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Extending the Temperature Window for Formate Drilling and Completion Fluids with Cellulose Nano Fibrils

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Abstract

This paper presents the results of extensive testing of cellulose nano fibrils (CNF) in a cesium/potassium formate brine. It was found that formate brine, unlike water and calcium bromide brine, which were also tested, can increase the temperature stability of CNF to at least 190°C (374°F). Compared with xanthan gum, which is also stabilized by formate brine, CNF was found to be much easier to hydrate, and exhibited a more pseudoplastic, or shear-thinning, rheology profile. In addition to extending the actual temperature window for viscosified formate brines, CNF also significantly extended the time span that the fluid can resist high temperature. A formate fluid viscosified with CNF maintained more than 50 % of its rheology after 30-days exposure to 160°C (320°F).

In a drilling fluid formulation CNF offers the best fluid properties when it is used in combination with xanthan gum. The synergy between these two viscosifiers delivers a fluid that remains homogeneous under extensive static exposure to high temperature.

CNF was found to be a powerful viscosifier for cesium/potassium formate brine both when used in the clear brine itself and when used in solids-laden drilling fluid formulations.

Introduction

High-density formate brines made their entry as HP/HT drilling and completion fluids in the 1990s after the discovery of their temperature stabilizing effect on xanthan gum and other biopolymers (Downs 1991, Howard 1995). The introduction of environmentally friendly formate brines enabled a shift away from traditional oil-based fluids without losing any of their beneficial properties. Important benefits were gained, including reduced ECD, zero sag, excellent reservoir compatibility (Saasen et al. 2002, Berg et al. 2007, Jøntvedt et al. 2018), and improved compatibility with completion equipment, including sand-screens (Jøntvedt et al. 2018). In some cases, even shale stability was improved (van Oort et al. 2017).

The remarkable temperature stabilizing effect of formate brines on xanthan gum has made it possible to formulate high-density fluids with pseudoplastic, or shear-thinning, rheology that are stable to temperatures higher than what can be reached with any other brine systems (Howard et al. 2015). An example of this was the 204°C (400°F) Tuscaloosa well cleanout with a xanthan-viscosified potassium formate fluid (Messler et al. 2004). By adding some stabilizing additives, Messler et al. were able to stabilize xanthan gum for the hours it took to successfully conduct this milling job.

Despite the good temperature stability of xanthan gum in formate brine, there have been applications where more temperature-stable viscosifiers have been desired. Therefore, many attempts have been made to identify synthetic viscosifiers that are compatible with formate brines. Some polymers have been identified that yield viscosity in formate brine, but unfortunately none of these polymers exhibit the desired pseudoplastic, or shear-thinning, rheology (Anderson 2013). It has also been found that to fully hydrate synthetic polymers in formate brines, an enormous amount of heat is required over an extensive period. This increases the risk that unhydrated polymers will enter the formation and cause formation damage.

One product that showed some promise in the past was cellulose nano fibrils (CNF), a bio-based nanocellulose, which was used by Equinor and Sinomine Specialty Fluids to tackle the 175°C (347°F) temperature challenge in the Kristin field. CNF was successfully used in combination with xanthan gum in cesium/potassium formate drilling fluid formulations for the Kristin and Kvitebjørn field developments (Berg et al. 2007). Despite its successful field use, commercial production of CNF stopped for a while, but it is now available again.

This paper introduces various types of bio-based nanocellulose and presents results of an extensive amount of formulation and qualification work that has been conducted on formate brines and drilling fluids viscosified with CNF.

CNF and its Thermal Stability in Oilfield Brines

Nanocelluloses are sustainable, wood-derived fibers with interesting rheological properties and high chemical and thermal robustness. They are mainly classified into two groups – crystalline nanocellulose (CNC) and cellulose nano fibrils (CNFs). CNCs appear as needle-like crystalline rods, whereas CNFs are long, flexible threads, shown in Figure 1. CNCs are usually prepared through hydrolysis and sonication, and CNFs are typically produced through shear force intensive mechanical processes. The focus of this paper is on CNFs.

Mechanical preparation of CNFs was first described by Turbak et al. in (1983) and received renewed attention the last 15 years as mechanical, chemical, and enzymatic pre-treatments and production methods with lower energy consumption surfaced (Dufresne 2012). Despite there being nearly 40 years since their discovery, no consensus is agreed on for standardized nomenclature when referring to CNFs. Therefore, one can also find CNF referred to in literature as “micro fibrillated cellulose” (MFC) and “nano fibrillated cellulose” (NFC).

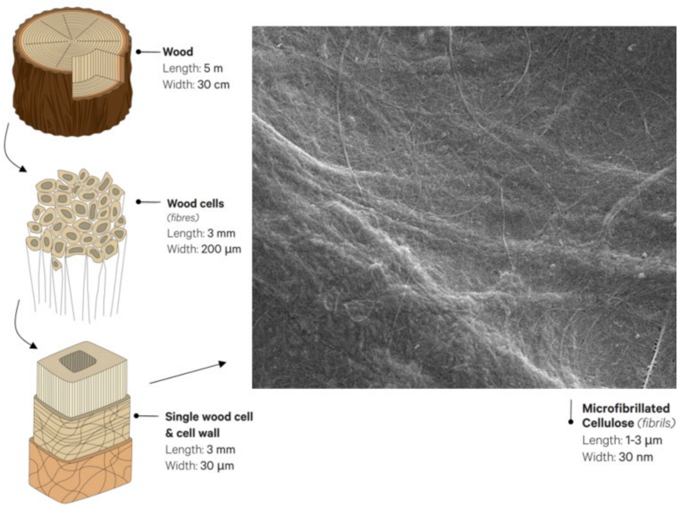

Figure 1 – Wood fibers are made up of small cells, where the cell walls are reinforced and given stability by nanocellulose. SEM image shows a CNF film, where large structures are fiber residue, and smaller “threads” are nano-sized fibrils.

Recently a CNF product was tested for use as viscosifier in halide brines (Deville et al. 2020). Deville found that in a low-density sodium chloride brine, it took a loading of three times as much CNF as xanthan gum to obtain the same rheology, and CNF exhibited a less shear-thinning, rheology profile than xanthan gum. The low-shear rheology was almost non-existent. In divalent halide brines (calcium chloride and calcium bromide), where xanthan gum is known to be ineffective, the CNF made a slight improvement over xanthan gum, but the rheology profiles were even poorer than in sodium chloride brine. Deville et al. were able to increase the rheology of their fluid formulations by introducing solids. With a solids-loading of 114 kg/m³ (40 lb/bbl) sized calcium carbonate, a pseudoplastic fluid could be formulated that survived 16-hours hot rolling at 150°C (302°F). However, the fluid loss control provided by this fluid was poor. In the solids-loaded calcium chloride and calcium bromide brines, good rheology and fluid loss control was achieved after hot rolling at 150°C (302°F). No test results were reported at temperatures above 150°C (302°F).

As to the thermal stability of CNF compared with other biopolymeric viscosifiers, a limited amount of test results has been reported by Heggeset et al. (2017), who compared the thermal stability of CNF, xanthan and guar gum dispersions in the temperature range from 110°C to 150°C (230 to 302°F) and found much higher temperature stability for CNF. Mechanically pre-treated CNF (Mech-CNF), chemically pre-treated TEMPO-oxidized CNF and carboxylated-CNF, as well as CNC were examined for their thermal stability in dispersions, and it was found that Mech-CNF and CNC exhibited the best thermal stability.

Heggeset et al. also studied the degradation mechanisms of these four nanocelluloses by use of additives that either stabilize pH or scavenge free radicals. The results indicate that ORD (oxidative-reductive depolymerization) is the main degradation mechanism as addition of the formate ion, which scavenges free radicals, significantly reduced thermal degradation. Without presence of the formate ion, the nanocellulose dispersions started to degrade already at 110°C (230°F) – a much lower temperature than they degrade at in their dry state The addition of a relatively low concentration of formate (0.066 and 0.37 M) increased the temperature stability to 140°C (284°F) after three days heat ageing. Unfortunately, this research has little relevance for formate oilfield brines, which are used in concentrations of approximately 10 M. Also, all testing was conducted with 0.8 wt% solids added, so the results are not valid for solids-free formate brines.

Qualification Testing of CNF in Formate Brines

A range of unmodified CNF products, prepared from once-dried kraft pulp according to the method described by Turbak et al. (1982), have been tested for compatibility with a formate brine. When nothing else is specified, the formate brine that was used for testing was a 1.89 g/cm³ (15.8 lb/gal) cesium/potassium formate brine, composed of equal parts of a 2.20 g/cm³ (18.4 lb/gal) cesium formate brine and a 1.57 g/cm³ (13.1 lb/gal) potassium formate brine. The brine was buffered to a pH of 10.3 with 9.1 kg/m³ (3.2 lb/bbl) potassium carbonate/bicarbonate. This brine composition is representative for what is typically used in cesium/potassium formate drilling fluids. The CNF products that were tested were hydrated in various amounts of water and contained 10–20 wt% CNF. All concentrations referred to here, apply to the equivalent amount of dry product. The xanthan gum that was used in some of the formulations is a standard oilfield grade product.

Hydration of CNF in Formate Brines

Cesium and potassium formate brines and their blends, when used in drilling and completion fluids, have salt concentrations of 75–80 wt% and their water activity is very low, <0.3. Hydrating polymers in formate brines is therefore very challenging, and different mixing regimes are required for different polymers. For xanthan gum to be fully hydrated in formate brines, temperatures greater than 90°C (194°F) are required. In the laboratory xanthan gum is sheared into the brine in a Silverson high-shear mixer for at least 40 minutes before the fluid is conditioned at a temperature above 90°C (194°F) for some hours. Synthetic polymers require even higher temperatures and more time to hydrate in formate brines. It was therefore surprising to learn that high shear was not required to hydrate CNF in formate brine. Mixing at ambient temperature with a standard paddle mixer for 90 minutes was enough to reach full viscosity.

Rheological Properties

An example of the rheology profile of a CNF product in buffered cesium/potassium formate brine is shown in Table 1 and Figure 2 along with the rheology profile of xanthan gum in the identical brine. As can be seen the CNF product exhibits a rheology profile that is more pseudoplastic than the rheology profile of the xanthan gum. This is in strong contrast to what was reported by Deville et al. (2020) for sodium chloride brine where CNF did not exhibit pseudoplastic rheology compared with xanthan gum.

To match the rheology of 2.9 kg/m³ (1.0 lb/bbl) xanthan, the equivalent of 3.0 kg/m³ (1.1 lb/bbl) dry CNF had to be added. This is also in contrast to the findings by Deville et al. for sodium chloride brine where a CNF concentration three times that of xanthan gum was needed to reach a similar viscosity level.

Table 1 – Rheological properties of 3.0 kg/m³ (1.1 lb/bbl) CNF and 2.9 kg/m³ (1.0 lb/bbl) xanthan gum in a buffered 1.89 g/cm³ (15.8 lb/gal) Cs/K formate brine. Rheology was measured at 49°C (120°F).

Property	CNF	Xanthan gum
600 rpm	28.1	29.7
300 rpm	18.7	18.4
200 rpm	14.8	14.1
100 rpm	10.3	8.7
60 rpm	8.4	6.5
30 rpm	6.5	4.3
20 rpm	5.3	3.7
10 rpm	4.3	3.0
6 rpm	3.4	2.7
3 rpm	3.0	2.1
PV	8.9	10.7
YP	10.1	7.5
10-sec gel	4.3	2.1
10-min gel	4.5	4.7

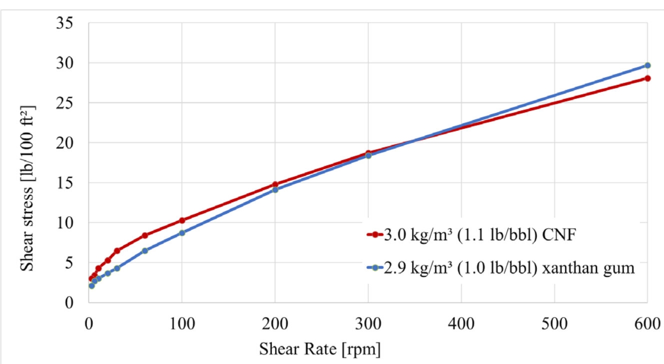

Figure 2 – Rheological profiles of CNF and xanthan gum in a buffered 1.89 g/cm³ (15.8 lb/gal) Cs/K formate brine. Rheology was measured at 49°C (120°F).

Effect of Brine Type

The extraordinary compatibility between the CNF products and formate brines was demonstrated by comparing their rheology profiles in cesium/potassium formate brine with their rheology profiles in water and calcium bromide brine. Figure 3 shows the rheology profile of the equivalent of 3.0 kg/m³ (1.1 lb/bbl) dry CNF in cesium/potassium formate brine, calcium bromide brine, and water. The rheology was measured before and after hot rolling at 150 and 160°C (302 and 320°F). This testing confirms that the tested CNFs are highly compatible with formate brines but do not yield at all in water and only slightly in calcium bromide brine. The small amount of viscosity that was present in the calcium bromide brine disappeared completely during hot rolling.

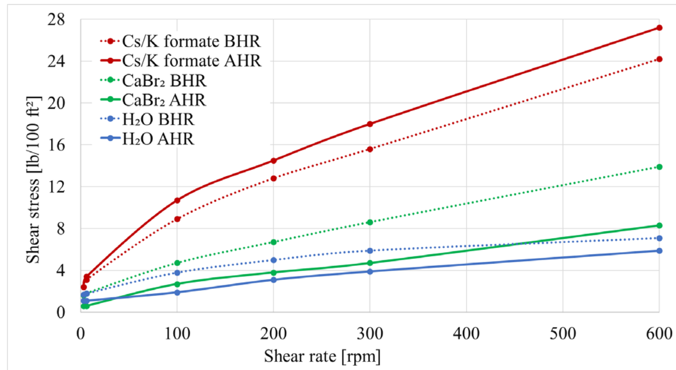

Figure 3 – Rheology of 3.0 kg/m³ (1.1 lb/bbl) CNF in a buffered 1.89 g/cm³ (15.8 lb/gal) Cs/K formate brine, a 1.68 g/cm³ (14.0 lb/gal) $CaBr_2$ brine, and water before and after hot rolling at 150°C (302°F). Rheology was measured at 49°C (120°F).

Extending the Temperature Limit

The exact upper temperature limit of the CNF products in formate brines has not been determined, and it is also uncertain to what extent this depends on the formate brine composition and concentration. However, testing for 16 hours at 190° (374°F) in 1.89 g/cm³ (15.8 lb/gal) buffered cesium/potassium formate brine has shown that the CNF can provide good pseudoplastic rheology for 16 hours to at least this temperature. Figure 4 shows the rheology of the equivalent of 6.3 kg/m³ (2.2 lb/bbl) dry CNF in the buffered cesium/potassium formate brine after hot rolling for 16 hours at temperatures up to 190°C (374°F). From the color of the hot rolled fluids, one can confirm that some thermal degradation takes place at the highest temperatures. However, even after the 190°C (374°F) exposure, the almost black fluid maintained its favorable pseudoplastic rheology, and less than 50% of its viscosity had been lost. Howard et a (2015) showed that xanthan gum in a cesium/potassium formate brine with the same composition is expected to lose 50% of its viscosity at approximately 173°C (343°F). This confirms that compared with xanthan gum, CNF extends the temperature stability of viscosified cesium/potassium formate brines by at least 20°C (36°F). When filtering the darkened fluids, the filtrate was clear and uncolored, indicating that no degradation product is likely to enter the formation.

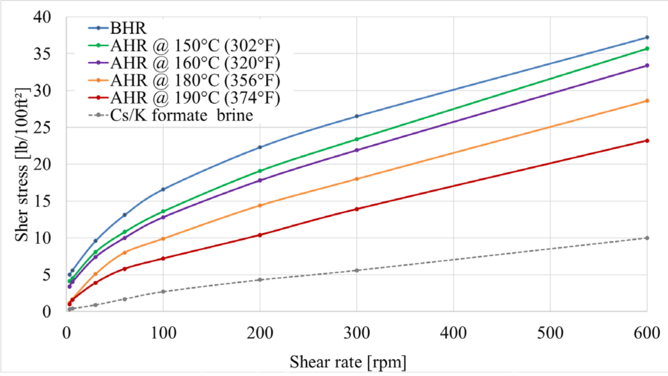

Figure 4 – High temperature thermal stability of CNF. Rheology of 6.3 kg/m³ (2.2 lb/bbl) CNF in a 1.89 g/cm³ (15.8 lb/gal) Cs/K formate brine after hot rolling for 16 hours at 150, 160, 170, 180, and 190°C (302, 320, 338, 356, and 374°F). Rheology was measured at 49°C (120°F).

Extending Exposure Time

CNF has also shown remarkably good stability in formate brines over periods of several weeks. Figure 5 shows its stability in a 1.89 g/cm³ (15.8 lb/gal) buffered cesium/potassium formate brine after hot rolling up to 30 days at 160°C (320°F). The rheology drops a little during the first five days but remains relatively stable during the remaining period. Its thermal stability over several months has still not been studied.

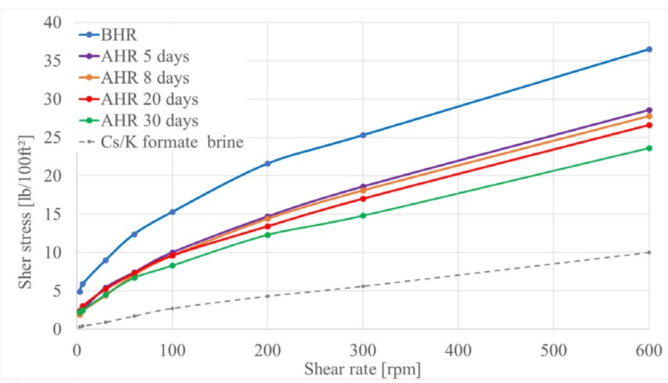

Figure 5 – Long-term stability of CNF. Rheology of 6.3 kg/m³ (2.2 lb/bbl) CNF in a buffered 1.89 g/cm³ (15.8 lb/gal) Cs/K formate brine after hot rolling at 160°C (320°F) for up to 30 days. Rheology was measured at 49°C (120°F).

CNF in Drilling Fluid Formulations

When used in formulations with other drilling fluid additives, CNF has shown to perform best when used in combination with xanthan gum. This is because in formulations with only CNF, a thin layer of brine forms at the bottom of the fluid after the fluid is left static at high temperature. In formulations with xanthan gum, a similar thin layer of brine appears on the top of the fluid column. See photos in Figure 6. The presence of brine on the top or at the bottom can affect performance of HP/HT fluid loss tests and PPA tests, depending on whether the equipment is configured with the filter medium on the top or at the bottom. This effect of the configuration can be seen in Figure 7, where the same fluid formulation, containing 6.3 kg/m³ (2.2 lb/bbl) CNF, 15.7 kg/m³ (5.5 lb/bbl) HT starch, and 57 kg/m³ (20 lb/bbl) sized $CaCO_3$, was tested under the same conditions in an HP/HT filter press with the filter medium at the bottom and in a PPA with the filter medium on the top. The results show how the CNF viscosified fluid performs much better with the filter medium on top.

Figure 6 – Photos of a hot rolled formate drilling fluids viscosified with xanthan gum (left) CNF (middle) and a blend of xanthan gum and CNF (right). In the xanthan viscosified fluid a thin layer of brine appears on top of the fluid. In the CNF viscosified fluid, a similar layer of brine appears at the bottom of the fluid. No brine has separated from the fluid with both CNF and xanthan gum. No solids sag is observed in any fluid.

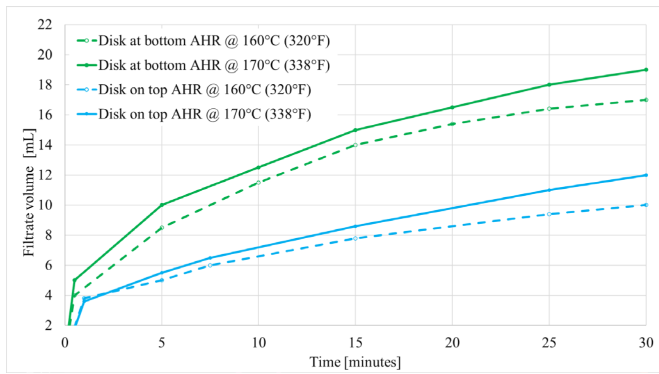

Figure 7 - HP/HT fluid loss test results from a HP/HT filter press with the disk at the bottom and a PPA configured with the disk on the top. The tests were performed with the same drilling fluid at 149°C (300°F) with 500 psi differential pressure on 10 µm aloxite disks.

Formulations with both xanthan gum and CNF have shown very good homogeneity when left over longer periods and provide good fluid loss control in configurations with the filter medium both on the top and at the bottom. Please note that presence of some brine on the top or at the bottom of the fluid column is not a case of sag. There is nothing to sag in a formate drilling fluid, and the 'free brine', whether it is present on the top or at the bottom has a density that is only marginally different from the formulated fluid. It is interesting to notice that in the CNF-viscosified fluid, no calcium carbonate particles are present in the brine layer at the bottom of the beaker glass despite it having a higher density than the brine. This did not change after the fluid was left at the benchtop for several weeks.

To further investigate the effect of the filter press configuration, three fluid formulations were tested for HP/HT fluid loss on 10-µm aloxite disks after hot rolling at 150, 160, and 170°C (302, 320, and 338°F). The formulations are shown in Table 2 and the fluid loss profiles after hot rolling at 150 and 160°C (320 and 338°F) are plotted in Figure 8. As can be seen, the formulation with both CNF and xanthan gum performed the best.

Table 2 – Formulations viscosified with only xanthan gum, only CNF, and a combination of both, which were used for comparing HP/HT fluid loss properties.

Additives	XC/starch		CNF/starch		XC/CNF/starch	
	kg/m³	lb/bbl	kg/m³	lb/bbl	kg/m³	lb/bbl
Xanthan gum	1.57	0.55			0.86	0.30
CNF			3.1	1.1	1.57	0.55
HT starch	15.7	5.5	15.7	5.5	15.7	5.5
Sized $CaCO_3$	57	20	57	20	57	20

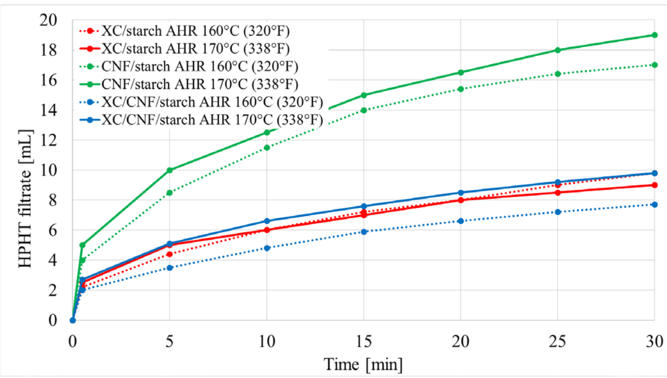

Figure 8 – HP/HT fluid loss for fluid formulations viscosified with xanthan only, CNF only, and mixed xanthan and CNF viscosifiers after 16-hours hot rolling up to 170°C (338°F). Fluid loss was measured at 149°C (300°F) with 500 psi differential pressure and on a 10 µm aloxite disk.

Compatibility with Completion and Reservoir

Formation and completion damage are concerns in HP/HT fluid systems due to the presence of high concentrations of solids weighting material and synthetic polymers. In the case of formate brines, even at very high densities, solids weighting material is not a problem as cesium formate can reach any desired density without adding solids. The question then is whether CNF will plug sand screens or formation. Some testing has been performed to investigate this further.

Most wells that have been drilled with formate fluids have been completed openhole with sand screens. After many cases of blocked screens when attempts have been made to run sand screens in oil-based muds, e.g., Equinor's Kristin field (Zaostrovski 2011), BP's Marnock field (Munro et al 2002), and Total's Martin Linge field (Jøntvedt et al. 2018), low solids formate fluids have for many operators become the preferred screen-running fluid. Formate drilling fluids, formulated with xanthan gum, some starch, and just a very low concentration of calcium carbonate bridging solids, have traditionally passed the production screen test (PST) directly without any need for a separate screen-running fluid to be deployed. It is therefore important that any viscosifier that either replaces xanthan gum or is added to the fluid together with xanthan gum doesn't promote completion damage by plugging sand screens. Production screen testing was therefore conducted on fluid formulations containing CNF.

Two drilling fluid formulations were compared, one viscosified with 1.57 kg/m³ (0.55 lb/bbl) xanthan gum and one with the equivalent of 3.14 kg/m³ (1.10 lb/bbl) CNF. The formulations that were tested are shown in Table 3 and the results are shown in Table 4. Testing was carried out with 1-liter samples that were passed through the screen three times at 30 psi. From the results one can see that both fluids passed through the 250-µm screen with similar performance.

Table 3 – Fluid formulations used for PST testing. In both formulations the additives were added to a standard 1.97 g/cm³ (16.4 lb/gal) cesium/potassium formate brine buffered to pH 10.3 with 6.86 kg/m³ (2.40 lb/bbl) potassium carbonate/bicarbonate buffer.

Additives	XC/starch		CNF/starch	
	[kg/m³]	[lb/bbl]	[kg/m³]	[lb/bbl]
Xanthan gum	1.57	0.55		
CNF			3.3	1.1
HT starch	15.7	5.5	15.7	5.5
Sized $CaCO_3$	57	20	57	20

Table 4 – Results of production screen testing on a 250 µm sand screen with 30 psi pressure. The results are expressed as the pass-through time for three runs of one liter fluid.

Run #	XC/starch [seconds]	CNF/starch [seconds]
1st	18.20	17.78
2nd	18.56	18.36
3rd	18.23	18.76

The main cause of permeability reduction in laboratory core flow tests with formate brines is fluid retention resulting from inadequate drawdown procedures. This laboratory artifact affects both tests with clear brine and tests with drilling fluids, which makes it difficult to assess to what extent drilling fluid additives affect the return permeability. Therefore, coreflood testing of drilling fluid with CNF in the formulation has not been performed.

It is known, however, that Deville et al. (2020) performed coreflood testing with two sodium chloride brines that had been added CNF and xanthan gum respectively. The fluid with CNF caused significantly less reduction in return permeability than the fluid with xanthan gum. Also, in a calcium bromide brine with CNF added, the measured return permeability was very high. One needs to be careful with these results as nothing is mentioned about whether the 'damaging' xanthan gum was properly hydrated and whether it had been given enough time to thermally degrade before the return permeability measurements were conducted. It is very encouraging, however, to see the good performance of CNF in these tests. One explanation for the good result with CNF could be that it tends to form a thin paper-like film on the filter medium rather than invading into it. Figure 9 shows a photo of a CNF filtercake that was formed in an API fluid loss test. It had the appearance of silk paper and peeled easily off the filter medium.

Figure 9 – Photo showing the CNF filter cake after an API filtration of a formate brine viscosified with CNF.

Conclusions

Based on an extensive test program that has been conducted on the use of CNF as a viscosifier for formate brines and drilling fluids, the following can be concluded:

- The CNF products that were tested are all fully compatible with formate brines.
- CNF hydrates much easier than xanthan gum in formate brines.
- CNF exhibits very pseudoplastic (shear-thinning) rheology when present formate brines — more shear-thinning than xanthan gum.
- Concentrated cesium/potassium formate brine protects CNF from thermal degradation. At 190°C (374°F), despite some degradation, still provides good rheology to the fluid.
- The CNF products that were tested do not work well with $CaBr_2$ brine and water. Both did not gain much viscosity and degradation was observed at 150°C (302°F). This agrees with the findings by Deville et al. (2020).
- In a drilling fluid formulation CNF works best when it is combined with xanthan gum. This combination provides a fluid that remains homogeneous under extensive static exposure to high temperature.
- PST testing has confirmed that CNF does not plug 250 µm sand screens.

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Nomenclature

AHR = *After hot rolling*
BHR = *Before hot rolling*
CNC = *cellulose nanocrystals*
CNF = *Cellulose nano fibrils*
HP/HT = *High-pressure/high-temperature*
MFC = *Micro fibrillated cellulose*
ORD = *oxidative-reductive depolymerization*
PST = *Production screen test*
PPA = *Permeability plugging apparatus*
XC = *Xanthan gum*

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