



Di-carboxylic acid cellulose nanofibril (DCA-CNF) as an additive in water-based drilling fluids (WBM) applied to shale formations

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Abstract This work proposes the application of di-carboxylic acid cellulose nanofibril (DCA-CNF) obtained through maleic acid hydrolysis as an additive in water-based drilling fluids (WBM). Specifically, the use of DCA-CNF as a replacement of xanthan gum (XGD) in the WBM formulations was evaluated. The effect of DCA-CNF on the main functional properties of WBM and their performance was evaluated and compared with that corresponding to XGD. To this end, interactions between DCA-CNF and bentonite (BT), as well as between DCA-CNF and polyanionic cellulose (PAC), were studied using quartz crystal microbalance with dissipation monitoring (QCM-D)

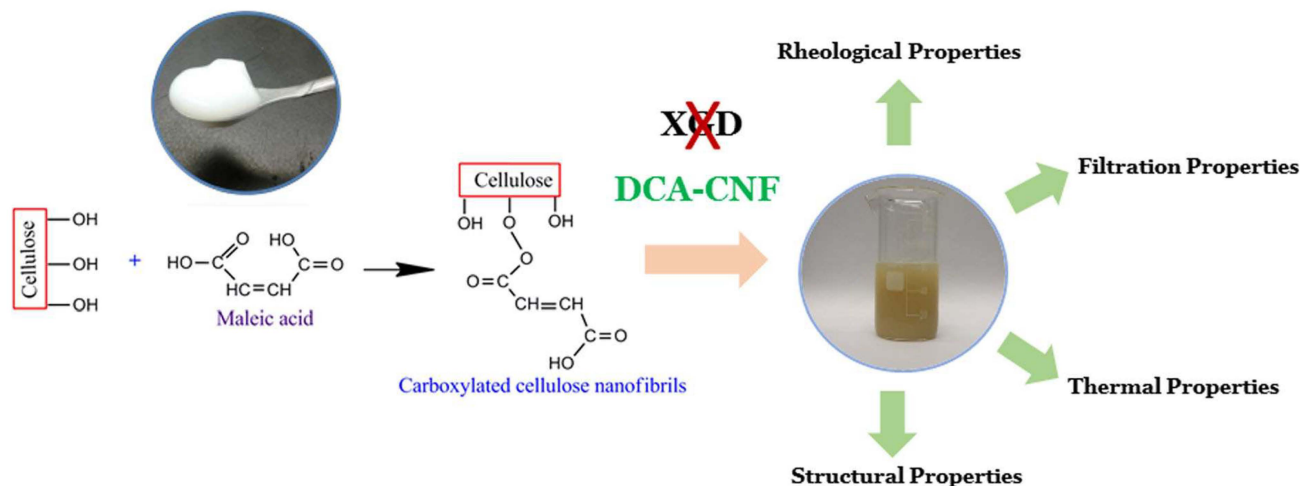
technique. The rheological analyses showed a shear-thinning behavior of WBM containing XGD similar to WBM with DCA-CNF, while filtration properties and thermal stability improved by the presence of DCA-CNF. Results obtained by QCM-D indicated higher interaction between PAC and DCA-CNFs when compared to BT and DCA-CNF. The Sisko model was implemented to simulate the relationship between viscosity and shear rate. WBM for Argentina shale containing the double concentration of DCA-CNF exhibited similar rheological properties to the base fluid.

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Graphic abstract



Keywords Di-carboxylic acid cellulose nanofibril · Maleic anhydride hydrolysis · Water-based drilling fluid · Surface interactions · Quartz crystal microbalance · Shale · Vaca Muerta

Introduction

Cellulose nanomaterials (CNM) such as cellulose nanocrystals (CNCs) and cellulose nanofibrils (CNFs) are products derived from cellulose, which have at least one dimension in the nanometer range. CNM are obtained through various chemical and mechanical processes (Zhu et al. 2016). Their properties have attracted great attention in several fields, such as composites (Kargarzadeh et al. 2017; Ng et al. 2017), energy devices (Du et al. 2017), packing, films and coatings (Ferrer et al. 2017; Hubbe et al. 2017; Li et al. 2015a), aerogels and hydrogels (De France et al. 2017; Nascimento et al. 2018), biodegradable composites (Sharma et al. 2019), water remediation (Mahfoudhi and Boufi 2017; Voisin et al. 2017), medicine applications (Lin and Dufresne 2014; Seabra et al. 2018), three-dimensional (3D) printing (Dai et al. 2019), printed electronics (Hoeng et al. 2016), food additives (Gómez et al. 2016; Sanchez-Salvador et al. 2019), and automotive industries (Ferreira et al. 2019), among others.

CNFs can be isolated through several mechanical processes, such as high-pressure homogenization, microfluidization, refiners, or grinding (Moon et al.

2011). Other less-used mechanical or thermomechanical treatments are cryocrushing, electrospinning, ultrasonication or steam explosion (Blanco et al. 2018; Khalil et al. 2012; Lavoine et al. 2012; Nechyporchuk et al. 2016). The main disadvantage of the mechanical processes is the high energy consumption (Wang et al. 2012). Therefore, different chemical and enzymatic pretreatments of pulp have been implemented to ease the mechanical treatment, reducing the energy consumption and improving the nanofibril properties (Blanco et al. 2018; Isogai et al. 2011; Moon et al. 2011; Saito et al. 2006; Zhu et al. 2016).

CNCs are mainly produced by acid hydrolysis of bleached cellulosic fibers of a variety of sources using concentrated mineral acids, a process developed in the 1950s (Rånby 1951; Mukherjee and Woods 1953; Wang et al. 2012, 2013). The disadvantages of this treatment are related to the acid recovery and the disposal of a large amount of salt used for acid neutralization, the surface functionality of sulfuric acid CNCs, as well as the low thermal-stability of the resultant CNC, and the low yield of the overall process (Chen et al. 2015; Roman and Winter 2004; Wang et al. 2012).

To address these issues, solid di-carboxylic acids (DCA) such as maleic and oxalic acid, have been used as a more sustainable and better alternative for producing CNCs. These acids are recoverable after hydrolysis treatment (Bian et al. 2017a, b; Chen et al. 2016; Jia et al. 2017; Wang et al. 2017). As di-carboxylic acids, they carboxylate CNCs through

Fischer-Speier esterification of one carboxyl group. However, the low acidity ($pK_a = 1.00 - 3.00$) resulted in low CNC yields (Chen et al. 2016). Fortunately, the remaining partially hydrolyzed fibrous cellulosic residue can be used to produce carboxylated CNF through mechanical fibrillation with minimal energy input, resulting in a high overall CNM yield and minimal cellulose loss. Therefore, DCA-CNF represents a new class of CNM that can be sustainably produced at high yield with high thermal stability and great surface charge and functionality.

The application of CNF as an additive in drilling fluids for hydrocarbon extraction is an environmentally friendly alternative and has been recently investigated (Hall et al. 2017, 2018; Li et al. 2015b, c; Song et al. 2016a, b; Villada et al. 2018). Drilling fluids are mixtures of natural and synthetic chemical components used to cool and lubricate the bit and the drill string, clean the hole bottom, carry cuttings to the surface, control formation pressures, among others (Fink 2015). An efficient drilling fluid must also present several characteristics, such as desirable rheological properties, fluid loss prevention, stability under high-temperature and high-pressure conditions, as well as stability against contaminating fluids such as salt water, calcium sulfate, cement, and potassium (Caenn et al. 2017; Fink 2015; Hossain and Al-Majed 2015). According to the nature of the continuous phase, fluids are classified into two main groups, water-based drilling fluids (WBM) and oil-based drilling fluids (OBM). OBMs are known to provide unequalled performance but they are subject to environmental regulations. WBMs present low environmental impact but exhibit some disadvantages associated with shale inhibition, lubricity, and thermal stability. To avoid these drawbacks, specific additives can be added to deliver properties close to OBMs performance while minimizing the environmental impact (Fink 2012). Actually, several additives have been added to the WBMs formulations to improve their main functional properties. Among them, natural polymers (Caenn and Chillingar 1996), synthetic polymers (Nunes et al. 2014), and inorganic nanoparticles (Vryzas et al. 2017) have been widely studied.

Hall et al. (2017), Li et al. (2015a, b), and Song et al. (2016a, b) evaluated the utilization of CNC and CNF as additives in WBMs. The results showed that the addition of these nanomaterials improved the rheological and filtration properties of the studied WBMs.

In our previous work (Villada et al. 2018), the replacement of XGD using two types of CNF with different chemical compositions, bleached and unbleached, was evaluated. The performance of WBMs containing CNF was comparable to the traditional WBMs. It was observed that the rheological and filtration properties of WBMs were affected by the structural characteristics (dimension, shape, surface charge), the rheological properties, and the chemical composition of the CNF.

Here we investigate DCA-CNF as an additive in WBMs for shale of Argentina, specifically for the partial or total replacement of XGD. Nanofibrils are characterized by scanning electron microscopy (SEM), thermogravimetric analysis (TGA), polyelectrolyte and conductometric titration, and steady shear rheological assays. The effect of the additives on the main functional characteristics of the WBMs is evaluated. In addition, interfacial interactions between some WBMs additives are studied through QCM-D (Quartz Crystal Microbalance with Dissipation Monitoring). Finally, specific studies on WBMs for shale formations of Argentina are provided.

Materials and methods

Production of DCA-CNF

DCA-CNF was produced from bleached kraft eucalyptus pulp using maleic acid hydrolysis at 70 wt.% and 100 °C for 120 min at the USDA Forest Products Laboratory, Madison, Wisconsin. After the separation of CNC, the hydrolyzed fibrous solid residue was directly fed into a pilot scale homogenizer (GEA Noro Soavi Ariete NS3015, GEA, Düsseldorf, Germany) for 3 passes of nanofibrillation. The detailed description of the production and characterization of the DCA-CNF can be found in a prior study (Wang et al. 2020, labelled as M2-3P). The final dry content of the DCA-CNF suspension as received was 2.6 wt.%.

Characterization of DCA-CNF

Morphology

Morphology of the samples was studied by two different techniques, Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM).

AFM measurements were performed in tapping mode at a constant force of $12\text{--}110\text{ Nm}^{-1}$ and resonance frequency of $210\text{--}490\text{ kHz}$ using silicon cantilevers (ATEC-NC-20, Nanosensors, Germany). Analyses were carried out using an Agilent 7500 AFM multimode scanning probe microscope (Keysight Technologies, Santa Barbara, USA). Acquired images sizes were $2.5\text{ }\mu\text{m} \times 2.5\text{ }\mu\text{m}$ and $5\text{ }\mu\text{m} \times 5\text{ }\mu\text{m}$, and they were processed using the Gwyddion software 2.49 (SourceForge).

SEM analyzes were performed in a scanning electron microscope (Carl Zeiss Supra 35VP, Germany). Samples were placed over a quartz piece; sputter coated with palladium and examined using an accelerating voltage of 1 keV at a working distance of approximately 4.5 mm .

Steady shear rheological behavior

Rheological properties of DCA-CNF aqueous suspensions (0.5 wt.%) were measured using a viscometer DV3TRV (Brookfield, USA) with a cone-plate geometry (CP-51Z with 2.4 diameter, and 1.56° angle) at $25\text{ }^\circ\text{C}$. Before each viscosity assay, the suspensions were pre-sheared at high shear rate (960 1/s). The flow curves were performed in duplicate.

Thermal stability

Thermal stability of DCA-CNF was evaluated through thermogravimetric analysis (TGA). The TGA was performed using a Q500 equipment from TA Instruments (Delaware, USA). For this analysis, 5 mg of DCA-CNFs were heated from 10 to $800\text{ }^\circ\text{C}$ with a heating rate of $10\text{ }^\circ\text{C/min}$ under nitrogen atmosphere.

Fourier transform infrared spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FTIR) analysis was implemented to verify the DCA-CNF esterification by maleic acid hydrolysis. The FTIR analysis was carried out with a Shimadzu FTIR-8201 PC Spectrometer (Kyoto, Japan) within the range of $4000\text{--}400\text{ cm}^{-1}$. The samples for FTIR were mixed with KBr.

Surface charge density and zeta potential

Charge densities of DCA-CNF suspensions were determined by the polyelectrolyte titration method. To this effect, the procedure reported by Villada et al. (2018) was followed. Zeta potential analysis was carried out with a Zetasizer Nano-series Malverl (ZS90, UK) at $\text{pH } 5.0$. Prior to analysis, the DCA-CNF suspensions were diluted (0.1 wt.%) in deionized water and sonicated for 10 min . The mean zeta potentials were averages of 5 measurements.

Carboxylation and degree of substitution of DCA-CNF

Conductometric titration of the DCA-CNF suspension was used to determine the carboxyl group content. The analysis was performed with an automatic titrator (AT 510-KEM, Japan) and according to the procedure described in Wang et al. (2017). A suspension of DCA-CNF was added to 1 mM NaCl solution and titrated using 2 mM NaOH by adding approximately 0.2 mL in 30 s intervals.

The degree of substitution (DS) was determined by the following equation (Stojanović et al. 2005; Buffa et al. 2016; Chen et al. 2016; Zhang et al. 2012; Zhong et al. 2012):

$$DS = \frac{162 \times n_{\text{COOH}}}{m - 116.1 \times n_{\text{COOH}}} \quad (1)$$

where 162 (g/mol) is the molar mass of the anhydroglucose unit (AGU), n_{COOH} is the molar number of carboxyl groups (mol), m is the sample weight (g), and 116.1 (g/mol) is the net increase in the mass of an AGU for each maleic acid substituted (g/mol).

WBMs preparation

In order to evaluate the replacement of the XGD additive in WBMs, both simple and complex WBMs for Argentina shale (Vaca Muerta) were studied. All WBMs components were provided by MI Swaco Company (A-H components are not reported for confidential reasons). To study the interactions between additives, the zeta potential of XGD and PAC was measured following the same procedure used for DCA-CNF characterization (“Characterization of DCA-CNF” section).

Simple WBMs

Two different simple systems were studied: S-DCA-CNF (BT/DCA-CNF/PAC/H₂O) and S-XGD (BT/XGD/PAC/H₂O). For each system, several fluids were designed, and the formulations are presented in Table 1. Note that for comparative purposes, the fluids containing XGD previously reported in Villada et al. (2018) have been used in this study.

Simple drilling fluids were prepared using a mechanical mixer (Kinematica Polytron PT 2500e, Switzerland), following API Standard (API Recommended Practice 13B-1, 2003) and the methodology of preparation of Villada et al. (2017). The following steps were conducted: (1) water and BT were added to a 500 ml vessel and stirred at 400 rpm for 30 min; (2) the BT suspension was allowed to stand for 16 h at room temperature; (3) the DCA-CNF or XGD were added and the mixtures were stirred at 400 rpm for 10 min; and (4) the PAC was added and the suspension was stirred at 400 rpm for 10 min.

Complex WBMs

A base fluid containing XGD, whose composition is indicated in Table 2, was designed. For its preparation, the API standard recommendations and a pre-established order of addition were followed. The procedure consisted of 5 steps: (1) water and BT were added to a 500 ml vessel and stirred at 400 rpm for 30 min; (2) the BT suspension was allowed to stand for 16 h at room temperature; (3) PAC, XGD, inhibitor, and encapsulator were added and the mixture was stirred at 400 rpm for 10 min; (4) a

lubricant, two plugging agents and a densifier were incorporated and the suspension was stirred at 400 rpm for 10 min; and (5) the pH was adjusted to 10, additives to simulate the solids in suspension were added and the suspension was stirred at 400 rpm for 5 min.

The base fluid exhibited rheological, thermal, and filtration properties similar to that of OBM used in Argentina shale. Also, some additional WBMs containing 0.15, 0.30 and 0.45 wt.% (named A, B, and C, respectively) for Argentina shale (Vaca Muerta) were prepared. To this effect, the base fluid preparation procedure was followed replacing XGD by DCA-CNF.

WBMs characterization

Rheological properties

Steady shear Steady shear rheology was measured using the same viscometer and the procedure described above for the aqueous suspensions of DCA-CNF. In addition, the Sisko model was applied to predict the rheological parameters.

Sisko model:

$$\eta = \eta_{\infty} + k\dot{\gamma}^{n-1} \quad (2)$$

where η_{∞} is the viscosity at infinite shear rate, k is the flow consistency coefficient at low shear rate, $\dot{\gamma}$ is the shear rate, and n is the flow behavior index.

Oscillatory shear The viscoelastic properties of WBMs were studied through oscillatory rheology. The assays were performed in a rheometer Haake

Table 1 Compositions of simple WBMs: S-DCA-CNF and S-XGD systems

Systems S-DCA-CNF or S-XGD	BT (wt.%)	DCA-CNF or XGD (wt.%)	PAC (wt.%)
1DCA-CNF or 1XGD	0.00	0.50	0.50
2DCA-CNF or 2XGD	1.00	0.50	0.50
3DCA-CNF or 3XGD	3.00	0.50	0.50
4DCA-CNF or 4XGD	4.50	0.50	0.50
5DCA-CNF or 5XGD	6.00	0.50	0.50
6DCA-CNF or 6XGD	4.50	0.00	0.50
7DCA-CNF or 7XGD	4.50	0.10	0.50
8DCA-CNF or 8XGD	4.50	0.25	0.50
9DCA-CNF or 9XGD	4.50	0.50	0.00
10DCA-CNF or 10XGD	4.50	0.50	0.10
11DCA-CNF or 11XGD	4.50	0.50	0.25

Table 2 Base fluid formulation

Order of addition	Component	Concentration (wt.%)	Function
1	Water		Base
	Na-Bentonita		Viscosifier and filtration control agent
2	(PAC)	0.8	Filtration control agent
	(XGD)	0.15	Viscosifier
	A		Encapsulator
	B		Inhibitor
3	C		Lubricant
4	D, E		Filtration control agent
	F		Densifier
5	G		pH control
6	H		Simulator of cuttings

RheoStress RS80 (Haake Instruments Inc., Paramus, NJ, USA) with a cone-plate geometry (60-mm diameter, 1° angle). To avoid the WBM's evaporation, silicone oil was added on the edge of WBM's.

Frequency sweep tests were performed from 0.01 to 10 Hz within the linear viscoelastic region at strains of 0.02, and room temperature. The linear viscoelastic region was determined through strain sweep tests from 0.01 to 0.10 at 10 Hz. The test was repeated two times. Prior to each assay, the WBM's were vigorously stirred at 5000 rpm for 3 min.

Filtration properties

Filtration properties of WBM's were determined following the API Recommendation (American Petroleum Institute 2003). A filter press (OFITE, Low Pressure, Houston, TX, USA) was used, containing filter paper (Whatman N° 50), and CO₂ as pressurizing gas at 6.80 atm. Measurements were performed at room temperature. Filtrate readings were monitored for a 30-minute period. The filtration rate was measured following the method described by Li et al. (2015b) and Villada et al. (2018).

The permeability of filter cake was calculated from the Darcy Law:

$$K_C = \frac{\mu t_c dv}{\Delta P A dt} = \frac{\mu t_c q}{\Delta P A} \quad (3)$$

where μ is the viscosity of the filtrate at 25 °C (1.00 cP), t_c is the thickness of filter cake (cm), ΔP is the pressure difference (6.80 atm), A is the cross section area (44.16 m²) and q is the filtrate rate (cm³/s).

Thermal stability: dynamic-aging test Thermal stability of WBM's was determined through an aging test using a roller oven, OFITE (Houston, TX, USA) at 91 °C for 16 h. In this assay, 100 mL of fluid were added in a stainless steel cell and placed in the oven, under continuous rotation. After thermal treatment, the rheological properties were determined using the methodology previously described for the fluids before treatment. The aging results were complemented with the morphological and structural study.

Structural characterization The microstructure of WBM's was observed by Scanning Electron Microscopy (Phenom World PRO X, Themofisher Scientific, USA). Samples were prepared by drying drops of the WBM's on aluminum mounts. All samples were sputter-coated with gold under argon atmosphere (SPI Supplies, 12157-AX) to provide adequate conductivity. The acceleration voltage applied was 15 kV.

Surface interactions studies

The interfacial interactions between BT and the different components of the WBM were studied using a quartz crystal microbalance with dissipation monitoring (QCM-D) (QSense Analyzer model QE401 from Biolin Scientific, Västra Frölunda, Sweden).

To evaluate the interactions between DCA-CNF/BT and DCA-CNF/PAC systems, a two stage-procedure was implemented. First, DCA-CNFs films (model surface) were prepared on a silicon coated quartz sensor. Then, QCM-D analysis was performed from measurements of the change in frequency and dissipation of a quartz crystal (Nypelö et al. 2012; Hall et al. 2018).

DCA-CNF model films preparation

DCA-CNF films were produced on the surface of the silicon-coated quartz sensor by spin coating. To this effect, DCA-CNFs suspensions (0.5 wt.%) were sonicated for 2 min at 25% amplitude; and then centrifuged at 10,000 rpm for 40 min. The supernatant was used to make the films by deposition onto the sensors. Prior to the supernatant deposition, the following procedure was performed: (1) silicon-coated quartz sensors were rinsed with Mili-Q water, dried with N₂ gas, and placed in a UV-ozonizer for 30 min at 25 °C; (2) the silicon-coated quartz sensors were rinsed with Mili-Q water and dried with N₂ gas; (3) the silicon-coated quartz sensors were pretreated by immersion in an anchor solution of polyethylene imine (0.1 wt.%) for 15 min; and (4), and the sensors were spin coated with ultrapure water for 30 s at 3000 rpm. Finally, 100 µL of DCA-CNF supernatant was deposited onto the surfaces of the sensor for 1 min at 3000 rpm and the resulting DCA-CNF films were left to dry in an oven for 20 min at 80 °C.

QCMD analysis

QCM-D measurements were performed at room temperature and maintained at a constant flow of 150 µL/min. DCA-CNF coated sensors were first stabilized in DI water and then suspensions containing BT or PAC at 1 and 0.5 wt.% respectively, were flowed through the QCM-D channels. Finally, systems were rinsed with DI water to observe the adhesion of

BT and PAC onto the DCA-CNF films. Measurements were performed by duplicate. In this paper, only the frequency change (Δf) in the 3rd overtone ($n = 3$) is discussed.

Substrates characterization

AFM was used to characterize the substrates after the BT and PAC adsorption. The analysis was performed in tapping mode with a Bunker (formerly Digital Instruments, Veeco) AFM Dimension 3100 (California, US). Images were obtained at 2.35 Hz of amplitude velocity with a Nano World (Innovative Technologies) FM 20 silicon SPM-sensor cantilever with a resonance frequency of 75 kHz and force constant of 2.8 N/m. Measurements were carried out at room temperature and images processed using the freeware Gwyddion software 2.49 package (SourceForge).

Results and discussion

DCA-CNF characterization

Morphology

The micrographs obtained by SEM and AFM are presented in Figs. 1 and 2, respectively. It is possible to observe nanofibrils homogeneous and evenly distributed on the surface. Furthermore, the fibrils are a few hundred nanometers in length and show small diameters. In general, they exhibit a lower aspect ratio than the conventional CNFs (Chen et al. 2016). Therefore, these nanofibrils can be considered CNF “whisker” particles (Anderson et al. 2014; Chen et al. 2016).

Surface charge density and zeta potential

Charge density and zeta potential results of DCA-CNF are -101.00 ± 0.35 µeq/g and -36.21 ± 1.40 mV, respectively. DCA-CNF presents negative surface charges due to the carboxylation through esterification by maleic acid hydrolysis. These results are consistent with those reported by Chen et al. (2016) and Wang et al. (2020), which observed increases in the surface charge with the concentration of maleic acid as a consequence of the reactivity of the hydroxyl groups

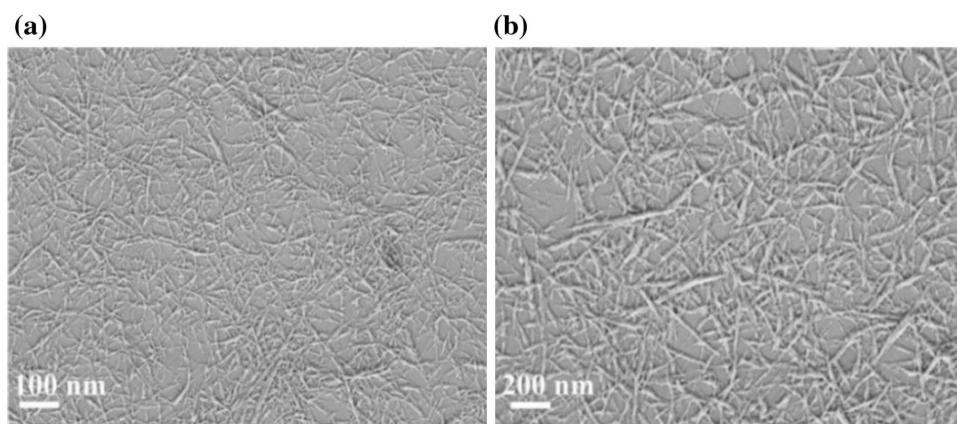


Fig. 1 Micrographs of DCA-CNF obtained by SEM: **a** 60 KX and **b** 150 KX

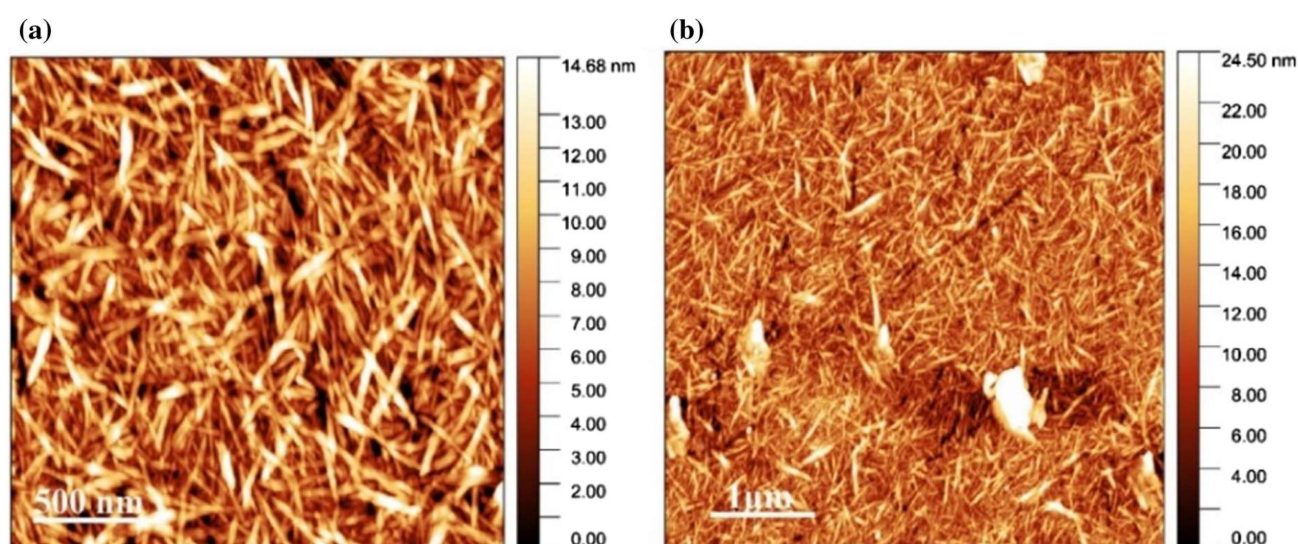


Fig. 2 AFM topographic images of the DCA-CNF samples obtained by AFM: **a** 60 KX and **b** 150 KX

during the cellulose esterification. Additionally, an absolute value of the zeta potential, lower or higher than 25 mV is indicative of suspensions stability, promoting flocculation and deflocculation, respectively (Mirhosseini et al. 2008; Morais et al. 2013).

Thermal stability

The thermal stability of additives is very important, due to the high-temperature conditions during drilling operation. Unlike sulfuric acid hydrolysis-produced CNC or oxidation-produced CNF such as TEMPO CNF, the DCA process produces highly stable materials, as previously reported. This behavior is associated with the use of the maleic acid in the CNCs isolation as reported by Chen et al. (2016).

Thermograms for DCA-CNF are shown in Fig. 3. The onset degradation temperature of DCA-CNF is 203 °C, the temperature at 5% weight loss is 249 °C, the peak temperature is 323.8 °C, and a residual weight of 1.47 g at 698 °C. These results indicate an improved thermal stability of DCA-CNF respect to XGD exhibits a peak temperature of 268 °C and a lower residual weight (Hall et al. 2017).

Carboxylation and degree of substitution

The amount of carboxyl groups was calculated from the consumed NaOH corresponding to the inflection point in the curve of conductivity versus consumed volume following the methodology proposed by Wang et al. (2017). The carboxyl group content was

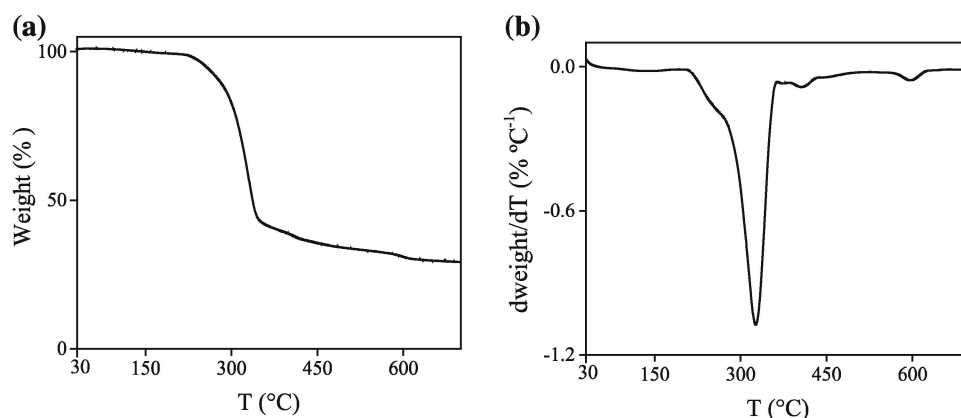


Fig. 3 Curves of DCA-CNF from **a** TGA and **b** DTG analyses

0.17 mmol per g DCA-CNF, which indicates a moderate carboxylation of DCA-CNFs. This result is consistent with that reported by Wang et al. 2020. The DS of DCA-CNF was determined by conductometric titration and using Eq. (1). The DS calculated by this method was 0.03, which is associated with the moderate carboxylation of DCA-CNF.

FTIR analysis

The FTIR spectrum of DCA-CNF is shown in Fig. 4. The peak at 3340 cm^{-1} corresponds to O–H stretching vibrations of hydroxyl groups. The peaks at 2895 and 1631 cm^{-1} are associated with C–H stretching vibration and O–H bending vibration of the absorbed water in cellulose, respectively. The signals at 1431 , 1367 and 1056 cm^{-1} are attributed to $-\text{CH}_2$ and $-\text{CH}$ bending, and vibration of C–O–C in a pyranose ring, respectively. Finally, a peak of β -glycosidic linkages

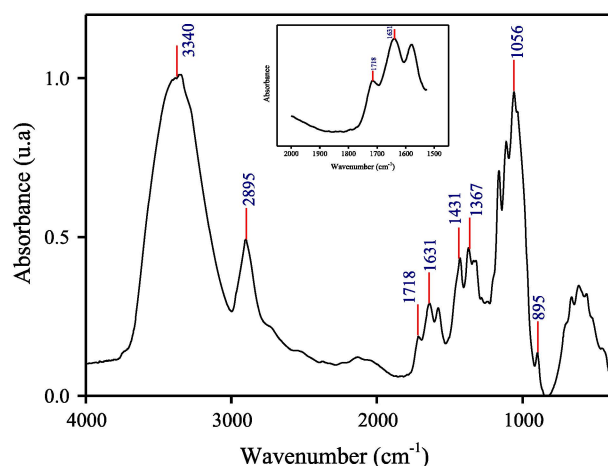


Fig. 4 FTIR absorption spectrum of DCA-CNF

at 895 cm^{-1} is observed (Chen et al. 2016; Dufresne 2017; Suhas et al. 2016).

A weak band is observed around 1718 cm^{-1} , corresponding to the vibrations of the carbonyl group ($\text{C}=\text{O}$). This suggests that one of the carboxyl groups of the maleic acid reacted with cellulose (most likely at C6 position) to form ester groups on DCA-CNF (Bian et al. 2017b; Chen et al. 2016). Nevertheless, the peak at 1718 cm^{-1} was weak, suggesting moderate degrees of esterification. This observation is in agreement with previous works (Bian et al. 2017b; Chen et al. 2016). Furthermore, the FTIR qualitative characteristics at 1718 cm^{-1} are consistent with the amounts of carboxyl groups determined by conductometric titration (Bian et al. 2017b).

Steady shear rheological behavior

Viscosity *versus* shear rate curves of DCA-CNF aqueous suspensions were obtained that indicated a shear-thinning behavior. Figure 5 shows the results for an aqueous suspension of 0.5 wt.% DCA-CNF that corresponds to the highest concentration used for WBM formulations (Table 1). The higher values of the charge density and zeta potential promote the formation of stabilized suspensions with DCA-CNF particles well dispersed to align under stress conditions. DCA-CNF exhibits slightly lower viscosities than the values reported for a different CNM reported in our previous work (Villada et al. 2018).

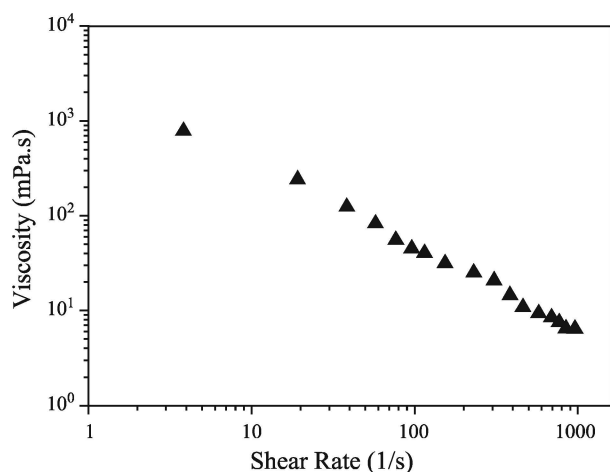


Fig. 5 Viscosity versus shear rate curve of DCA-CNFs

WBMs characterization

Steady-shear rheological properties

The results of rheological properties for simple WBMs (See Table 1) are presented in Fig. 6. All fluids exhibit a shear-thinning behavior. As the concentration of additives increases, higher pseudoplasticity, yield stress, and viscosifier effects are observed. Note that

the fluids without bentonite (1DCA-CNF, 1XGD) also present shear thinning behavior, showing the potential properties of DCA-CNF/PAC or XGD/PAC additives as rheological modifiers Li et al. (2015b). Furthermore, the effects of DCA-CNF, XGD, and PAC are more significant at high additive concentration, indicating a non-linear dependence (Fig. 6b, c, e, f).

Furthermore, when analyzing the effect of the polymers (DCA-CNF, XGD, or PAC) on the rheological behavior, it is observed that at the same concentration, the fluids with varying DCA-CNF and XGD amounts (Fig. 6b, e) exhibit slightly higher viscosity values than those with varying PAC concentration (Fig. 6c, f). This behavior can be explained by the surface interactions occurring between polymers and BT layers in combination with the properties of each polymer.

BT is a mineral clay composed of superimposed montmorillonite (MMT) platelets with two types of surface charge. In general, the MMT platelets have a negative charge on the flat surface; and negative, neutral, or positive charge on their edge, which depends upon the pH value. Therefore, three possible association modes (face-to-face, face-to-edge, and edge-to-edge) can be produced between adjacent

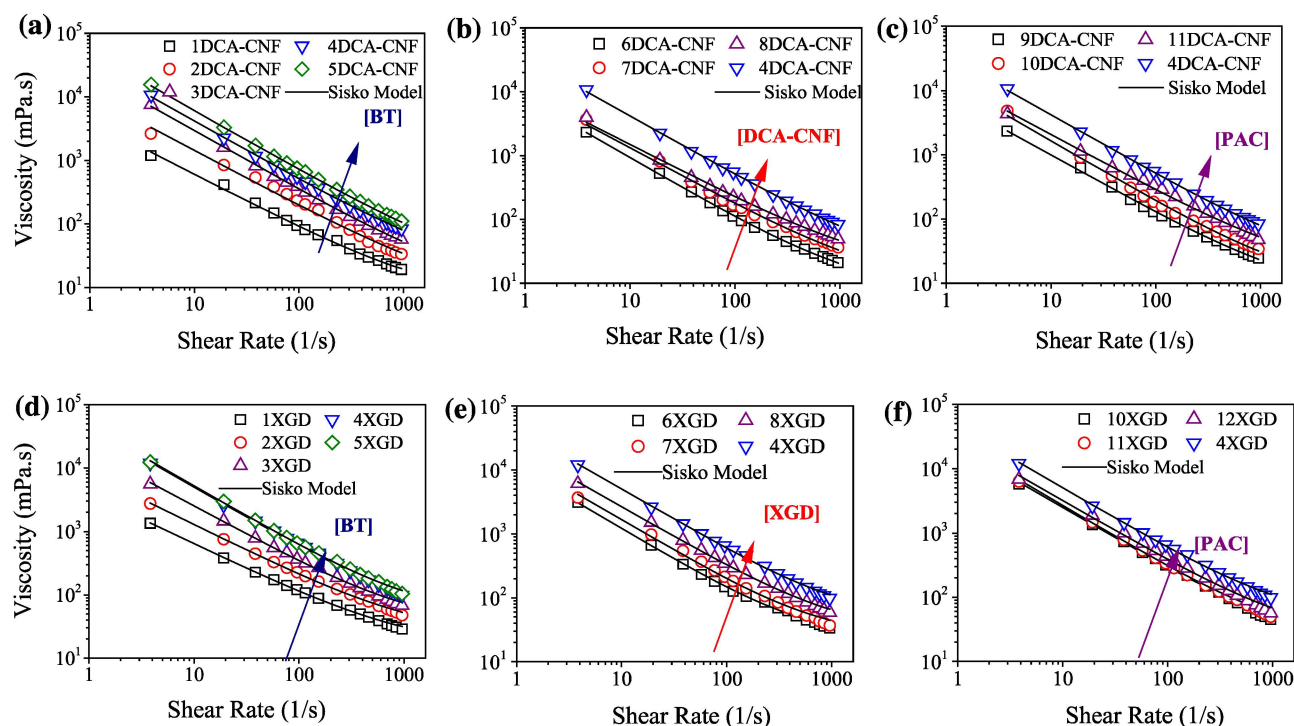


Fig. 6 Viscosity versus shear rate curves for S-DCA-CNF (a, b, c) and S-XGD (d, e, f) systems. Effect of the additive concentration of: BT (a, d); DCA-CNF or XGD (b, e); PAC (c, f). Simulated curves with Sisko model are indicated in solid lines

MMT particles through Van der Waals and electrostatic repulsion/attraction forces (Li et al. 2015b, c, 2018).

In particular, in a neutral condition—as is the case corresponding to the WBM's preparation—a positively-charged edge could also be created due to the exposed octahedral Al layers. This hypothesis is in agreement with the previous studies (Li et al. 2015b, c, 2018).

When BT and the polymers (DCA-CNF, XGD, or PAC) are in an aqueous solution, they could be attached to the positive edge of the BT layers by electrostatic attraction. Simultaneously, some repulsive forces could appear on the face of BT (Li et al. 2015b, c). Therefore, a change in fluid viscosity is expected (Caenn et al. 2017).

From the rheological behavior analysis for the S-DCA-CNF system as a function of DCA-CNF or PAC concentrations, a slightly higher impact in viscosity of DCA-CNF concentration is observed (Fig. 6b, c). This result is a consequence of a combination of different characteristics of the polymers, such as the particle size, surface charge, and functionalization, among others. DCA-CNF and PAC are negatively charged polymers due to the presence of carboxylic groups. As evaluated previously, the zeta potential values for DCA-CNF and PAC were -36.21 ± 1.4 mV and 48.70 ± 0.5 mV, respectively. The higher superficial charge of PAC induces higher repulsive forces with BT particles resulting in a reduced fluid viscosity. Furthermore, the carboxyl groups in DCA-CNF from maleic acid ($pK_a = 2.0$) hydrolysis are preferentially more ionized in solution than the carboxyl groups of PAC ($pK_a = 4.8$) (Li et al. 2015b; William et al. 2014). Thus, the electrostatic attractions between the DCA-CNF and BT are stronger than those between the PAC and BT. In addition, the expected greater effect on WBM viscosity by PAC with a higher substitution degree (0.65–0.9) (Sigma Aldrich, CAS number: 9004-32-4) than by DCA-CNF with a lower substitution degree (0.01) was not experimentally observed due to the intrinsic characteristics such as particle size, crystalline structure, hydrodynamic size, and nanoscale dimension.

Similar results are also observed when comparing the effect of varying XGD or PAC concentrations in the S-XGD system (Fig. 6e, f). The superficial charge of XGD was 36.10 ± 0.9 mV, inducing lower

repulsive forces with BT particles, and a slightly higher impact on viscosity (Fig. 6d, e).

In general, the rheological behavior of S-DCA-CNF (Fig. 6a–c, 7a–c) and S-XGD (Fig. 6d–f) systems are comparable due to the similar molecular structures between XGD and DCA-CNF. XGD is a high molecular weight exo-polysaccharide with polymeric branches. Chemically, it is composed of D-glucosyl, D-mannosyl, and D-glucuronyl acid residues in a 2:2:1 molar ratio with varying proportions of O-acetyl and pyruvyl residues (Kumar et al. 2018). Furthermore, the backbone structure of XGD is a linear (1-4)-linked D-glucose, similar to that of cellulose.

Moreover, the DCA-CNF used in this work has a higher surface charge (-101.00 ± 0.35 $\mu\text{eq/g}$) than the CNM previously studied (Villada et al. 2018). However, the WBM's for the same composition at high concentrations of DCA-CNF only exhibited slightly higher viscosities. These results indicate that other intrinsic properties of DCA-CNF may also contribute to the viscosity of WBM's.

Correlation coefficients (R^2), experimental errors, and estimated parameters obtained with the Sisko model are presented in Table 3. The flow behavior index (n) indicates a non-Newtonian trend of the WBM. As expected, the consistency index (k) and the viscosity at infinity (η_∞) increased with the concentration of the additives. This behavior is more noticeable with the increases in BT. It is also observed good agreement between the results simulated from the Sisko model and experimental data (Fig. 6).

Oscillatory shear-behavior

Based on the previous results of the effect of the concentration of BT and polymers on the rheological properties, two fluids were selected for this study: 4DCA-CNF and 4XGD, corresponding to S-DCA-CNF and S-XGD systems. The additive concentrations are shown in Table 1.

Frequency sweep results for fluids 4DCA-CNF and 4XGD are presented in Fig. 7. It is observed that the WBM's exhibit a viscoelastic behavior, more noticeable for the fluid 4DCA-CNF. Furthermore, the elastic module (G') is higher than the viscous module (G''), indicating dominant elastic properties and gel features. In the range of frequency evaluated (0.01–10 Hz), G' and G'' are constant, which indicates that the WBM's

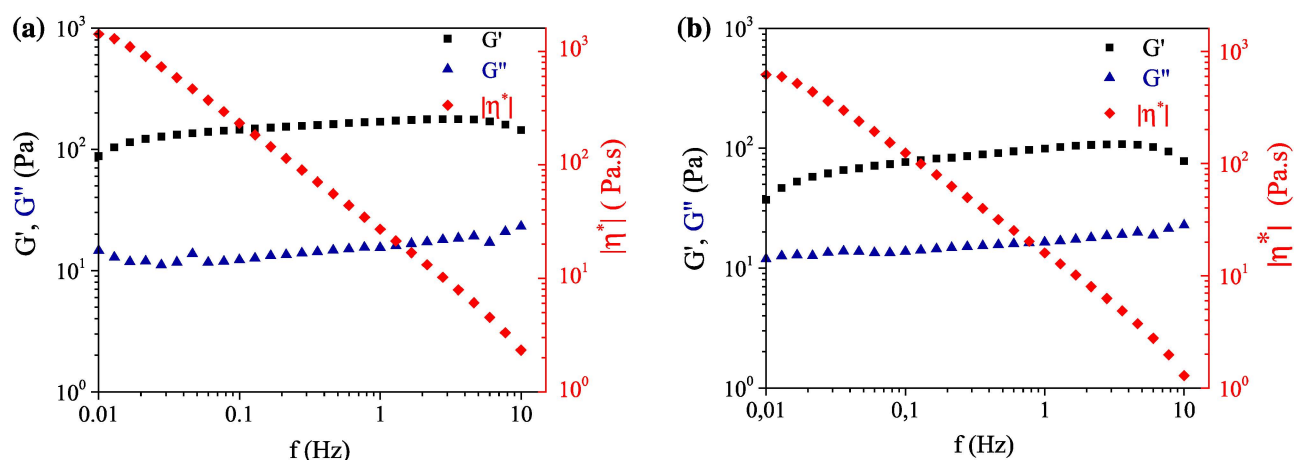


Fig. 7 Frequency sweeps of simple WBMs: **a** Fluid 4DCA-CNF and **b** Fluid 4XGD

Table 3 Parameters of the Sisko model for analyzed drilling fluids

System	Fluid	η_{∞} (mPa s)	K (mPa.s)	n	RSMD	R^2
S-DCA-CNF	1DCA-CNF	7.22	4147.41	0.16	0.37	0.99
	2DCA-CNF	8.53	10,567.81	0.13	1.48	0.99
	3DCA-CNF	11.52	24,868.45	0.07	0.11	0.99
	4DCA-CNF	19.73	34,775.43	0.08	0.06	0.99
	5DCA-CNF	21.26	51,894.99	0.07	0.04	0.99
	6DCA-CNF	8.27	8129.43	0.05	0.08	0.99
	7DCA-CNF	10.83	10,132.80	0.11	0.69	0.99
	8DCA-CNF	16.10	10,184.22	0.16	0.36	0.99
	9DCA-CNF	8.01	7939.26	0.09	0.15	0.99
	10DCA-CNF	10.14	14,499.38	0.05	0.50	0.99
	11DCA-CNF	15.79	15,471.42	0.12	0.65	0.99
S-XGD	1XGD	15.17	3974.62	0.20	0.24	0.99
	2XGD	23.45	8461.66	0.18	0.28	0.99
	3XGD	33.67	19,636.99	0.10	0.45	0.99
	4XGD	46.72	44,667.65	0.05	0.54	0.99
	5XGD	51.09	47,082.81	0.04	0.45	0.98
	6XGD	23.18	11,390.52	0.02	0.24	0.99
	7XGD	24.66	15,248.65	0.03	0.96	0.99
	8XGD	30.74	23,103.56	0.06	0.40	0.99
	9XGD	18.65	22,033.76	0.05	0.79	0.99
	10XGD	23.82	24,402.48	0.04	0.45	0.99
	11XGD	25.47	27,548.85	0.06	1.03	0.94

are nearly independent on the frequency (0.01–10 Hz), suggesting the development of stable structures. This behavior can be associated with the existence of a system with a high degree of structuring caused by physical cross-linking between polymer chains, and the interactions of BT and PAC with DCA-CNF or XGD (Ferry 1980). The complex viscosity (η^*)

decreased with the increase in the frequency indicates a shear thinning behavior. The viscosities under oscillatory-shear tests are similar to those from steady-shear tests.

Furthermore, the formed gels can be considered as “true gels” due to: (1) G' and G'' are independent of oscillation frequency; (2) G' is higher than G'' by an

order of magnitude; and (3) G' and G'' are constant in the range of frequency evaluated (Rao 2013; Schorsch et al. 1997). It is worth noting the WBM exhibits a gel behavior since the beginning of the frequency sweep.

Viscoelastic properties are important to gel formation and gel structure of drilling fluids. The formation of stable structures within the fluid contribute to the maintenance of the suspended particles, to enhance cutting carrying, and to prevent fluid invasion into the formation and lost circulation problem (Bui et al. 2012).

Filtration properties

The performance of filtration for simple WBMs (described in Table 1) was also evaluated by analyzing the properties of filter cake. The filtrate volumes of filter cakes are similar from both systems (Fig. 8a–f). Permeability, thickness, and filtration rate are presented in Table 4. In general, increases of BT, DCA-CNF, XGD, and PAC concentration increased thickness and decreased filtration rate and permeability. Additionally, when analyzing permeability, it is observed that for the same composition, S-DCA-

CNF exhibits slightly lower permeability values than the S-XGD system. It can be noted that fluids without BT or with low BT concentration did not present a filter cake (1DCA-CNF, 2DCA-CNF, 1XGD, 2XGD, and 3XGD).

Overall, the filtration properties of WBMs are mainly controlled by the build of a competent filter cake, which bridges porosity at the surface of a formation, as well as the viscosity of fluids (Hall et al. 2018). The differences in filtration results between S-DCA-CNF and S-XGD systems can be associated with the variations of the particle size of the polymers and the viscosity of WBMs, as well as possible interactions of the additives.

In comparison with the results of nanocelluloses previously studied (Villada et al. 2018), the S-DCA-CNF system exhibited improved filtration properties. The WBMs containing DCA-CNF presented lower filtrate volume than those with other nanocelluloses. This can be attributed to the higher viscosity of the fluids containing DCA-CNF, the “core-shell” structure formed by DCA-CNF that encapsulated and bound large amounts of water, and the property of DCA-CNF as a sealing agent, attributed to its smaller

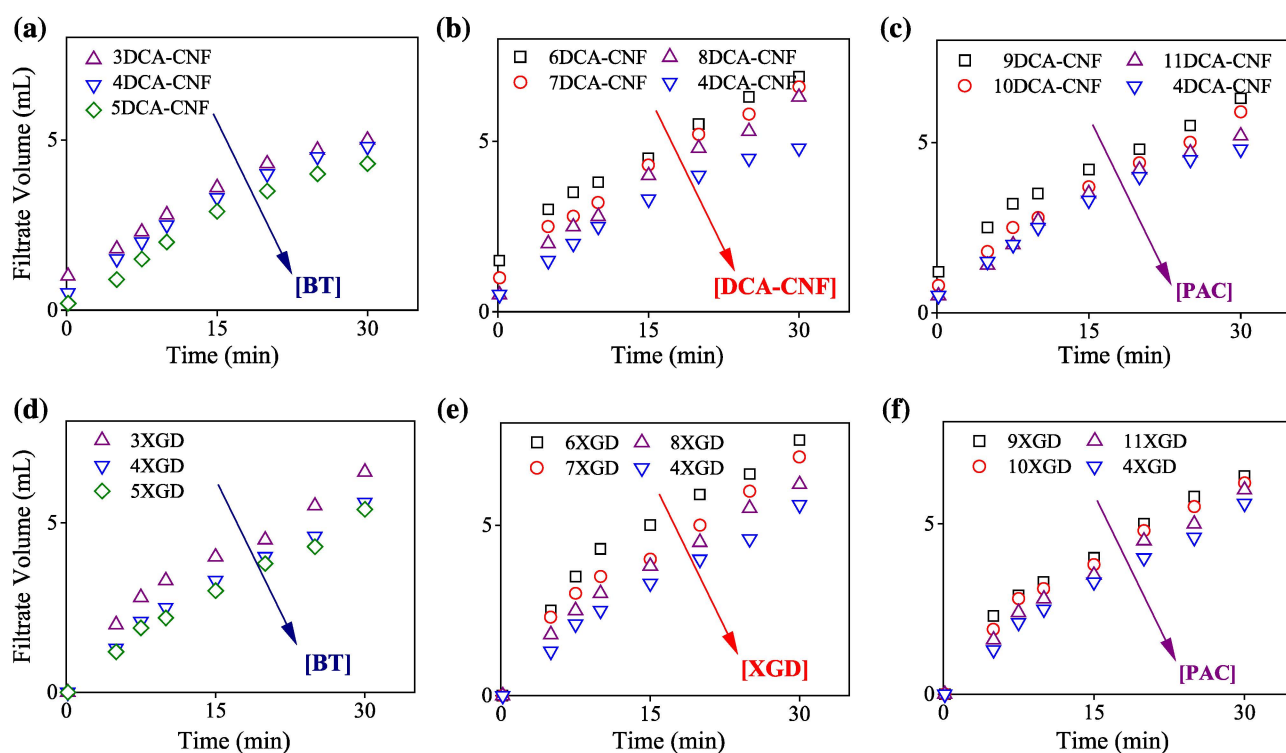


Fig. 8 Filtrate volume versus time curves for S-DCA-CNF (a, b, c) and S-XGD (d, e, f) systems. Effect of the additive concentration of: BT (a, d); DCA-CNF or XGD (b, e); PAC (c, f)

Table 4 Properties of the filter cakes

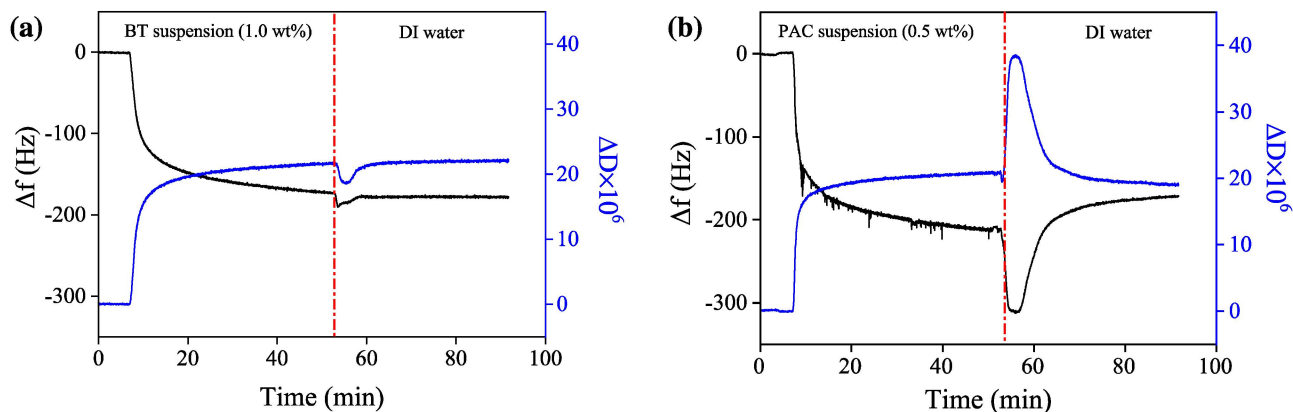
System	Fluid	T_c (cm)	$q \times 10^{-3}$ (cm ³ /s)	$K_c \times 10^{-3}$ (mD)
S-DCA-CNF	3DCA-CNF	0.10	2.33	0.77
	4DCA-CNF	0.11	2.33	0.85
	5DCA-CNF	0.12	2.33	0.93
	6DCA-CNF	0.08	2.88	0.76
	7DCA-CNF	0.09	3.02	0.90
	8DCA-CNF	0.10	3.02	1.01
	9DCA-CNF	0.10	2.67	0.88
	10DCA-CNF	0.11	2.67	0.97
	11DCA-CNF	0.12	2.67	1.07
S-XGD	3XGD	0.09	3.22	0.96
	4XGD	0.11	2.92	1.07
	5XGD	0.12	2.83	1.13
	6XGD	0.09	3.73	1.05
	7XGD	0.11	3.48	1.21
	8XGD	0.12	3.22	1.23
	9XGD	0.09	3.25	0.91
	10XGD	0.09	3.18	0.95
	11XGD	0.11	3.09	1.08

particle size (observed in micrographs obtained by SEM and AFM). These observations are in agreement with Li et al. (2018), who studied CNM with similar particle size to that of DCA-CNF.

Surface interactions

Figure 9 presents the results of the interactions of BT and PAC with DCA-CNF using the QCM-D technique. In this study, dissipation effects were observed due to the formation of a soft layer onto the sensor. As a result, surfaces were modeled using the Broadfit

model from the QSense DFind software. Figure 9a) shows the extent of BT adsorption on the DCA-CNF coated quartz sensors. Initially, deionized water was injected into the flow chamber of the QCM-D until a stable signal was attained. This step promotes swelling of the model surfaces and confirms their stability in water. Afterward, BT suspension (1.0 wt.%) was immediately injected, and the adsorption process starts, which can be observed as a drop in the frequency of the system ($\Delta f \sim -170$ Hz). The decrease in frequency suggests the adsorption of BT molecules on the surface of the DCA-CNF. After

**Fig. 9** Results obtained by QCM-D. Adsorption isotherm on DCA-CNF of **a** BT and **b** PAC

50 min, the flow of BT suspension was stopped, and the system was injected with water as a rinsing step.

Additionally, the increase in dissipation enables the assessment of the viscoelasticity of the formed layer. The slight changes observed in frequency and dissipation at $\Delta f \sim -189$ Hz and $\Delta D \sim 18.5 \times 10^{-6}$ respectively, could be linked with the water molecules absorbed and desorbed from the DCA-CNF surface. At the end of the rinsing step, the frequency did not return to zero, indicating that the BT mass absorbed on the DCA-CNF surface was irreversible.

The adsorption of PAC on the DCA-CNF film was analyzed following the same procedure as for BT suspension (Fig. 9b). Once the system was stabilized with deionized water, PAC suspension at 0.5 wt.% was flowed through the chamber, observing a change in frequency ($\Delta f \sim -180$ Hz). This behavior is associated with the adsorption of PAC particles on the surface of the DCA-CNF. After the changes in frequency and dissipation were constant, a rinsing step was applied using DI water. A pronounced shift was observed in frequency and dissipation at $\Delta f \sim -311$ Hz and $\Delta D \sim 38.3 \times 10^{-6}$, respectively. These sharp changes could be associated, as in the previous case, with adsorption and desorption of water molecules on the DCA-CNF surface and reorganization of the molecules along the surface. Again, at the end of the rinsing step, the frequency did not return to zero, indicating that the PAC mass adsorbs irreversibly on the DCA-CNF surface. Although PAC is considered as an anionic additive, it was adsorbed on the DCA-CNF surface. This observation could indicate the presence of hydrogen bonding and hydrophobic interactions (Hall et al. 2018).

In general, it can be concluded that the PAC exhibits a higher interaction with DCA-CNF than BT. The greater adsorption of PAC on the DCA-CNF surface was related to the similarity in cellulosic structures, as PAC is a cellulose derivative, specifically a functionalized cellulose. On the other hand, by correlating the dissipation and frequency changes presented in Fig. 9, $\Delta D/\Delta f > 5\%$, indicates that all the formed surfaces exhibit a viscoelastic behavior (Reviakine et al. 2011).

Figure 10 presents the AFM phase images of the DCA-CNF substrates after the QCM-D tests for the adsorption of BT and PAC on DCA-CNF. Different adsorption features were observed depending on the additive used. Figure 10a) suggests that BT particles

are deposited as a thin layer, whereas in Fig. 10b) clusters of PAC particles can be observed. This observation can be related to the association mode of additives. BT exhibits both negative and positive charges that promote the association with the DCA-CNFs through electrostatic interactions. Additionally, although PAC and DCA-CNF are both anionic, it was probed the irreversible interactions between them. This behavior could suggest the formation of hydrogen bonds, and hydrophobic interactions between the components, which agrees with the data reported in the literature (Hall et al. 2018). Additionally, the filter cakes containing PAC were shown to create oriented multilayer structures, compared with the filter cakes containing only BT (Li et al. 2015b). Thus, due to hydrophobic interactions, the crystallization of the material could occur on the surface of the DCA-CNF substrate, giving rise to the formation of clusters (Fig. 10).

Complex WBMs

Taking into account that the drilling fluids are a complex colloidal suspension of different additives with specific functions, DCA-CNF was studied as replacement of the XGD in complex WBM for a shale formation of Argentina (Vaca Muerta). Three fluids modifying DCA-CNF concentration were designed, following the previously described methodology and the composition presented in Table 2. It is important to note that Fluid A corresponds to the base fluid containing DCA-CNF instead of XGD.

The results of the rheological properties of complex WBMs are shown in Fig. 11. It is observed that fluid B exhibits a rheological behavior similar to the base fluid, and for this reason, it was selected for the evaluation of thermal stability and filtration properties.

Figure 12 presents the results of the aging assay for Fluid B. Fluid B exhibits less variation in viscosity after thermal treatment than WBM containing XGD (Base fluid). This is due to the high thermal stability of DCA-CNF, as discussed earlier. Likewise, as reported in the literature, the temperature corresponding to the peak degradation rate for a standard XGD viscosifier is 268 °C (Hall et al. 2017). Hence, WBMs with DCA-CNF exhibit higher thermal stability than the WBMs containing XGD, which improves the functions of WBMs at high temperatures.

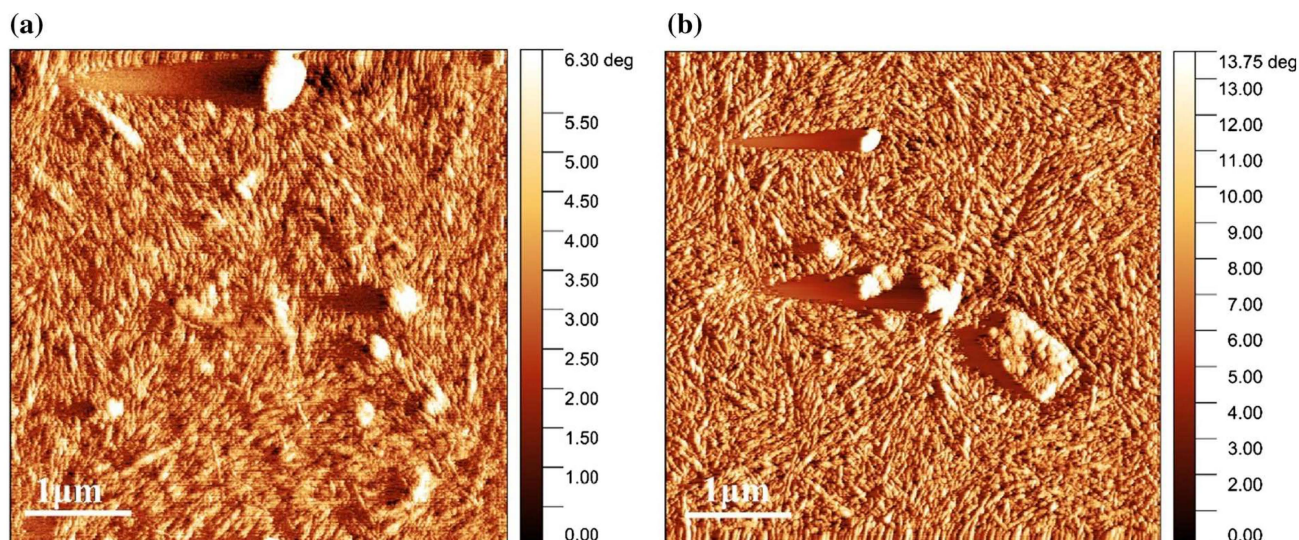


Fig. 10 AFM phase images of the coated sensors: **a** after the deposition of BT and **b** after the deposition of PAC

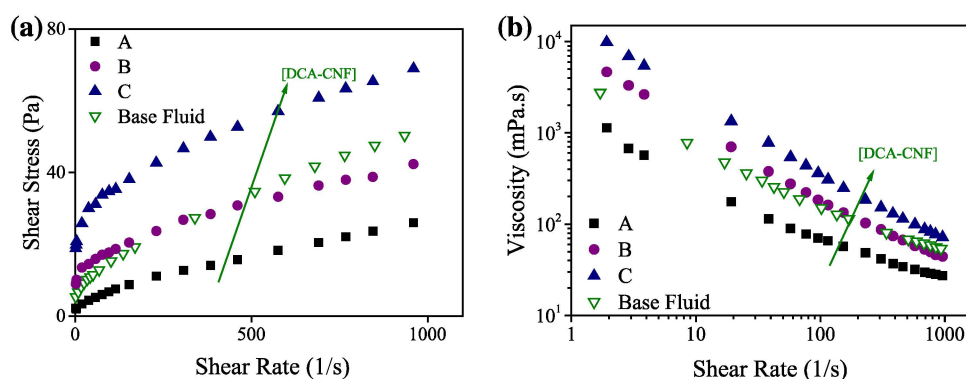


Fig. 11 Curves of a shear stress (**a**) and viscosity (**b**) as a function of shear rate for complex WBMs at various R-CNF concentrations. A: [DCA-CNFs] = 0.15 wt.%; B: [DCA-CNFs] = 0.30 wt.%; C: [DCA-CNFs] = 0.45 wt.%

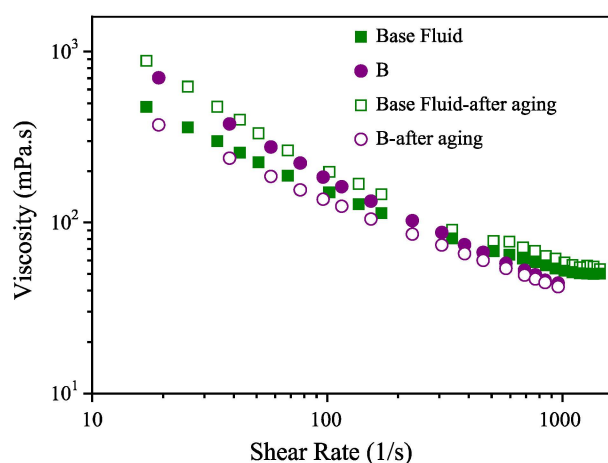


Fig. 12 Rheological results for complex WBMs containing XGD (base fluid) and DCA-CNF (Fluid B). Evaluation of thermal treatment

In Fig. 13 the filtration properties of the fluid B are presented. The filtrate volume at 30 min for the formation of filter cake is 16.5 mL (see Fig. 13a). Furthermore, the filtrate volume for the filter cake formed is 10.5 mL (Fig. 13b). In comparison with the results of filtrate volume for the base fluid reported by Villada et al. 2017, fluid B produced a lower filtrate volume. These results are likely related to its slightly higher viscosity than base fluid viscosity (Fig. 11b) and the nanoscale dimension of DCA-CNF that allowed the buildup of a more compact filter cake (Fig. 13c). Additionally, the surface chemistry of DCA-CNF improved the formation of a gel network structure with strong hydrogen bonding that restricted the movement of water (Hall et al. 2017).

Figure 14 shows the micrographs obtained by SEM for the complex WBM (Fluid B) before and after

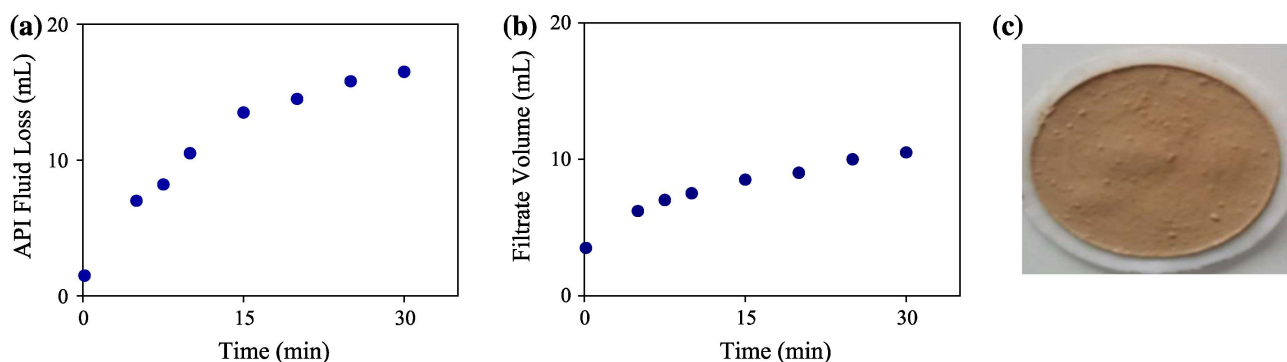
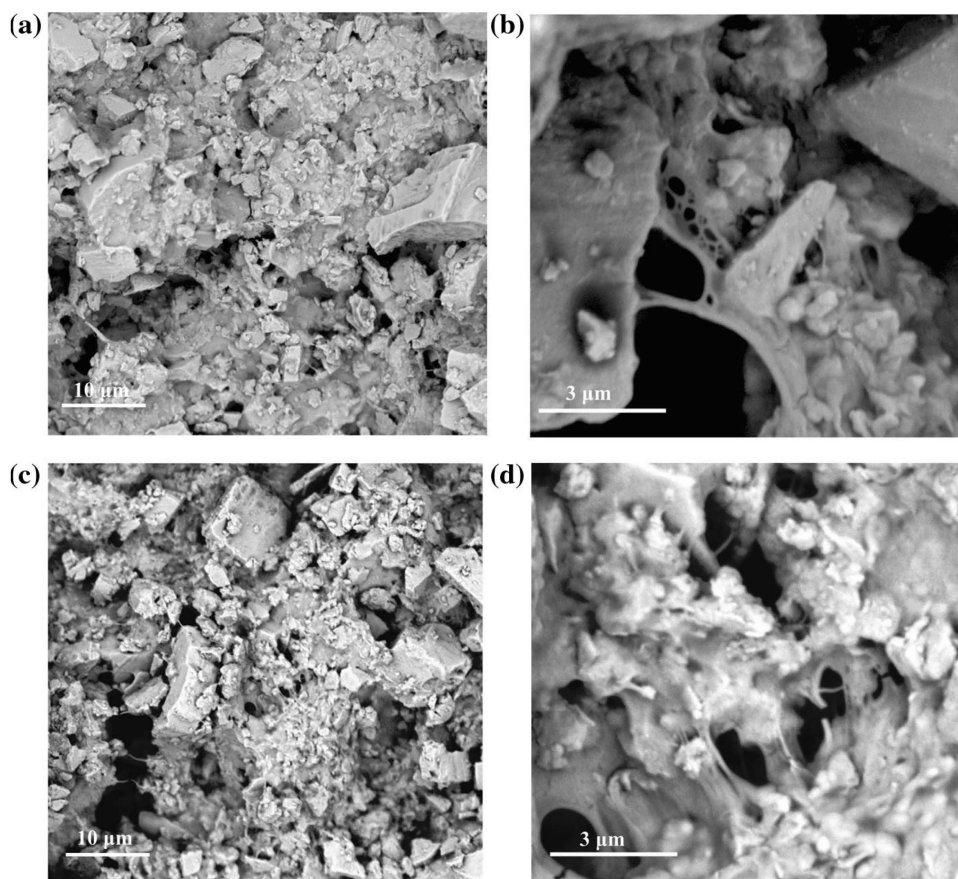


Fig. 13 Filtration properties of complex WBM for Argentina shale (Fluid B)

Fig. 14 Micrographs obtained by SEM. Effect of temperature in the microstructure of Fluid B. **a**, **b** Fluid before thermal aging, and **b**, **c** fluid after thermal aging at $T = 91\text{ }^{\circ}\text{C}$



thermal aging test. Fluid B shows a similar structure before and after thermal aging, which indicates good thermal stability. This result is in accordance with the lower variability of the viscosity evaluated previously. Also, the formation of the films and the agglomeration of the particles are observed. The formation of films benefits the filtration properties, due to the reduction of fluid loss by the buildup of compact filter cake, which bridges porosity at the surface of the formation (Hall et al. 2018).

Conclusions

The specific characteristics of DCA-CNF (such as charge density, particle size, and functionalization) fulfill an essential function in the properties of the fluids through their interactions with water, as well as with polymers, minerals, or the nanofibers themselves.

Simple WBMs containing DCA-CNF show very similar rheological and filtration properties to those of S-XGD system.

Complex fluids studies for Argentina shale showed optimal rheological properties, very similar to the base fluid, which was achieved using a DCA-CNF concentration of 0.30 wt.%. Furthermore, DCA-CNFs containing fluid had better thermal and filtration properties than the base fluid, resulting from the high stability of DCA-CNF, which is beneficial for practical applications.

The viscoelastic properties in the linear viscoelastic range were investigated using oscillatory tests. The frequency sweeps for the simple WBM showed an elastic module (G') higher than the viscous module (G''). This behavior is characteristic of strong gel-type structures. In addition, the formation of stable gels contributes to the colloidal stability of the WBM that improve the suspension and carrying of cuttings generated from the formation.

DCA-CNF produced from recyclable di-carboxylic acid hydrolysis with high thermal stability promises a renewable, non-toxic, and potentially less expensive alternative to partial or total replacement of synthetic polymers or conventional biopolymers in WBM. The investigation supports that DCA-CNF can be used as an additive in WBM for rheology, filtration control, and thermal properties. It is expected that DCA-CNF can be applied in other shale formations and hydraulic fracturing fluids. In a future work, economic studies will be considered to evaluate the application of DCA-CNF in WBM for Argentina shale.

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Compliance with ethical standards

Conflict of interest The authors declare no conflict of interest.

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