Research paper

Functional characterization on colloidal suspensions containing xanthan gum (XGD) and polyanionic cellulose (PAC) used in drilling fluids for a shale formation

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ABSTRACT

Drilling fluids are employed in the operation of hydrocarbon exploitation. Two kinds of drilling fluids are commonly used: water based muds and oil based muds. The oil based muds are characterized by their high performance in unconventional reservoirs due to the very low interaction with the reactive formation. Their environmental impact promotes the design of water based muds that present low toxicity. In this paper a water based mud for shale formation with similar rheological characteristics to the oil based mud used in the oil industry of Argentina was designed and the effect of xanthan gum and polyanionic cellulose on the main functional characteristics was studied. Rheometric analysis showed a shear-thinning behavior with notable effect on the concentration of polymers. After dynamic aging test, fluids with composition of PAC = 8.00 g/L and XGD = 3.00 g/L exhibited rheological properties very close to oil base mud. Structural changes were assessed from optical microscopy and scanning electron microscopy. Particles agglomeration due to the presence of polymers was observed. Furthermore, mud filtration essays allowed to evaluate the performance of PAC as control agent. The Carreau model and statistical analysis were used to determine rheological parameters.

1. Introduction

Drilling is a major step towards the success of hydrocarbon exploitation, which depends not only on the formation geological conditions but also on the implemented technology for its development. Oil drilling is the next stage after exploration and represents an expensive step in the oil and gas industry. In the last decade, the expenditures for drilling represented 25% of the total cost of exploitation (Khodja et al., 2010a). Also, drilling fluids are critical during this stage and account for up to a fifth (15–18%) of the total drilling cost. The design and composition of such fluids are determined by the geological characteristics of the formations and the additives available in the location. Fluids must also meet requirements physicochemical, economical and environmental (Darley and Gray, 1988). Density, filtration control, pH, rheological behavior and chemical composition of drilling fluids must contribute to fulfill different functions that include cleaning of well, keeping of cuttings suspended, preventing the cavitation, forming an impermeable cake near the borehole, cooling and lubricating tools, promoting hydraulic power, and being compatible with logging tools.

Water based muds (WBMs) and oil based muds (OBMs) are two types of fluids used in Argentina for the hydrocarbon extraction. OBMs exhibit high performance regarding penetration rate, clay swelling in the shale, excellent filtration control, wellbore stability, high lubricity, high thermal stability, high salt tolerance and good ability to transport cuttings (Shah et al., 2010; Zhong et al., 2012). But, they present some disadvantages related to the environmental impact, cost and negative effects on cementing of the well due the poor adhesion between the casing and the formation.

From an environmental point of view, the use of WBMs represents an attractive alternative. However, it also has drawbacks, such as its interaction with the highly hydrophilic clay in the formation, which causes instability of the walls due to scattering problems and clay swelling (Gholizadeh-Doonechaly et al., 2009; Carvalho et al., 2015). For this reason, specific additives are used in order to obtain properties comparable to OBM (Patel et al., 2007). WBMs are complex systems consisting of an aqueous continuous phase with other components such as clays, viscosifier agents, deflocculants, lubricants, clay inhibitors, filtrate control additives and weighting filters.

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Technical performance of WBMs is highly associated with the rheological behavior (Drilling control parameters: plastic viscosity, yield value, low-end rheology and gel strength), density, solids content and pH (Baker, 1998; Instituto Americano del Petróleo, 2001).

Polymeric additives such as xanthan gum (XGD) and hydroxymethyl cellulose are usually used in WBMs as viscosifiers, depending on their molecular structure and chemical configuration (Caenn and Chillingar, 1996; Benyounes et al., 2010; Jang et al., 2015). The addition of these viscosifiers results in a shear-thinning and thixotropic behavior of the mud that improves the filtrate control and facilitates the cleaning of the well and the suspension, and removal cuttings in absence of fluid flow (Ezell et al., 2010). Polymers such as polyanionic cellulose (PAC), starch and others act as filtrate reducers minimizing the fluid flow towards the formation by generating a thin cake with low permeability in the wellbore (Kok and Alikaya, 2004; Mahto and Sharma, 2004; Gholizadeh-Doonechaly et al., 2009; Khodja et al., 2010b; Vipulanandan and Mohammed, 2014; Khamehchi et al., 2016). Finally, encapsulant polymers such as partially hydrolyzed polyacrylamide (PHPA) and/or inhibitors such as quaternary amines potassium may be added in order to avoid dispersion and hydration of hydrophilic clays (Gholizadeh-Doonechaly et al., 2009; Hanyi et al., 2013).

There is relatively little research respect to the effect of polymeric additives on the rheological behavior of WBMs. Several WBMs based on aqueous solutions containing bentonite, barite, polymers (XGD, PAC, starch, carboxyl methyl cellulose, acrylamide, scleroglucan, guar gum) and electrolytes (Iscan and Kok, 2007; Hamed and Belhadri, 2009; Khodja et al., 2010b; Olatunde et al., 2011; Wan et al., 2011; Zhong et al., 2012; Fakoya and Shah, 2013; Vipulanandan and Mohammed, 2014; Jang et al., 2015; Ziaee et al., 2015) were investigated. Khodja et al. (2010b) studied the rheological and filtration characteristics of WBMs prepared with XGD, PAC, PHPA and electrolytes used for shale formations of Algeria. In spite of the important effect of the polymers on the physicochemical and rheological properties of the WBMs, no publication has been found regarding WBMs used of shale formations of Argentina.

This work is the first attempt to evaluate the effect of XGD and PAC on the functional properties of WBMs. Low permeability shale reservoirs of Argentina are considered for the study. A WBM with rheological characteristics similar to OBM used in Argentina is experimentally formulated, containing bentonite, barite and different types of additives (viscosifier, filtering agents, lubricants, inhibitors, densifier, solids, encapsulator). The effects of XGD and PAC on the rheological and filtering properties and structural characteristics of fluids are analyzed. Also, thermal stability was assessment by dynamic aging tests. Carreau model and statistical analysis are employed to theoretically describe the rheological behavior of the fluids as a function of the polymer composition.

2. Experimental study

2.1. Polymers characterization

The average molecular weights of polymers were determined by size exclusion chromatography (SEC). A Waters 1525 pump with five Ultrahydrogel® columns (120, 250, 500, 1000, 2000, 7.8 mm × 300 mm, 5 μm) and a Waters 2412 refractive index detector were used. The eluent was NaCl (0.1 M) with a flow rate of 0.8 mL/min, at 25 °C. Pullulans Shodex Standards (No 90401-Showa Denko) were used for calibration.

2.2. Na-Bentonite characterization

X-ray diffraction (XRD) (Shimadzu, model XD-D1) analysis was carried out to obtain information on the mineralogical components of Na-Bentonite. The following parameters were used for XRD characterization: 2 theta ranges of 5–85°, Cu-Kα radiation, a scan speed of 2°/min, an acceleration of voltage and current of 40 kV and 30 mA, respectively. The XRD diffractogram was obtained with DP-D1 software.

Table 1
Base mud composition.

Order of addition	Component	Function
1	Na-Bentonite	Base
	A	Viscosifier and filtration control agent
2	Polyanionic cellulose (PAC)	Filtration control agent
	Xanthan gum (XGD)	Viscosifier
	B	Encapsulator
	C	Inhibitor
3	D	Lubricant
4	E	Filtration control agent
	F	Filtration control agent
	G	Densifier
5	H	pH control
6	I	Cuttings

Table 2
Average molecular weights of polymers.

Polymer	$\overline{M_n}$ (g/mol)	$\overline{M_w}$ (g/mol)
PAC	692.000	1.146.000
XGD	881.000	1.622.000

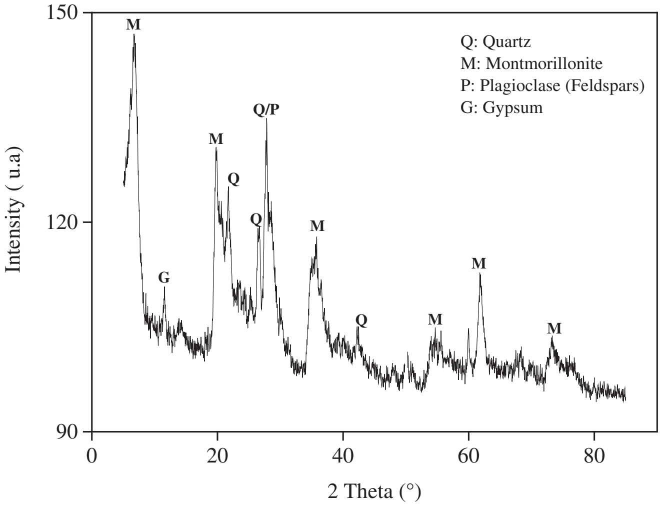

Fig. 1. XRD patterns of the Na-Bentonite.

Fourier transform infrared spectroscopy (FTIR) analysis was also performed in the sample. The spectrum was obtained with a Shimadzu FTIR-8201 PC Spectrometer at 4000 and 400 cm^{-1} range, using a sample pressed as KBr pellet. Hyper IR software was used to analyze the spectrum.

2.3. Shale characterization

The shale samples were obtained from a drilled well in Neuquen, Argentina at 2700 m depth (TVD True vertical depth). The qualitative identification of the minerals present in the samples was obtained through the X-ray diffraction method, carried out for both total (< 2 mm) and fine grain fraction (< 0.0074 mm).

A PHILIPS PW3710 with cupper tube diffractometer was used. The sample preparation and testing methodology was divided in two sections:

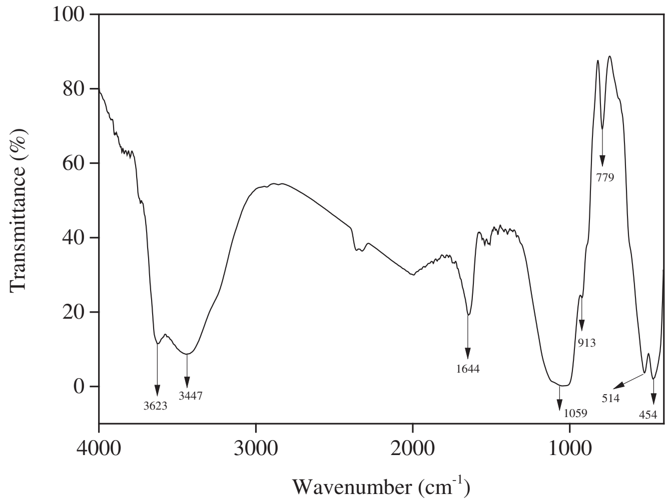

Fig. 2. FTIR spectrum of Na-Bentonite.

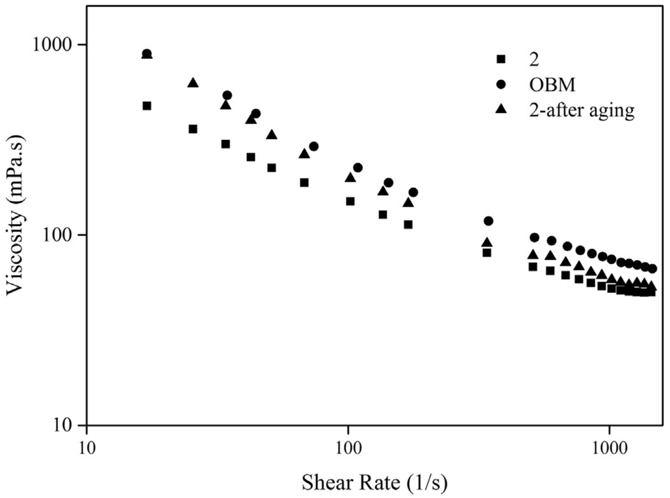

Fig. 3. Viscosity *vs* shear rate curves for OBM, optimized WBMs (Fluid 2, after aging) and base mud (Fluid 2, unaged).

Table 3
Composition of shale cuttings obtained from XRD analysis.

Component (%wt)	Sample 1	Sample 2	Sample 3	Sample 4
Quartz	72.8	68.8	66.5	55
Feldspar	1.2	1.3	1.2	0.5
Plagio	1.2	1.4	3.9	2.1
Calcite	8.2	17	6.8	21.3
Pyrite	1.2	0.7	1.6	0.7
$CaTiO_3$	–	–	–	0.7
Fe_2O_3	–	–	0.2	0.6
MnO_2	–	–	–	0.8
Carbon	0.6	0.4	0.6	–
TiO	–	0.2	–	–
Clay	14.8	10.2	19.2	18.3

Table 4
Clay minerals content in shale cuttings obtained from XRD analysis.

Clay (%)	Sample 1	Sample 2	Sample 3	Sample 4
Kaolinite	8.6	–	7.9	20.4
Illite	68.5	68.6	44.7	67.3
Smectite	8.2	31.4	47.4	12.3
Illite/smectite	14.7	–	–	–

Table 5
XGD and PAC concentrations of drilling muds.

Fluid	C_{XGD} (g/L)	C_{PAC} (g/L)
1 (Mud without polymer)	0.00	0.00
2 (Base mud)	1.50	8.00
3 (Mud without PAC)	1.50	0.00
4 (Mud without XGD)	0.00	8.00
5 (Mud 0.5 C_{XGD})	0.75	8.00
6 (Mud 2 C_{XGD})	3.00	8.00
7 (Mud 3 C_{XGD})	4.50	8.00
8 (Mud 0.25 C_{PAC})	1.50	2.00
9 (Mud 0.5 C_{PAC})	1.50	4.00
10 (Mud 2 C_{PAC})	1.50	16.00

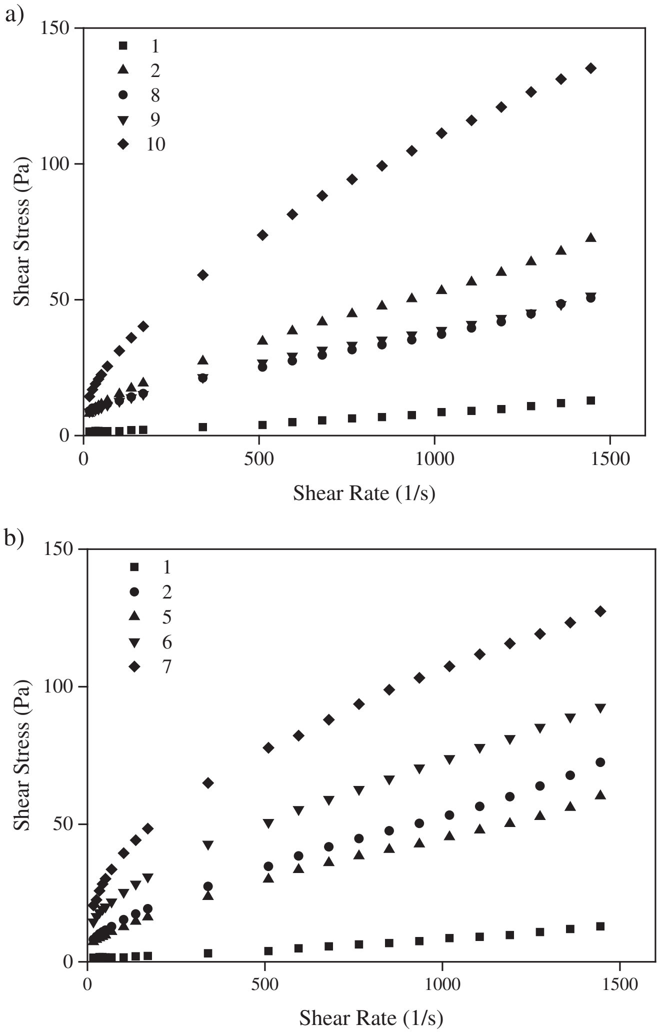

Fig. 4. Shear stress *vs* shear rate of drilling fluids. Effect of PAC (a) and XGD (b) concentrations.

Total grain fraction: Four rocks (samples 1–4) were washed with toluene and crushed to a 2 mm size.

Fine grain fraction (clay): 3 g of crushed rock sample were suspended in distilled water. Two sample holders were prepared for each rock sample, one without chemical treatment and the other with an ethylene glycol treatment to eliminate carbonates and organic matter. The comparison between the diffractograms (scattered angles between 2° to 14°) allowed the identification of clay species and quantification.

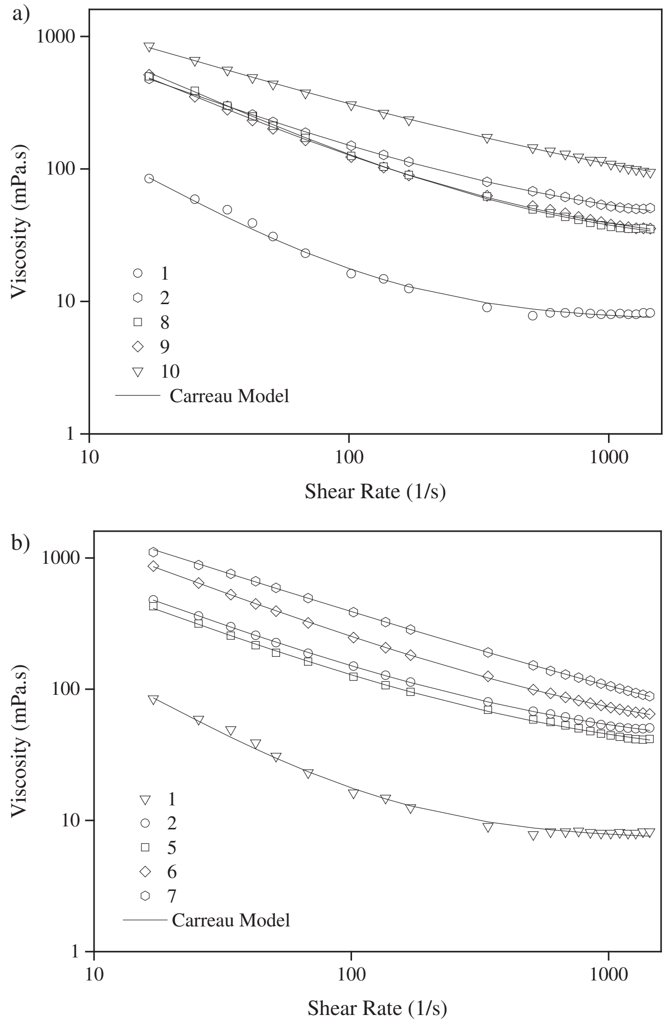

Fig. 5. Viscosity *vs* shear rate curves of drilling fluids. Effect of PAC (a) and XGD (b) concentrations. Simulated curves using the Carreau model are indicated in solid lines.

Table 6
Experimental plastic viscosity.

Fluid	Plastic viscosity (mPa.s)	Standard deviation (S)
1	9.40	1.20
2	37.20	0.77
3	19.00	2.19
4	21.40	1.20
5	30.80	0.35
6	46.60	1.27
7	59.20	2.76
8	28.80	0.14
9	24.00	0.40
10	75.00	2.82

2.4. WBMs preparation

Different WBMs containing an aqueous solution of bentonite, polymers and other additives were prepared (Table 1). All additives were provided by service companies of Argentina (A-I components are not reported for confidential reasons).

API Recommended Practice 13B-1, 2003, was followed in the preparation of fluids. The order of additives addition is also indicated in Table 1. First, water and bentonite were added to a 500 mL vessel and stirred during 30 min and letting it stand for 16 h at room temperature. Then, polymers (PAC and XGD), and inhibitor were added and the mixture was stirred for 10 min. Two plugging agents and a densifier were incorporated under agitation during 10 min. Finally, pH was corrected using NaOH (until pH 10) and additives for the simulation of solids in suspensions (cuttings) were added. The mixture was stirred for 5 min.

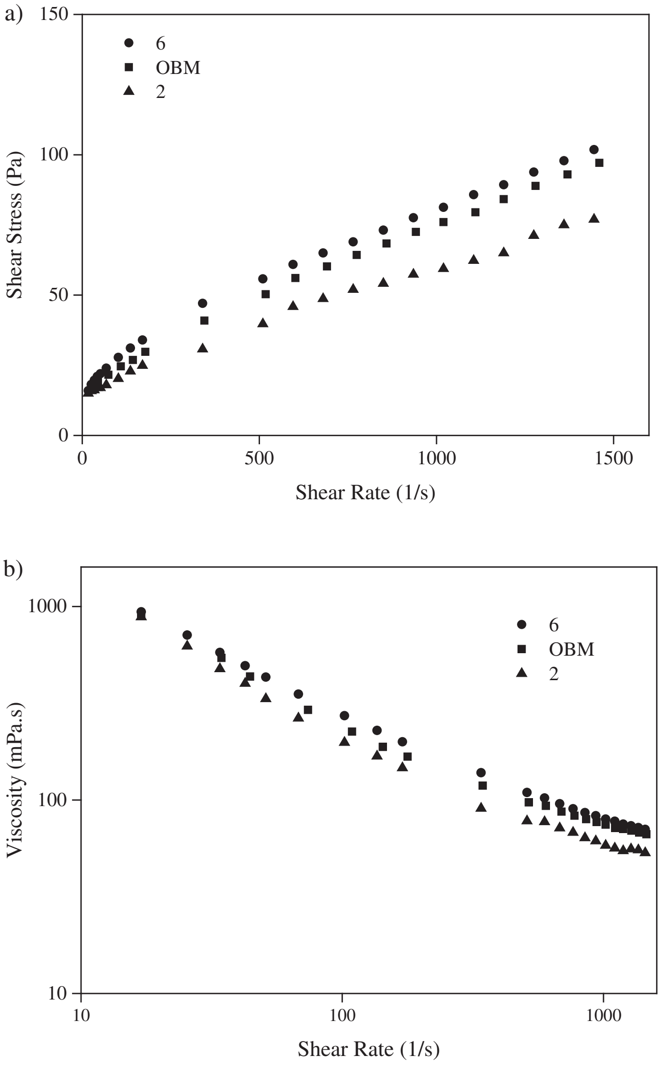

Fig. 6. Rheometric results after aging for base Mud 2, OBM, and Mud 6.

Different compositions were tested in order to select a base mud with rheological behavior similar to that of OBM used in Argentina shale. Also, some additional samples were prepared in the same way from the base mud, but modifying polymer concentration.

2.5. Rheometric characterization of drilling fluids

The rheometric properties of the drilling fluid were measured by using OFITE (model 900) rotating coaxial cylinder viscometer. The configuration R1B1 was used with a shear rate range of 17–1445 s^{-1}, temperature of 25 °C and the readings were made after constant stirring for 30 s.

2.6. Structural characterization of fluids

The morphology of drilling fluid was observed by optical microscopy using a Leica DM2500 M microscope with a coupled camera Leica DFC 290 HD.

In order to complement the observed results, samples were also

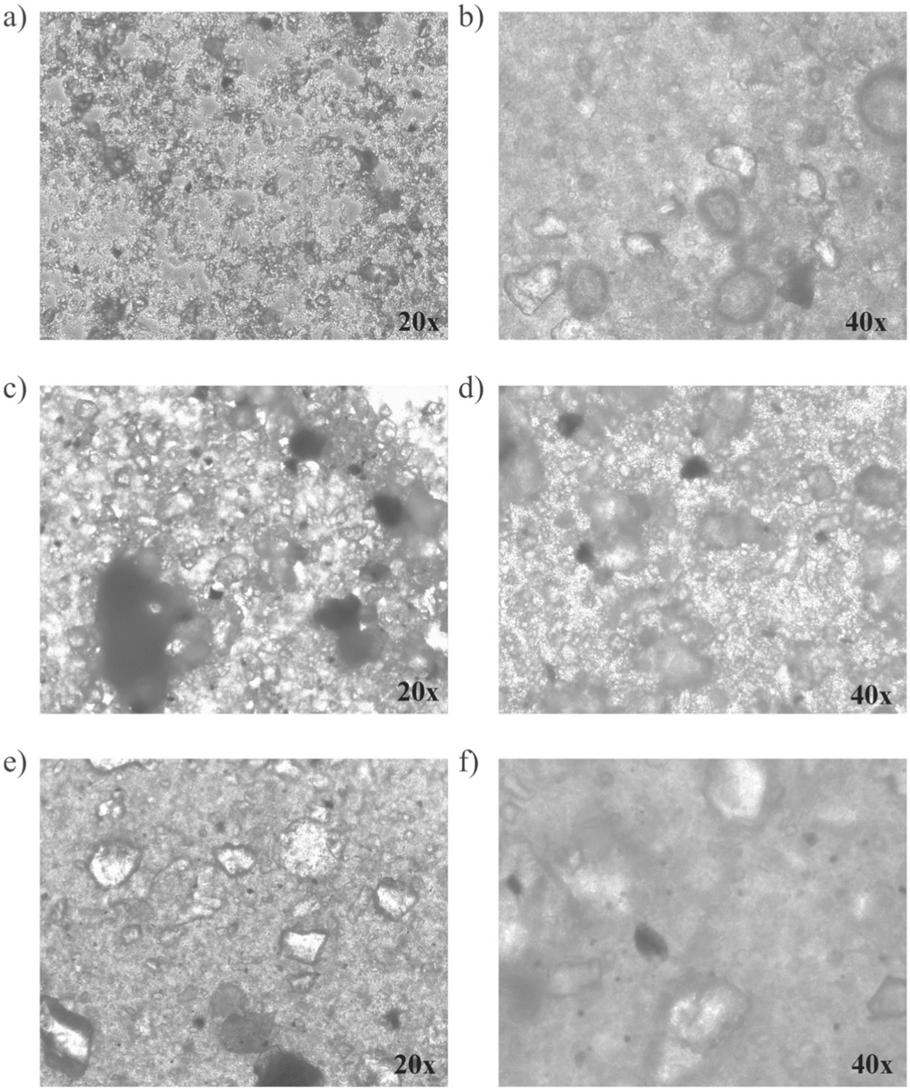

Fig. 7. Micrographs obtained by optical microscopy: a–b) Mud 1; c–d) Mud 6; and e–f) Mud 10.

studied by a scanning electron microscopy (SEM) in a JEOL JSM-35C equipped with the image acquisition program JEOL SemAfore. Samples were put over an aluminum stub, sputter coated with gold under an argon atmosphere (SPI Supplies, 12157-AX) and then examined using an acceleration voltage of 20 kV. These analyses were carried to study morphological and structural changes of drilling fluid in the presence of polymers.

2.7. *Filtration properties of drillings fluids*

Filtration tests were carried out according to the API standards (API recommended practice 13B-1, 2003). To this effect a filter press was used with CO_2 as pressurizing gas at 100 psi and with No. 50 Whatman filter paper. Filtrate readings were performed at 5, 7.5, 10, 15, 20, 25 and 30 min at room temperature. First reading was considered to determine the instantaneous filtrate.

2.8. *Dynamic-aging test*

All aging tests were developed using a roller oven, OFITE at 91 °C for 960 min. After aging, the rheometric properties were determined by following the same procedure used for original fluids.

3. Theoretical study

3.1. *Carreau model*

The rheological data of the drilling fluids were fitted to Carreau rheological model. The Carreau model can be expressed as (Jang et al., 2015):

$$\eta = \eta_\infty + \frac{\eta_0 - \eta_\infty}{[1 + (\lambda_c \dot{\gamma})^2]^n} \tag{1}$$

where η_o (mPa.s) is the zero-shear viscosity, η_∞ (mPa.s) is the infinite-shear viscosity, $\dot{\gamma}$ (1/s) is the shear rate, λ_c is a constant parameter with the dimension of time, and n is the flow behavior index.

3.2. *Statistical analysis*

Data were analyzed by ANOVA using Statgraphics (Statgraphics Inc., Rockville, MD, USA). When differences between treatment effects were significant (p-value < 0.05), a multiple comparison of means was performed.

4. Results and discussion

The weight-average molecular weights ($\overline{M}_w$) and number-average molecular weights ($\overline{M}_n$) of polymers are presented in Table 2.

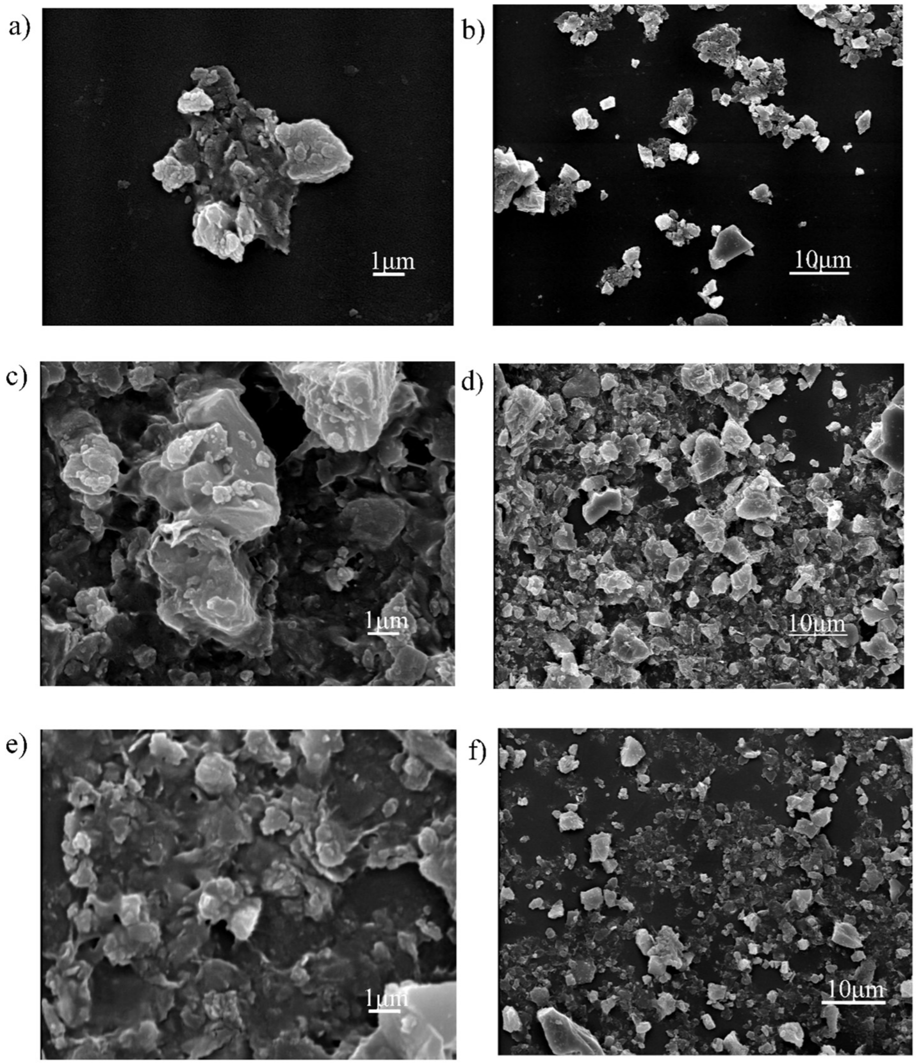

Fig. 8. Micrographs obtained by SEM: a–b) Mud 1; c–d) Mud 6; and e–f) Mud 10.

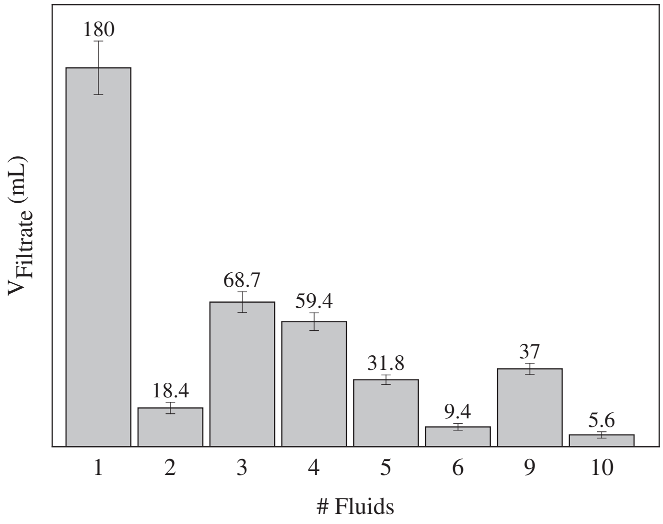

Fig. 9. Filtrate volume (mL) after 1800 s.

The bentonite is constituted mainly by smectite with quartz, feldspar and gypsum as impurities (Fig. 1). The detected crystalline phases were in concordance with similar previous studies (Volzone and Torres Sanchez, 1993; Tunç and Duman, 2008; Duman and Tunç, 2009; Bertagnolli et al., 2011; Ikhtiyarova et al., 2012; Choo and Bai, 2015; Andrini et al., 2017; Vryzas et al., 2017).

The FTIR spectrum of Na-Bentonite is shown in Fig. 2. The peak of Al–Al–OH stretching vibration at 3623 cm^{-1} is typical of smectites with high amount of Al in the octahedral layer. The peaks at 3447 and 1644 cm^{-1} correspond to the H—O—H stretching and bending vibrations of the adsorbed water, respectively.

The sharp peak at 1059 cm^{-1} was attributed to Si—O stretching frequency. Tetrahedral bending modes were observed for Si—O—Al at 514 cm^{-1} and for Si—O—Si at 454 cm^{-1}. OH bending vibrations of dioctahedral 2:1 layer silicates were assigned for Al—Al—OH at 913 cm^{-1}. Finally, a peak of quartz near 779 and 795 cm^{-1} was observed (Tunç and Duman, 2008; Bertagnolli et al., 2011; Ikhtiyarova et al., 2012; Tunç et al., 2012).

Tables 3 and 4 present a summary of the mineralogical and chemical composition of the samples. X-ray diffraction tests indicate that quartz is the main mineral and that all samples content clay. The most likely cause of shales instabilities is related to the clay mineral present in shales and its expansive characteristic when exposed to WBM. The proper selection of drilling fluids and its additives for a given situation is essential to reduce physico-chemicals interaction. The WBMs studied have different types of polymers aiming to prevent these interactions.

After testing different fluid compositions, a base mud was selected for this study exhibiting a composition optimized from the point of view of rheological characteristics after aging. The PAC and XGD concentrations of this base mud were: $C_{PAC} = 8.00$ g/L and $C_{XGD} = 1.50$ g/

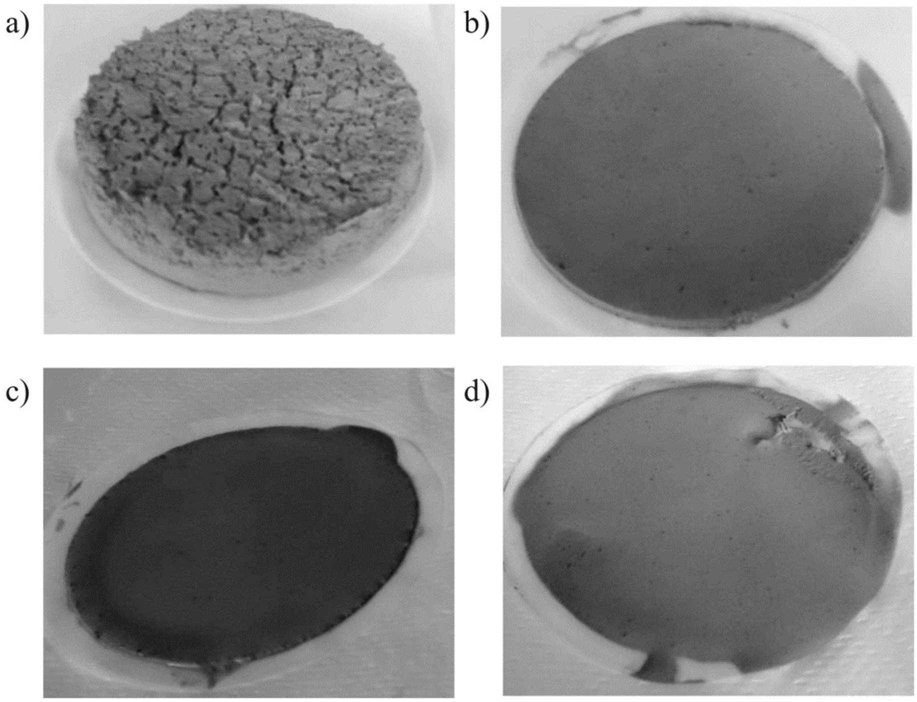

Fig. 10. Photographs obtained from filtration tests: a) Mud 1, b) Mud 2, c) Mud 10, and d) Mud 6.

Table 7
Parameters of the Carreau Model from analyzed drilling mud.

Fluid	η_0	η_∞	λ	n	Error (%)	S	R^2
1	6.50E + 04	7.83^a	8.77^a	0.675^f	0.28	0.03	0.99
2	6.29E + 04	34.53^d	46.75^d	0.37^b	0.17	0.24	0.99
3	7.23E + 04	16.75^b	$24.78^{b,c}$	0.44^d	0.06	0.01	0.99
4	7.51E + 04	22.19^c	$25.36^{b,c}$	0.49^e	0.12	0.02	0.99
5	7.21E + 04	30.32^d	45.94^d	$0.39^{b,c}$	0.17	0.26	0.99
6	8.06E + 04	32.25^d	27.16^c	0.37^b	0.01	0.01	0.99
7	7.34E + 04	$21.12^{b,c}$	37.85^d	0.33^a	0.01	0.01	0.99
8	3.96E + 04	23.19^c	$16.09^{a,b}$	$0.43^{c,d}$	0.03	0.01	0.99
9	8.06E + 04	24.61^c	$23.18^{b,c}$	$0.43^{c,d}$	0.13	0.15	0.99
10	5.40E + 04	45.02^e	59.57^e	0.31^a	0.03	0.02	0.99

Average values in the same column with different superscript letters are significantly different ($p < 0.05$).

L, respectively. On the basis of the selected base mud, nine fluids with different PAC and XGD compositions were prepared and are shown in Table 5. Solutions of 0.2 L for each fluid and replicas were prepared to obtain four measurements for each sample.

In Fig. 3, viscosity *vs* shear rate curves of aged OBM, aged base mud and unaged base mud (Fluid 2, Table 5) were compared. It was observed that both the aged and unaged base mud showed a relatively similar rheological behavior.

Figs. 4 and 5 show the rheometric experimental results of the analyzed drilling fluids. As the polymer concentration increases, a shear-thinning behavior and a larger yield stress are observed in all cases. Besides, a higher viscosifier effect for XGD is verified, also taking into consideration the lower concentration level of XGD. This characteristic can be associated to the branched molecular structure, the presence of hydrogen bonds and the higher molecular weight of the polymer.

In Figs. 4 and 5, it can be noted the non-linear dependence between the viscosity and polymer concentration, that is expected in this colloidal system. This behavior is associated to the physicochemical colloid-colloid and colloid-solvent interactions (both of long range and of short range) together with the hydrodynamic interaction energy imposed by the shear.

In Table 6, the results of plastic viscosity (calculated in the range of 300–600 rpm) are presented following standard API 13B1. The plastic viscosity for the base mud is very close to the expected value (approximately 20–30 mPa.s). Even using a lower concentration of XGD than PAC, an increase in the concentration of XGD had a significant effect on the plastic viscosity.

In Fig. 6, rheometric results after aging for base Mud 2, OBM and Mud 6 are presented. It is observed that aged mud 6 exhibits a rheological behavior very close to OBM. These results show a second level of optimization yielding an optimum composition of $C_{XGD} = 3.00$ g/L and $C_{PAC} = 8.00$ g/L for the analyzed concentrations.

In Figs. 7 and 8, micrographs of some prepared fluids obtained by optical microscopy and SEM are shown. The effects of the studied polymers on the structure of drilling fluids can be observed. In accordance with the rheometric analysis, a higher particle agglomeration was observed for fluids containing polymers (PAC, XGD). This effect is most noticeable for the fluid with XGD, confirming again their higher viscosifier effect.

Filtration results before aging are presented in Figs. 9 and 10. An important effect of polymers (PAC and XGD) on the filtering properties is observed. The results indicate a complementary action of polymer, XGD increases the filtrate viscosity and PAC seals the filter cake. It can be noted that higher concentrations of polymer are required to reduce the volume of filtrate under the appropriate values (< 10 mL), while the thickness of the plaster is generally thinner than 6 mm.

In order to identify and model the rheological behavior, different models were tested and compared with the experimental results. It was found that all muds follow the Carreau model for the studied conditions.

From the obtained experimental data, the rheological parameters of Carreau model were adjusted using an error minimizing routine. Parameters, correlation coefficient (R^2), experimental error and standard deviation (S) are presented in Table 7. The results show average values of the four experimental replicas performed for each sample.

In Fig. 5, the simulation and the experimental results are compared and a good agreement for theoretical and experimental apparent viscosity can be observed.

The parameters η_∞ and η_0 show a higher shear-thinning effect of PAC noticeable at the high concentrations. The highest behavior index corresponds to the mud without polymer that exhibits a tendency to Newtonian behavior.

5. Conclusions

A water based mud with rheological characteristics similar to the oil based mud used in drilling operation for Argentina shale was designed.

The effect of two commercial polymers on the main functional properties of fluids such as rheological and filtering properties, thermal stability and structural was evaluated. Rheometric analysis showed a shear-thinning behavior in the range of 17 to 1445 s^{-1}.

From the technological point of view an optimal polymers composition of water base mud ($C_{PAC} = 8.00$ g/L and $C_{XGD} = 3.00$ g/L) was found to assure properties similar to oil base mud.

The structural characterization indicated a high viscosifier effect of polymers associated to particle agglomeration more noticeable for xanthan gum. In addition, it was verified the performance of polyanionic cellulose as a good filtration control agent.

Rheological parameters were obtained by following the Carreau model. The adjusted model can be used to obtain a better optimization of the drilling fluid composition.

In a future work, other additives and/or components of drilling fluid will be investigated in order to study the relationships between composition and physicochemical characteristics of the fluid.

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