



Research paper

Laser beam backscattering as a new tool to study the effect of inhibitors on shale particles-water interactions: A real-time analysis



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ABSTRACT

The interaction between clay-rich shales formations and water is one of the most relevant factors for well instability in drilling operations, representing a significant challenge for the oil and gas industry. To minimize the problems related to water sensitivity, chemical additives or inhibitors are incorporated into water-based drilling fluids, acting over the water-shale interactions and maintaining the integrity of the sedimentary rock. In this work a novel approach is proposed to gain new information about the dynamics of the inhibitory process and to evaluate the shale inhibition efficiency of different chemical additives using a laser beam backscattering technique. This technique is based on the analysis of laser backscattering profiles promoted by dispersed particles in a suspension, which results in a particle chord length distribution (CLD). The technique was adequate for monitoring the consequences of the interactions between water and shales, differentiating its reactivities by monitoring the effect of different inhibitors on each system's CLD over time. Two cationic inhibitors (KCl and PDADMAC, a cationic polymer) were tested, and the obtained results showed that the inhibition phenomenon is more than the inhibitor itself, and it depends on the shale structure and the capability of the inhibitor to interact with its clay stacks. A methodology was also developed to calculate an inhibition efficiency score (IES), in which the technique's real-time factor enables it to estimate how much better an inhibitor is when compared to others over time. Among the evaluated systems, the inhibitor that achieved the biggest efficiency for shale was KCl, resulting in 69%.

1. Introduction

The interaction between clay-rich shales formations and water, is one of the most relevant factors for wells instability and represents a huge challenge for the oil industry. It is estimated that the industry loses worldwide about US\$ 1 billion per year only with problems related to instability of shales (Zeynali, 2012).

The instability of shales is caused by several factors, mechanical or physico-chemical, which may be associated with the drilling operation itself or the nature of the formation. Technical drilling events such as the friction between the drilling column and the well walls, pressure peaks and excessive overbalance pressures constitute causes of instability of shales. As factors related to the nature of shale formation could be cited its texture, structure and fabric (Wilson and Wilson, 2014), and the distribution of stresses *in situ* (Bell, 2003). While it is generally accepted that the nature of shales is the main cause of

instability, there is no general consensus as to the mechanism involved. Several authors consider that the instability trigger is the osmotic swelling that occurs in the interlamellar region of the sodium smectites (Anderson et al., 2010; Bailey et al., 1994; Reid et al., 1995). The evidence reported in these studies indicates that a good number of shale stabilizing additives act by intercalating within expandable clays and inhibiting their hydration and swelling. Another approach, proposed by Wilson and Wilson (2014), shares the cause of instability between the interlayer region of expandable clay minerals and the pores of the shales, with main emphasis on the latter. This new conceptual proposal emerged from the observation that not only smectic shales present instability. Shales consisting mainly of non-reactive clay minerals such as illite and kaolinite may also be unstable. Studies have shown that the thickness of the diffuse double layer (DDL) around external clay mineral surfaces is probably of the same order and even larger than the size of a significant proportion of pores found in shales. (30–1830 Å)

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(Johnston and Tombacz, 2002; Mojid and Cho, 2006; Sridhara and Satyamurty, 1996). Under these circumstances, the overlap of DDL associated with exposed clay surfaces on opposite sides of macropores and mesopores could result in electrostatic repulsion and lead to increased pore pressure in all types of shales. According to the novel approach, the stabilizing effect of shales produced by inhibitors such as K^+ and PDADMAC would be more related to contraction of DDL thickness than reduction of swelling pressure in expandable clay minerals.

The most common methods to evaluate inhibitor performance include Hot-rolling Cuttings Dispersion, Bulk-hardness, and Bentonite Inhibition tests. The Hot-rolling Cuttings Dispersion test involves shale cuttings being exposed to the fluid under drilling average conditions (66 °C), and after 16 h, the dispersed and intact cuttings are recovered and measured. Bulk-hardness is a test in which the sample's hardness is measured after the shale has maintained contact with the drilling fluid; a good inhibitor prevents the clay's capacity to absorb water from the fluid. The Bentonite Inhibition test basically determines the maximum amount of fine solids that can be inhibited, *i.e.* maintained with a low rheological profile when gradually added to an inhibitor solution. The suppression of the rheological profile reflects the ability of the tested product to prevent hydration and dispersion of particles of reactive clays. It is important to emphasize that the methods used to evaluate shale inhibition do not provide the same information, and do not show always the same trend. For example, hot-rolling cuttings dispersion and bentonite inhibition tests showed different results when the same systems containing poly(ethylene glycol) derivatives were compared (Villabona-Estupiñán et al., 2017). Thus, the inhibition tests themselves do not provide clear and complete information on the dynamics of the inhibitory process. It is necessary to use complementary techniques, such as X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), Scanning electron microscopy (SEM), and adsorption isotherm studies, to understand said dynamics. In this work a novel approach is proposed to gain new information about the inhibition mechanism, evaluating the shale inhibition performance of different chemical additives through the laser beam backscattering technique. This technique operates with a probe immersed in a dispersion mixture, which uses a focused laser beam to evaluate the presence and the size of particles. When the light hits a particle, the laser beam is backscattered to the detector, allowing the equipment to determine the cross-section length of the particle (chord length) and providing information about the chord length distribution (CLD). Such analysis is carried out continuously over time, which enables the technique to monitor real-time dynamic changes in the system's particle size distribution (PSD) (Li and Wilkinson, 2005). In addition, since the analysis is performed *in situ*, it does not require sampling, which facilitates the process and minimizes the risk of measurement errors.

This technique has been widely applied in different areas, as in flocculation/aggregation studies (Antunes et al., 2015; Jarabo et al., 2010; Miranda et al., 2013; Uduman et al., 2010; Zakrajšek et al., 2009), emulsion reactions (Esteban et al., 2015; Ravelo et al., 2015), protein dissolution (Fang et al., 2011), granulation (Alshihabi et al., 2013), and crystallization processes (Al Nasser and Al Salhi, 2015; Frawley et al., 2012; Melchuna et al., 2016; Saleemi et al., 2012; Sun et al., 2015). In the context of the present study, the suggestion to use this technique to evaluate the interaction between clay-rich shales formations and water is based on the hypothesis that the dispersion of particles of shales in aqueous media is influenced by the interlamellar swelling and the repulsion between diffuse double layers in the pores of the material. Particles of natural clay materials are not made up of separate units of lamellae. Lamellae of natural phyllosilicates occur associated in piles forming quasicrystals commonly called mesoscopic tactoids (20–500 Å). These tactoids, in turn, are forming macroscopic clay particles or aggregates (~1 µm) (Kuila and Prasad, 2013). Inhibitors could act on the interlamellar region of clay minerals or pores, retarding the disintegration of said particles in to smaller particles. On

the other hand, hydration inhibitors have agglutinating effect which would lead to aggregates formed from smaller particles.

The present work is the first that proposes the use of laser back-scattering as an in-line technique to evaluate the shale inhibition process based in CLD changes. It is not the aim of this work to replace the techniques already well established by the new one presented here. Instead, the goal is to provide different information about the inhibition phenomena through a new approach. If the inhibitor could minimize shale reactivity in the presence of water, then the number of fine particles will not increase as much as in the absence of the shale inhibitor. Comparing the two cases' changes in CLD will indicate reactivity, which can provide information regarding how much better one inhibitor is when compared to the other over time. Since the technique is automated and works in real time, the changes can be monitored throughout a given period, which may also be useful for estimating the kinetic factor of reactivity. To evaluate the methodology, the stability of three different shales was monitored in an aqueous medium in the presence or absence of an inhibitor (PDADMAC or KCl). The shale's reactivity and the inhibitor's efficiency were also tested for different temperatures. It should be noted, however, that the goal to this study was to establish if the analytical design implemented is able to differentiate the reactivity of several types of clay in the presence of inhibitors and not to deepen the evaluation of the different mechanisms of inhibition.

2. Materials and methods

2.1. Materials

Potassium chloride and Poly(diallyldimethylammonium chloride) - PDADMAC - solution 20 wt% in H_2O with a medium molecular weight (M_w : 200,000–350,000 $g\text{mol}^{-1}$), were supplied by Sigma Aldrich. Shale samples (A1, A2, and A3) were obtained from the Bentonorte Company (Paraíba, Brazil), and they were classified by size using 150 and 270 mesh (106–53 µm) sieves. The aqueous systems used in the experiments were prepared using type I water from a Milli-Q system (Millipore).

2.2. Shale characterizations

All shales were previously characterized by X-ray Diffraction (XRD) to determine the mineral content through the Rietveld refinement method. This was performed by analyzing the shales *in natura*, glycolated (saturated with ethylene glycol) and calcinated (heated at 500 °C for 2 h). The samples' X-ray diffraction patterns were recorded using a RIGAKU Ultimate IV X-ray diffractometer, with $Cu-K\alpha$ radiation ($\lambda = 1,5418 \text{ \AA}$), operating at 30 kV and 15 mA and with a fixed monochromator. Data were collected in a 2θ range of 2 to 30°, with a step size of 0.01°, a scan rate of 2.00° min^{-1} and an incident slit of 1.25°.

The chemical composition was measured by X-ray fluorescence (XRF) in a WDS-2 model AXIOS (Panalytical) fluorescence spectrometer.

Loss on Ignition (LOI), a measure of volatile content, was determined by storing the samples at 1000 °C for 16 h, and then they were weighed after cooling.

The samples' cation exchange capacity (CEC) was quantified through the methylene blue test, which is a measure of the exchangeable cations that neutralize the negatively charged clay particles (Stephens et al., 2009).

Nitrogen adsorption and desorption data were obtained on the volumetric apparatus ASAP2010 (Micromeritics) to determine the textural analysis. The BET method was used to calculate the specific areas, and the Barrett-Joyner-Halenda (BJH) method was used to calculate the porous diameter and the porous volume. Samples were outgassed at 80 °C for 24 h prior to the analyses.

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