition rate was calculated according to the following equation.

$$R = \frac{\phi_{100} - \phi_{100'}}{\phi_{100}} \times 100\% \quad (1)$$

where, R is relative inhibition rate, ϕ_{100} and ϕ_{100}' are 100 dial reading of control sample and inhibitor sample respectively.

2.2.2. Shale cuttings hot-rolling dispersion test

This test was designed to evaluate the dispersion of shale cuttings after exposing to a formulated fluid in a rolling oven cell at setting temperatures. In this test, mass of 40 g shale cuttings from Well Yueman-6 and 280 mL aqueous solution containing 2 w/v% inhibitor samples were added to the roller oven cell. After hot rolling at 77 °C for 16 h, the fluid and shale were cooled to ambient temperature and screened with 40 mesh sieve. The remaining shale cuttings were washed and recovered. After dried at 105 °C for 4 h, the recovered shale cuttings were weighed and the percentage recovery of the shale cuttings was determined.

2.2.3. Particle size distribution measurement

Particle size distribution measurement can be used to assess the inhibition ability of shale inhibitors (Liu et al., 2004). Mass of 16 g sodium bentonite was added into 400 mL deionized water and stirred vigorously for 1 h. Then the dispersion was incubated for at least 24 h. After that, 4 g shale inhibitors were added into the dispersions and stirred for 24 h. The particle size distribution of the dispersion was measured by Bettersize 2000 particle size analyzer (Bettersize instruments Ltd., China).

2.2.4. Zeta potential measurement

Zeta potential was measured with a Zetasizer Nano ZS90 (Malvern instrument, United Kingdom). Inhibitor solution containing 1 g inhibitor and 97 mL deionized water was prepared. Then 3 g Boyou sodium bentonite was added into the solution and vigorously stirred for 24 h. Before the measurement, the dispersion was diluted to make the particles visible under the microscope. The zeta potential of each sample was tested three times and their average was taken as the final result. After adjusting the pH value to about 9 with hydrochloric acid, the pH value of the dispersion was measured with pH meter and the related zeta potential was derived.

2.2.5. Measurement of interlayer spacing with X-ray diffraction (XRD)

Firstly, sodium bentonite dispersion containing 7 g sodium bentonite and 350 mL deionized water was prepared first. Then PAMAM dendrimers were added into the dispersion with the content of 0.35, 0.70, 1.75, 2.80 and 7.00 g respectively. The dispersion was stirred for another 24 h to establish adsorption equilibrium. The pH value of the dispersion was adjusted by 0.1 mol/L hydrochloric acid. Then the mixture was centrifuged at 8000 r/min for 20 min and washed three times to remove the un-

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