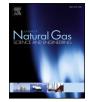
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Application of core-shell structural acrylic resin/nano-SiO₂ composite in water based drilling fluid to plug shale pores



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ARTICLE INFO	A B S T R A C T
Keywords: Plugging agent Shale Core-shell Acrylic resin Nano-SiO ₂ Drilling fluid	Integrating the advantages of inorganic and polymeric nanoscale plugging agents, this paper introduced the synthesis of a nanoscale acrylic resin/nano-SiO ₂ composite (AR/SiO ₂) with a core-shell structure for water based drilling fluid (WBM), which can enhance the plugging efficiency of shale pores, reduce fluid invasion and improve wellbore stability in shale gas drilling process. The core-shell structure of AR/SiO ₂ was characterized by transmission electron microscope (TEM). Thermal Gravity Analysis (TGA) indicated the thermal resistance of AR/SiO ₂ up to 250 °C. Theoretical analysis implied that improved plugging property of AR/SiO ₂ might be due to good adhesion of deformable shell parts with shale matrix and the supporting provided by rigid core parts. The plugging ability was evaluated by pressure transmission tests and N ₂ adsorption tests. The results demonstrated that WBM containing AR/SiO ₂ can substantially enhance plugging efficiency and reduce fluid invasion. Characterization of filter cakes revealed that AR/SiO ₂ improved the quality of filter cakes, which resulted in a

considerable improvement in filtration properties.

1. Introduction

China has the world's largest shale gas reserves, according to reports prepared by the U.S. Energy Information Administration (Administration (2013). However, because of the complex geological conditions and the deep buried depth, the development of Chinese shale gas faces great challenges (Lukosavich, 2011). During drilling watersensitive shale formations, Wellbore instability (Kanfar et al., 2017; Zevnali, 2012) is a major concern that may cause hole collapse, tight hole and hole erosion. The fluid invasion into shale pores accelerates the swelling rate of shale. For high permeability formations, a filter cake which is a layer of solid particulates, forms inside of a borehole during drilling process and provides a physical barrier to prevent fluid invasion. However, shale formation normally has extremely low permeability, thus no effective or very low-quality filter cakes can form (Khodja et al., 2010; Labenski et al., 2003). Therefore, the plugging property of drilling fluids plays an important role to reduce fluid invasion (Ghanbari and Naderifar, 2016; Xu et al., 2017), which can maintain wellbore stability (Feng and Gray, 2016), reduce drilling accidents (Feng and Gray, 2017) and save costs while drilling shale formations.

Most shale pores are on the order of nanometer to micrometer, of which nanoscale pores comprise a dominant proportion (Clarkson et al., 2013; Kuila and Prasad, 2013; Webber et al., 2013). Nevertheless, conventional plugging agents such as fine calcium carbonate, asphalts and walnut shell powder are in micron-scale, which cannot plug the nanoscale pores effectively during shale gas drilling process.

Inorganic nanoparticles (Cai et al., 2012; Hoelscher et al., 2012; Sensoy et al., 2009; Sharma et al., 2012) were used to plug shale pores at first. Taner Sensoy et al. (2009) found that nanoscale silica spheres can reduce fluid invasion into shale when used in an appropriately designed drilling fluid. KP Hoelscher et al. (2012) observed a good plugging property of WBM containing graphene oxide. However, inorganic nanoparticles are easy to aggregate and weaken the plugging ability. Besides, inorganic nanoparticles are rigid and difficult to deform. Their interaction with the shale matrix is weak. Thus, the plugging strength of inorganic nanoparticles is not good.

Polymeric nanoparticles having hydrophilic groups can be better dispersed in WBM. Yuxiu An et al. (2015) introduced a nano terpolymer with good dispersion into drilling fluid. The study by Xie Gang et al. (Xie et al., 2015) showed that hyperbranched polyamine with an average particle size of 49.5 nm could achieve good plugging rate. Although polymeric nanoparticles can deform and establish relatively higher interaction with the rock matrix, on the other hand, they are easily broken through by high-temperature high-pressure (HTHP) fluids when bottom hole temperature is above their glass transition temperature (Tg), which weakens the plugging performance.

The introduction of polymer-inorganic nanocomposites (Jain and

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Mahto, 2015; Jain et al., 2015; Sadeghalvaad and Sabbaghi, 2015) in drilling fluids has become research hotspot in recent years, which are not only helpful to reduce filtration volume but also beneficial to the rheological property of drilling fluids. However, because the polymers used in these nanocomposites are mostly water-soluble, these nanocomposites cannot deform in WBM. Thus, these nanocomposites cannot have the advantages of both rigidity of inorganic nanoparticles and flexibility of polymeric nanoparticles.

The objective of this paper is to develop an organic-inorganic nanocomposite as plugging agent that can combine the advantages of inorganic and polymeric nanoscale plugging agents. In this paper, we synthesized a nanoscale acrylic resin/nano-SiO₂ composite, measured its morphology and thermal stability, evaluated its influence on the properties of drilling fluids and its plugging ability for shale pores using pressure transmission experiments and BET pore analysis.

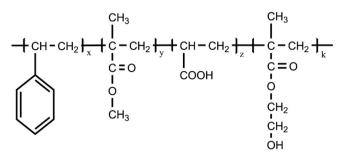
2. Experimental section

2.1. Reagents and materials

The nano-silicon dioxide modified by silane coupling reagent KH-570 (nano-SiO₂) was purchased in Nanjing XFNANO Company (99.5 wt %, 20 nm). Styrene (St, 99.5 wt%), methl methacrylate (MMA, 99 wt%), acrylic acid (AA, 99 wt%), hydroxyethyl methacrylate (HEMA, 97 wt %), polyethylene glycol octylphenyl ether (OP-10, 99.5 wt%), sodium dodecyl benzene sulfonate (SDBS, 95 wt%), polyvinyl alcohol (PVA, fully hydrolyzed), potassium persulfate (KPS, 99 wt%), and other reagents were obtained from J&K Scientific Ltd., Beijing. The monomers (St, MMA and HEMA) were washed by 5 wt% NaOH aqueous solution to remove the inhibitor, followed by deionized water washing for several times until pH = 7, and dewatered by passing through anhydrous sodium sulfate before use. Shale cores (Φ 25 mm × 50 mm, black muddy shale) and bentonite were provided by CNPC Chuanqing Drilling Engineering Company, which was acquired from a shale gas well at 1520 m in Weiyuan area, Sichuan Province, China.

2.2. Preparation of latexes of acrylic resin (AR) and acrylic resin/nano-SiO₂ composite (AR/SiO₂)

The AR and AR/SiO₂ latexes were prepared by the semi-continuous emulsion polymerization method. 2.5 g modified nano-SiO2 was added into the monomer mixture consisting of 32.5 g St, 16 g MMA, 1.5 g AA and 1.0 g HEMA. The nano-SiO2/monomers mixture was treated by ultrasonic dispersion for 2 h, and pre-emulsified with 20 g water solution containing 0.34 g OP-10 and 0.17 g SDBS by a high shear mixer. A 250-ml round-bottomed 4-neck flask equipped with a reflex condenser, an electrical mixture was successively charged with 15 ml 2 wt% PVA water solution and one fourth of the pre-emulsion. The flask was immersed into a water bath at 71 °C. The mixing speed was set at 300r/ min. When the temperature of the liquid in the flask reached 70 °C, 2 ml 5 wt% KPS water solution was added. After conditioning at 70 °C for 10min, the water bath was heated to 81 °C. The remaining pre-emulsion and 15 ml 1 wt% KPS aqueous solution were then added into the flask using two dropping funnels simultaneously. After adding the preemulsion and KPS aqueous, the heating was last for additional 1 h. The reaction was terminated by cooling down to 40 °C, and AR/SiO₂ latex was obtained by adding ammonia aqueous solution (25 wt %) to adjust the pH to 7. Pure acrylic resin (AR) latex was obtained using the same method without adding of nano-SiO₂ (see Scheme 1). The solid samples were obtained by repeated centrifuging (10000r/min) combined with washing treatment by ethanol and deionized water, and finally dried in an oven at 105 °C which were used for FTIR, TGA and DSC.



Scheme 1. Chemical structure of synthesized acrylic resin.

2.3. Characterization

2.3.1. Characterization of AR and AR/SiO2

The Fourier transform infrared (FTIR) absorption spectra of AR and AR/SiO₂ were measured in the range from 4000 to 500 cm⁻¹ using an Agilent Technologies Cary 670 FTIR spectrometer. Thermal measurements (DSC and TGA) of solid samples were carried out using a simultaneous Thermal Analysis apparatus (STA 409 PC, Netzsch, Germany) under argon flow from 25 to 400 °C with a ramping rate of 5 °C/min. The glass transition temperature was determined as the temperature at the midpoint of slope shifting.

The latexes of AR and AR/SiO_2 were suspended into deionized water, treated by ultrasonic dispersion for 2 h and then characterized with a Zetasizer Nano ZS90 (Malvern Instruments) and a high-resolution transmission electron microscopy (TEM, Tecnai G2 F20, FEI). Samples for TEM were prepared via drying of a drop of the diluted latexes on an ultra-thin carbon supporting film.

2.3.2. Characterization of shale

The mineral compositions of shale core were analyzed by a Shimadzu XRD-6000 diffractometer.

The shale sample was crushed and sieved through 100 meshes. BET adsorption method was utilized to analyze shale pore structure using a nitrogen adsorption analyzer (Autosorb-iQ2-C, Quantachrome Instruments, USA).

2.4. Application

2.4.1. Preparation of plugging fluids and measurements of corresponding properties

Base fluid was prepared by dispersing 4 g bentonite into 100 ml distilled water, then blended with 1%AR or +1%AR/SiO₂ to obtain plugging fluids. The AR and AR/SiO₂ samples used in drilling fluids are in latex form in order to make sure that nanoscale particles can be fully dispersed. Static filtration tests and rheology measurements of plugging fluids were conducted according to API RP 13B-1 Recommended Practice for Field Testing Water-Based Drilling Fluids. The filter cakes were freeze-dried and studied using scanning electron microscope (Quanta 200F, FEI).

2.4.2. Shale pore plugging test

Shale pore plugging tests, as described in details before (Akhtarmanesh et al., 2013), were conducted using a pressure transmission testing apparatus (Fig. 1) produced by Jinzhou Modern Petroleum Science & Technology CO., LTD. The schematic of the pressure transmission test are shown in Fig. 2.

Before the test, shale cores were vacuumed and saturated with 20 wt % NaCl aqueous solution. Then, the shale core was placed in the holder with a constant confining pressure of 15 MPa. The pressure of the downstream line filled with 20 wt% NaCl aqueous solution was initially set at 1.0 MPa, and the pressure of the upstream line filled with one of the plugging fluid was set to a constant value of 11 MPa. During the whole test, the pressure change of the downstream line was monitored,

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