



# Synergistic stabilization of shale by a mixture of polyamidoamine dendrimers modified bentonite with various generations in water-based drilling fluid



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## ABSTRACT

The amine terminated polyamidoamine (PAMAM) dendrimers were investigated as potential shale stabilizers for the first time. Its inhibitive properties were characterized by a bentonite inhibition test and a shale cuttings dispersion test. The interaction between PAMAM dendrimers of various generations and sodium bentonite was examined by X-ray diffraction (XRD), Fourier transform infrared (FT-IR) spectroscopy, transmission electron microscopy (TEM) and thermogravimetric analysis (TGA). The hydration and dispersion of shale can be effectively inhibited with PAMAM dendrimers. The decrease of pH value in the dendrimer aqueous solution improved the inhibitive performance. For lower generations of dendrimer growth (G0 to G3), the inhibitive level decreased with the increase of generation. For higher generations (G4, G5), the inhibitive level increased with the increase of generation. Low generation PAMAM dendrimers could penetrate into the clay interlayer space, but exhibited highly oblate intercalation behaviors. G0 intercalated into the interlayer space with a monolayer conformation as concentration increased, while G1, G2, G3 and G4 intercalated into the interlayer space with monolayer arrangement at low concentrations, and mixed phase orientation at high concentrations. G5 could not fully intercalate into the interlayer space due to the steric hindrance. According to the interaction between PAMAM and clay, effective internal and external inhibition on shale hydration and dispersion could be obtained with the combination of low generation G0 and high generation G5. A new water-based drilling fluid including G0 as shale swelling inhibitor and G5 as shale dispersion inhibitor was established and exhibited excellent inhibitive properties.

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## 1. Introduction

Wellbore instability due to shale hydration and dispersion triggers many problems, which has been a challenge in the oil and gas industry for many years (O'Brien and Chenevert, 1973; Simpson et al., 1994; Lal, 1999; Bai, 2013). To alleviate the complicated problems, oil-based drilling fluids are always the principal choice. However, high cost and rigorous regulations restrict their wide use (Al-Ansari et al., 2005). Therefore, identifying and developing a high performance water-based drilling fluid with the properties approaching that of oil-based drilling fluids becomes the alternative research goal. In this case, inhibiting shale hydration and dispersion is the first to be considered among the properties of water-based drilling fluid (Young and Friedheim, 2009).

In order to minimize the hydration and dispersion of shale, various chemicals have been used to stabilize reactive shales (Bruton and McLaurine, 1993), which contain a significant amount of smectite clays and easily hydrate and swell when exposed to water acting through a variety of mechanisms and have shown satisfying results around the

world (Caenn and Chillingar, 1996; Van Oort, 2003; Anderson et al., 2010; Sharma et al., 2012). However, there is still not a simple or general chemical solution to control the instability of shale formations (Gomez and Patel, 2013). Most of the current water-based drilling fluids show marginal success due to their inadequate characteristics (Patel et al., 2001; Fletcher et al., 2003; Young and Ramses, 2006). For this reason, attention has been focused to design new strategies to enhance shale stability (Schlemmer et al., 2003; Morton et al., 2005; Patel, 2009).

Dendrimers, first discovered by Tomalia (Tomalia and Frechet, 2002; Dang et al., 2013), are different from traditional polymers in that they possess a multi-branched, three-dimensional structure with very low polydispersity, high polyfunctionality and huge surface areas (Chao et al., 2006). Because of this special molecular structure, dendrimers and dendritic polymers have attracted considerable attention worldwide, and are ideal candidates for a wide range of applications (Qu et al., 2012; Amin et al., 2013; Garea et al., 2013; Chiu et al., 2014). Recent study has shown that dendrimers can be used in the area of oilfield chemistry (Kaiser, 2013; Zhang et al., 2014). In terms of wellbore stability, dendrimers may provide effective external and internal inhibition to reactive shale surface (Amanullah and Aramco, 2013). Miller (2011) reported that hydrogenated poly (propylene imine) dendrimers

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could be used as shale hydration inhibition agents and were not hydrolyzed up to 260 °C, exhibiting high temperature resistance. Teixeira et al. (2014) found that the hyperbranched polyglycerols were promising environmentally friendly inhibitors in water-based drilling fluids.

As the first synthetic dendrimers, amine terminated polyamidoamines (PAMAM) are one of the most studied dendritic macromolecules (Maiti et al., 2004; Xu and Zhao, 2006; Cheng et al., 2007). The ellipsoidal or spheroidal shape and much higher amine group density compared to the classical linear polymers, enable PAMAM to exhibit some special properties, and they may be expected to have potential applications in improving shale stability. To date no one has reported the use of PAMAMs for shale stabilizers. Therefore, the intent of this study is to explore the feasibility of PAMAM dendrimers as shale hydration and dispersion inhibitors in water-based drilling fluids.

## 2. Materials and methods

### 2.1. Materials

Amine terminated polyamidoamine (PAMAM) dendrimers ranging from generation 0 (G0) to generation 5 (G5), were synthesized by the divergent method from the Michael reaction of ethylene diamine and methyl acrylate, both provided by Sigma-Aldrich. Sodium bentonite (Na-Bent) with a cation exchange capacity (CEC) of 74 mmol/g was purchased from Boyou bentonite Group Co., Ltd, China, and the mineralogical composition was presented in Table 1. Drilling fluid bentonite was obtained from Weifang Huawei Bentonite Group Co., Ltd, China, following the American Petroleum Institute (API) standard. The hydrochloric acid used for adjusting the pH value and the potassium chloride were purchased from Sinopharm Chemical Reagent Co., Ltd, China with analytical purity. Polyoxypropylene diamine with a molar mass of 230 (abbreviated as POP230) was provided by BASF Chemical Co. XC (xanthan gum), PAC-L (polyanionic cellulose with low viscosity), SD-505 (lubricant), PHPA (partially hydrolyzed polyarylamide), FA367 (amphoteric polymeric encapsulator), polyglycerol and superfine calcium carbonate were kindly provided by Shida Chuangxin technology company, Ltd., China. Barite was provided by the Drilling Technology Company of Shengli Oilfield of China. The additives for oil-based drilling fluid including mineral oil, organic bentonite, primary emulsifier, assistant emulsifier, wetting agent, and fluid loss reducer were all purchased from the Guangdong Kaiping Keheng Oilfield Additive Factory, China. All of the chemicals were used as received without further purification.

The shale cuttings samples used for hot-rolling dispersion tests and bulk hardness tests were obtained from well TH10261 of Tahe oilfield in west China, and the main mineralogical compositions were presented in Table 2.

### 2.2. Methods

#### 2.2.1. Inhibitive properties evaluation

The testing procedure for the bentonite inhibition test, the shale cuttings hot-rolling dispersion test and the bulk hardness test were carried out according to Zhong et al. (2011). The concentrations of shale inhibitors were 1%(w/v) for all the tests. The pH value of the PAMAM dendrimer solution was adjusted by 1 mol/L hydrochloric

**Table 1**  
The mineralogical composition of Boyou bentonite.

Mineralogical composition	Content (wt%)
Montmorillonite	57.6
Illite	1.2
Kaolinite	0.6
Chlorite	0.6
Quartz	32
Feldspar	8

**Table 2**  
The mineralogical composition of shale cuttings.

Mineralogical composition	Content (wt%)	Component of clay mineral	Content (wt%)
Quartz	30	Kaolinite	2
Potassium feldspar	7	Chlorite	16
Plagioclase	16	Illite	39
Calcite	18	Illite/smectite mixed layer	43
Dolomite	3		
Siderite	1		
Hematite	1		
Clay mineral	24		

acid. The linear swelling test was conducted by a linear swelling tester. Ten grams of Na-Bent was pressed into a sized pellet under a pressure of 10 MPa for 5 min. Then the reconstituted shale pellet was placed in the shale chamber that limited the sample swelling to the vertical direction, and was then immersed in the testing fluid. As the shale pellet came into contact with testing fluid, the variation of shale pellet height with time was recorded by a transducer. The swelling rate of shale pellet was determined (Donham and Young, 2009). Bulk hardness test and linear swelling test were both performed in triplicate. The results were expressed as the mean for each formulation, assessed in triplicate. The reproducibility of the tests was quantified by the standard deviation of the experimental data.

#### 2.2.2. Drilling fluid properties

The rheological properties including apparent viscosity (AV), plastic viscosity (PV) and yield point (YP) were measured by a ZNN-D6 rotational viscometer (Qingdao Haitongda Special Instrument Co., Ltd). According to the API recommended practice of standard procedures, the rheological parameters were calculated from 600 and 300 rpm readings with the following formulas (Recommended Practice, 1988),

$$AV = \varphi_{600}/2 \text{ (mPa.s)}$$

$$PV = \varphi_{600} - \varphi_{300} \text{ (mPa.s)}$$

$$YP = 0.5(\varphi_{300} - PV) \text{ (Pa)}$$

The fluid loss of testing drilling fluid was measured by using an API filter press under a pressure of 689.5 kPa for 7.5 min (Meng et al., 2012).

#### 2.2.3. Characterization of bentonite-PAMAM composites

The composites were prepared through the solution intercalation method using deionized water as the solvent medium. Sodium bentonite (7 g) was dispersed in 350 mL deionized water and was stirred vigorously for at least 24 hours to reach well dispersion. PAMAM dendrimers with the concentration ranging from 0.1% to 2%(w/v), were added into the dispersion and were stirred vigorously for 4 hours. Then the mixture was incubated for 24 hours at ambient temperature under shaking. The obtained mixture was centrifuged at 8000 r/min for 20 min and was washed several times to eliminate the unadsorbed polymers. After centrifugation, the sedimentation was collected, dried at 105 °C and ground to fine powders for characterization. The hybrid materials were abbreviated as G0-Bent to G5-Bent.

Fourier transformation infrared spectra (FT-IR) were recorded by a NEXUS FT-IR spectrometer (Thermo Nicolet Corporation), scanning from 400 to 4000  $\text{cm}^{-1}$  with 32 scans and a resolution of 4  $\text{cm}^{-1}$  in transmission by using KBr pellets. The KBr pellets were prepared by pressing mixtures of 1 mg of powder sample and 100 mg of KBr. The  $d_{001}$ -value of the modified bentonite was measured by X-ray diffraction (XRD). Samples were pressed in glass sample holders and measurements were obtained with X'pert PRO MPD diffractometer operating at 45 kV and 40 mA. The Ni-filtered CuK $\alpha$  radiation ( $\lambda = 0.154 \text{ nm}$ )

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