Comparison of Lost Circulation Material Sealing Effectiveness in Water-Based and Oil-Based Drilling Fluids and Under Conditions of Mechanical Shear and High Differential Pressures

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A study was conducted to assess the performance of granular and fibrous lost circulation materials as preventative treatments and in remedial treatment of lost circulation in water-based and oil-based drilling fluids. For the preventative treatments, a factor that introduced increased mechanical wear on the particles was added to the hot-rolling process, to identify signs of deterioration of performance of certain materials. The study of remedial treatments of lost circulation was conducted on slotted discs with apertures of 750 μm and up to 5 mm and with a differential pressure of up to 34.5 MPa (5000 psi). To compare the sealing pressures of the different tests, a simple statistical analysis was introduced to differentiate between the peak holding pressures and the sustainable holding pressures of the various material and fluids combinations. The material degradation studies showed that $CaCO_3$-based lost circulation materials rapidly experienced significant particle degradation after exposure to fluid shear and mechanical degradation and that this considerably reduced the sealing performance of the materials. Also, synthetic graphite-based products showed clear signs in particle size degradation and a significant reduction in sealing performance. Cellulose-based products showed superior resistance toward mechanical wear and only small changes in sealing performance. When comparing water-based and oil-based fluids, it was clear that granular lost circulation materials showed considerably lower sealing efficiency in oil-based drilling fluids compared to water-based drilling fluids. In contrast, cellulose-based materials showed similar sealing performance in oil-based fluids and water-based fluids. [DOI: 10.1115/1.4054653]

Keywords: lost circulation, lost circulation material sealing effectiveness, particle size degradation, sealing pressure, geothermal energy, petroleum engineering, petroleum wells-drilling/production/construction

1 Introduction

Lost circulation is a critical factor that may reduce drilling efficiency, increase cost, and increase the risk of well collapse. Oil-based drilling fluids are often considered as superior to water-based drilling fluids with regards to obtaining a low fluid loss and achieving high rates of penetration. Water-based drilling fluids are in contrast often preferred due to a lower cost if the risk of large or total losses of drilling fluid is expected.

A considerable number of studies have been conducted on the classification of lost circulation materials (LCMs) and the sealing abilities of different materials. Alsaba et al. [1] classified lost circulation materials into categories based on physical and chemical characteristics. Alshubbar et al. [2] found that higher circulation rates led to higher fluid loss and observed that lost circulation materials with lower density were less impacted by annular flow, and that such materials therefore may be more effective for preventative treatment. Alsaba et al. [3] compared lost circulation materials from different material categories and found that fibers gave the best seals on tapered slotted discs. Furthermore, they found that granular materials such as $CaCO_3$ and graphite created seals with lower integrity. Khalifeh et al. [4] also tested fiber-based lost circulation materials and found that these seals were dynamically built to withstand gradually higher pressures without failing.

The use of nanoparticles in drilling fluids has received significant attention recently. For example, Alvi et al. [5] have shown that it is possible to reduce filtration loss measured on filter paper by the addition of 0.5 wt% iron oxide nanoparticles to an oil-based drilling fluid. In a series of experiments, such filtration loss was nearly halved. Most attention with the nanoparticle studies has been directed toward conventional fluid loss tests against filter paper or porous formation like Contreras et al. [6]. They found also an optimum effect by the addition of 0.5 wt% graphite together with 0.5 wt% nanoparticles based on iron or calcium. The role of particle size distribution (PSD) for fluid loss materials without nanoparticles on the formation of filter cakes and avoiding formation damage can be found consulting Klungtvedt and Saasen [7].

The main cost related to lost circulation treatment is normally the nonproductive time incurred to treat the loss or to remedy other consequences of the lost circulation, such as differential sticking. Grelland [8] studied how lost circulation is treated by most companies in the North Sea area and found that only 1–2% of the costs of treating lost circulation was related to the lost circulation material cost. He also found that the main materials used for treating losses on the Norwegian continental shelf were $CaCO_3$ and

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graphite. These materials were used alone or in combination and most of these treatments were insufficient to cure lost circulation. Furthermore, he concluded that the LCM treatments did not differ between formations drilled even though there were different pore and fracture sizes in different formations. Application of LCM with higher density than the typical fluid density was used consistently. As such, his findings related to actual field application procedures appear to be in contrast with the conclusions of Alshubbar et al. [2] and Alsaba et al. [3], where Alshubbar et al. concluded that in a circulating well LCM with lower specific gravity were better preventative candidates and Alsaba et al. showed that LCMs that have irregularity in particles shapes and a degree of deformability are effective in improving sealing strength and reducing fluid loss.

The research on lost circulation materials and sealing effectiveness does not provide a standard for determining which sealing pressure should be recorded for a given test as it may be a pressure held over time of the maximum pressure obtained before the seal broke. The present study proposes a simple metric for measuring a peak hold pressure (PHP) and a sustainable hold pressure (SHP) to provide as a minimum method for classifying a sealing pressure using slot testing of lost circulation materials.

To test the proposed metric for measuring sealing pressures, a typical lost circulation treatment recipe for both preventative treatments and remedial treatments of lost circulation following the findings of Grelland [8] was used. These recipes were applied to both an oil-based drilling fluid and a water-based drilling fluid and compared with a recipe like that tested by Khalifeh et al. [4]. This would allow for comparing the different treatments of lost circulation and to compare performances in oil-based and water-based drilling fluids.

Scott et al. [9] presented a pragmatic approach to lost circulation treatment and concluded that bridging is achieved when the particles are equal to or slightly larger than the loss zone opening and present in the fluid at a concentration of 10–20 lb/bbl (28.5–57 kg/m^3). Furthermore, due to solids content in field mud, for the LCM to be effectively enhancing the performance of a field mud, LCM should have a D_{50} value of 400 μm or larger.

Hoxha et al. [10] used a flow loop and shearing facilities to test the degradation of the particle size distribution of lost circulation materials under the influence of shear. They found that both $CaCO_3$ and graphite suffer from shear degradation. To build on these findings and the conclusions of Alsaba et al. [3], a method was proposed and tested in the present study to introduce mechanical wear into an ordinary hot-rolling process for drilling fluids. The method was applied to lost circulation materials that are designed to be a part of the circulating system, and this will experience mechanical wear and potential degradation.

To summarize, a series of experiments were conducted where the objectives were to:

- apply a simple statistical method for measuring the sealing strength of lost circulation materials against a specific fracture size;
- identify if lost circulation treatment is equally effective in KCl/polymer water-based drilling fluids and oil-based drilling fluids;
- investigate sealing mechanisms and sealing strength of granular and fibrous lost circulation materials; and
- identify how a method for applying mechanical shear in the hot-rolling process impacts particle size distribution and sealing ability of lost circulation materials for preventative treatment.

2 Materials and Methods

The tests were conducted using a permeability plugging apparatus where drilling fluid can be tested on either ceramic discs or slotted steel discs with a 63 mm diameter. Pressure can be applied by either a pressured gas source or by a hydraulic pump which allows for logging the applied pressure digitally at 1 s intervals during the test. The tests were conducted at a temperature of 60 °C.

Fig. 1 Slotted steel discs for testing of LCM pills

The slotted steel discs each have multiple slots without any tapering. With limited side wall friction, the sealing would primarily need to take place at the fracture tip, thus likely making the test more difficult than to seal a subterranean fracture where the friction within the fracture may help to form a deep seal. As such, the tests will not fully replicate the deep sealing of a fracture, but they may be a practical approach to understand the potential for sealing the fracture opening. For the testing of high-concentration LCM pills, discs with slot widths of 0.75, 1.5, 2.0, and 3.5 mm were used. In addition, a disc with a combination of single slots of sizes 0.5, 1.0, 2.0, 3.0, and 5.0 mm was selected. The discs used for testing LCM pills are shown in Fig. 1.

The metric proposed to determine the sealing strength of the lost circulation materials was calculated as a moving average over time periods of 10 or 60 s and the highest average value during 10 and 60 s averaging periods was selected as the peak hold pressure and sustainable hold pressure, respectively

$$P(MA_n) = \sum_{t=0}^{t=n} P_t/n \tag{1}$$

$$\text{Peak hold pressure} = \max P(MA_{10}) \tag{2}$$

$$\text{Sustainable hold pressure} = \max P(MA_{60}) \tag{3}$$

The tests were conducted with the objective of obtaining the highest sealing pressure for each combination of the material and slotted disc. Limitations were set for the fluid loss of 275 mL out of an applied volume of 400 mL, to ensure that sufficient fluid was left in the test cylinder, pressures exceeding and holding above 34.9 MPa (5000 psi) or a period of 20 min.

An overview of the equipment used is presented in the Appendix.

2.1 Particle Size Distribution of Materials.

The materials were selected to replicate the materials references by Grelland [8] and Khalifeh et al. [4], with some additions. A description of each material is shown in Table 1. The granular products are ground marble, hereinafter referred to as $CaCO_3$, and resilient graphite, whereas the cellulose-based products have three different natures. One is an ultra-fine cellulose powder, another is a hard and granular cellulose, and the third is a mixture of various cellulose fibers and granular particles.

2.2 Mechanical Wear and Particle Degradation.

For materials used as part of the active system, the particles will experience wear as part of the circulation in the well. To simulate this, a

Table 1 LCM materials

Material	D_{50} (μm)	D_{90} (μm)	D_{99} (μm)	Specific gravity	Description
$CaCO_3$ 150	150	325		2.7–2.78	Ground marble
$CaCO_3$ 600	600	1125		2.7–2.78	Ground marble
$CaCO_3$ 1200	1200	1489		2.7–2.78	Ground marble
Graphite 100	100	182		1.82	Resilient graphite
Graphite 400	400	744		1.71	Resilient graphite
Ultra-fine cellulose	–	75	90	0.97–1.0	Cellulose fiber
Granular cellulose	–	–	600	1.3	Cellulose fiber
Cellulose LCM blend	425	<3200		1.02–1.04	Cellulose fiber

threaded steel rod was placed into the hot-rolling cell for some of the samples, and the pressure testing was compared with samples where ordinary hot rolling had been conducted. For the tests with high-concentration LCM pills, no hot rolling was used as the pill would normally be prepared just before application in the well.

One representative was selected for testing particle degradation from each of the material categories using a simple high-speed shearing process, as an alternative method to the hot-rolling process for testing material degradation. The materials were selected based on having a significant portion of particles in the range between 200 μm and 1000 μm for ease of sieving. The materials were each mixed into a fluid containing xanthan gum (3.3 kg/m^3) and low viscosity poly-anionic cellulose (11 kg/m^3), to reflect the viscosity of a typical drilling fluid. One same of each product was then wet-sieved after 10 min of mixing at normal speed. The other sample was sheared at full speed on a Hamilton Beach mixer for 30 min and then wet-sieved.

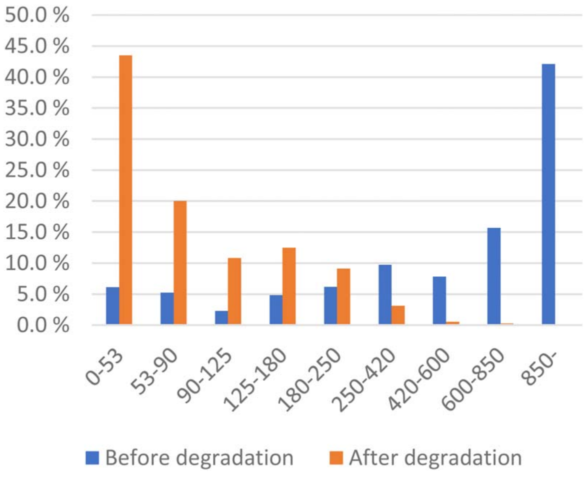

Fig. 2 PSD of $CaCO_3$ with D_{50} approximately at 600 μm before and after degradation

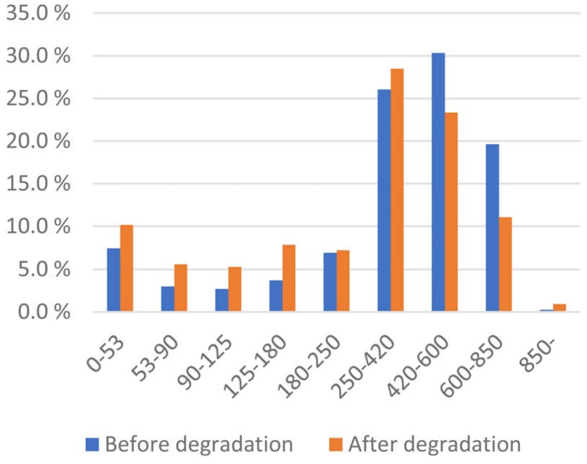

Fig. 3 PSD of graphite with D_{50} approximately at 400 μm before and after degradation

Figures 2–4 show the PSD of the respective materials with the normal mixing process to represent the material before degradation and after the high-speed mixing to represent the materials after degradation. The measurements were conducted using wet sieving on a sieve shaker with American Petroleum Institute (API) rated sieves. It should be noted that the samples that were hot rolled were not exposed to the high-speed mixing process.

The degradation process showed considerable change in the particle size distribution of the $CaCO_3$ particles, some reduction in the PSD of the resilient graphite, and very little change in the PSD of the granular cellulose. For the $CaCO_3$, 99% of the particles initially above 420 μm were finer than 420 μm after the high-speed shearing. For the resilient graphite, the reduction in particles above 420 μm was 30% and for the granular cellulose, it was only 5%.

3 Measurements and Results

The tests are separated into four different test series. Tests were conducted in oil-based and water-based drilling fluids with high-concentration LCM pills and with lower concentration preventative treatment recipes.

3.1 Pressure Measurement. The tests were by recording the applied pressure relative to ambient pressure every second. The pressure source was a hydraulic hand pump where the pressure was applied through regular pumping. The applied hydraulic pressure moves a piston within the test cell, which then transfers the pressure to the drilling fluid.

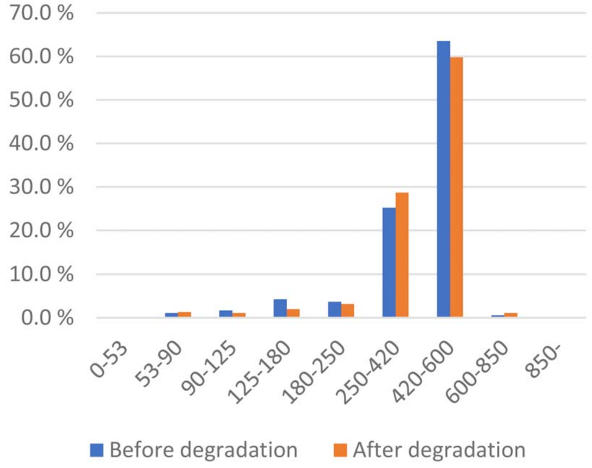

Fig. 4 PSD of granular cellulose before and after degradation

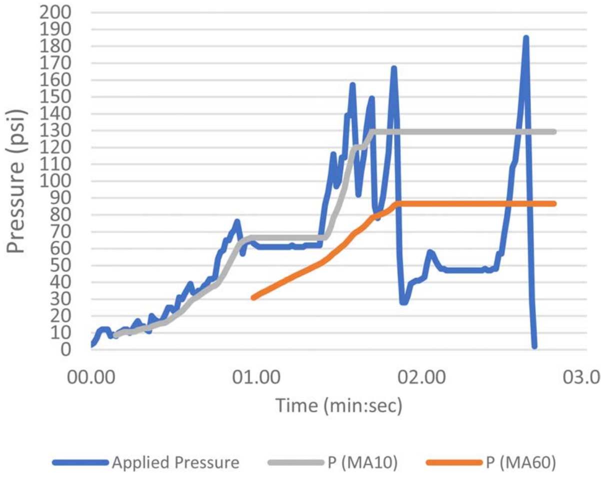

Fig. 5 Example of pressure chart with granular LCM in oil-based drilling fluid

Figure 5 shows the example of a pressure chart where both the highest achieved PHP and SHP are plotted. An oil-based fluid with pill number 5, shown in Table 5, was tested on a 2.0 mm slotted disc. The pressures are calculated according to Eqs. (1)–(3). The raw plot of the applied pressure presents a series of sharp peaks, where the pressure rises for periods shorter than 10 s. The highest recorded pressure reading in the specific test was 185 psi. The PHP was 129 psi, whereas the SHP was significantly less, 87 psi. Considering that the pressure collapsed multiple times and that the peak was only recorded in one instance it seems natural that the peak of 185 psi is not used to represent the sealing capacity of the test. Moving to the PHP, which is calculated as the highest 10 s average, there are four periods where the pressure exceeds the PHP level. However, the longest recorded period above the PHP level was a four-second period. For tests where the PHP and SHP were in the range of less than 1000 psi, the ratio of the SHP and PHP was often in the range of 60–80%. For such tests, it may be that a higher fluid flowrate, facilitated for example by a pressurized gas source instead of a hydraulic pump might have led to a more effective sealing. For tests where the highest pressures obtained were exceeding 2000 psi, the ratio of the SHP to PHP was consistently above 90%. Due to the high losses and high pressures, a gas source was considered to be too risky to operate in a laboratory condition. The four-test series described in the following are therefore presented in terms of the sustainable hold pressure where a high pressure was maintained over time, whereas the PHP is presented for certain tests where it was difficult to achieve a seal with the given fluid flow.

3.2 High Pressure Testing of Preventative Lost Circulation Materials in Oil-Based Drilling Fluid.

Two recipes for preventative treatment of lost circulation were mixed into a barite-weighted oil-based drilling fluid with the presence of fine drill solids and measured density of 1.49 s.g. as shown in Table 2. The fluid was described by the supplier as a high-performance nonaqueous drilling fluid, with a low odor hydrocarbon base. Two samples of each fluid were mixed and hot rolled at 90 °C for 16 h. For each fluid, one sample was hot rolled in the conventional way, and one with the addition of a rod to simulate downhole mechanical wear on the fluid particles during the hot rolling. The rod was a 13.5 cm long M16 threaded steel rod placed in a 500 cm^3 cell. A threaded rod was chosen to enlarge the surface area to detect any accretion and to facilitate that both small and large particles may be exposed to the pressure from the rod. With an un-threaded rod, the main wear would be on the largest particles.

After hot rolling, the fluid samples were used in a lost circulation test on a slotted disc with 500 μm slot apertures. The pressure plots are shown in Fig. 6. For the tests with normal hot rolling, both fluids performed well and enabled high sealing pressures over a 60 s period. For fluid 1, without cellulose-based LCM, the highest sustainable hold pressure was 4182 psi before the fluid loss reached 275 mL, whereas the test for fluid 2 (with cellulose-based LCM) was stopped with an SHP of 5374 psi, due to the pressure approaching the set limit at 5500 psi. At the time, the measured fluid loss was only 13 mL.

Table 2 Oil-based fluid recipes

LCM additive into recipe for 1 L of fluid	Fluid 1: Granular LCM	Fluid 2: Granular and cellulose LCM
Oil-based drilling fluid (g)	1432	1417
$CaCO_3$ 150 (g)	24.5	24.5
$CaCO_3$ 600 (g)	24.5	24.5
Graphite 100 (g)	24.5	–
Graphite 400 (g)	12.25	–
Ultra-fine cellulose	–	8.6
Granular cellulose	–	28.5

Thereafter, the tests were repeated with the fluid samples that have been exposed to mechanical wear by the inclusion of a threaded steel rod in the hot-rolling cell. For fluid 1, the highest recorded SHP was 302 psi when a fluid loss of 275 mL was reached. For fluid 2, also a noticeable change was recorded relative to the first sample. A larger fluid loss was recorded; however, the pressure reached an SHP level of 4689 psi. Following the degradation tests in Sec. 2.2, it may be assumed that only the granular cellulose particles of fluid 2 were intact and equivalent to the slot size after the hot-rolling process with the steel rod. Therefore, in these tests, the concentration of LCM that was similar to or larger than the slot aperture size was around 28.5 kg/m^3 or slightly in excess of 2% by volume.

3.3 High Pressure Testing of Preventative Lost Circulation Materials in Water-Based Drilling Fluid.

The preventative LCM mixtures used in Sec. 3.2 were mixed into a water-based drilling fluid as shown in Table 3 and hot rolled with and without a threaded steel rod. The recipes included bentonite to represent fine drill solids.

The pressure tests were conducted on a slotted disc with 0.50 mm apertures, as for the tests with the oil-based drilling fluid. The pressure plots are shown in Fig. 7. Also in these tests, a significant difference was recorded for the samples where the fluid had been exposed to mechanical wear during the hot-rolling process. Without the mechanical wear, the results for fluid 3, with the granular LCM, were very similar to the results obtained for fluid 1 as an SHP pressure in the region of 4200 psi was achieved. For the sample with the mechanical shear, fluid 3 registered an SHP in excess of 1000 psi, or more than three times the SHP for fluid 1, with granular LCM in an oil-based drilling fluid. In contrast, the SHP of 3981 psi obtained for fluid 4 after mechanical wear was a little lower than for fluid 2 after the same mechanical exposure. However, in all tests, the fluid samples with the combined granular and cellulose-based LCM showed significant improvements in sealing strength and fluid loss over the formulations with granular LCM only. Also, it appears that the addition of cellulose-based LCM provided significantly higher sealing strength after exposure to mechanical wear.

3.4 High Pressure Testing of Lost Circulation Materials in Water-Based Drilling Fluid.

Three recipes were mixed of LCM pills into a water-based fluid with a density of 1.4 s.g. The recipe of the base fluid and the pills are shown in Table 4.

The LCM pills were tested to achieve the highest sealing pressure before a fluid loss of 275 mL was recorded or until s SHP of

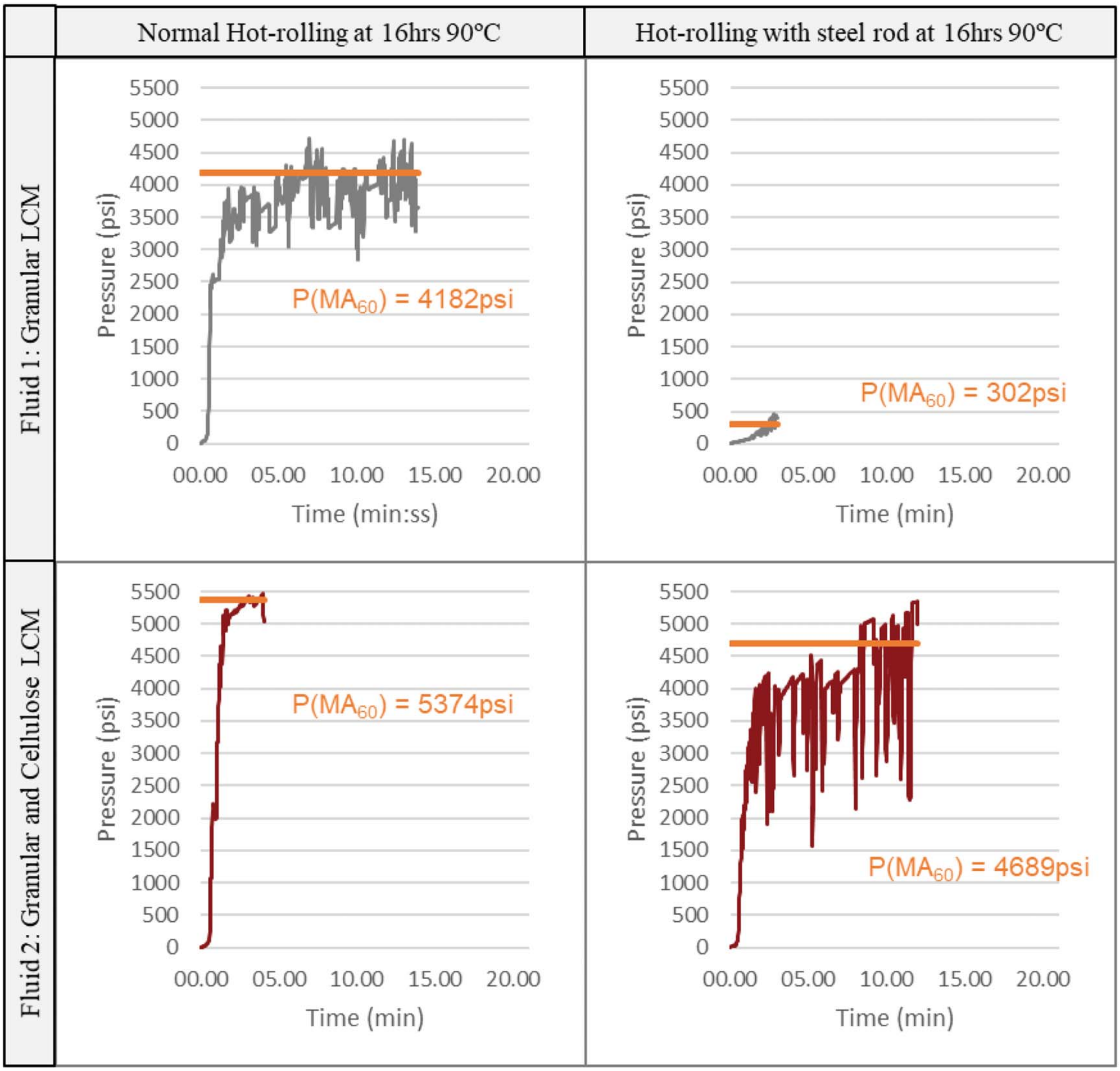

Fig. 6 Pressure charts for preventative LCM in oil-based drilling fluid

Table 3 Water-based fluid recipes

LCM additive into recipe for 1 L of fluid	Fluid 3: Granular LCM	Fluid 4: Granular and cellulose LCM
H_2O (g)	817	817
Na_2CO_3 (g)	0.055	0.055
NaOH (g)	0.69	0.69
Xanthan gum (g)	3.32	3.32
Poly-anionic cellulose (g)	11.05	11.05
MgO (g)	2.77	2.77
KCl (g)	48.3	48.3
Bentonite (g)	13.8	13.8
Barite (g)	464	464
$CaCO_3$ 150 (g)	24.5	24.5
$CaCO_3$ 600 (g)	24.5	24.5
Graphite 100 (g)	24.5	–
Graphite 400 (g)	12.25	–
Ultra-fine cellulose	–	8.6
Granular cellulose	–	28.5

5000 psi was achieved. The sustainable hold pressures are shown in Fig. 8. All three pills achieved a sealing pressure in excess of 5000 psi on the disc with a 750 μm slot width. As the disc slot with increased, the performance of the different pills deviated increasingly more. Pill 1 achieved a PHP of 628 psi on the 1.5 mm disc, whereas pill 2 achieved a PHP of 217 psi on the 2.0 mm disc. In contrast, pill 3 sealed the disc with the 5.0 mm slot up to a PHP of 1347 psi.

3.5 High Pressure Testing of Lost Circulation Materials in Oil-Based Drilling Fluid.

The LCM concentrations for pills 1–3 used in Sec. 3.4 were mixed into a barite-weighted oil-based fluid with a density of 1.49 s.g. as per Table 5 to make up three LCM pills. By doing so, pill 4 would correspond to pill 1, pill 5 to pill 2, and pill 6 to pill 3, with the difference being the drilling fluid base.

The first tests were conducted on a disc with a 1.5 mm slot width for comparison of the performance with the results from testing the pill formulations in the water-based drilling fluid.

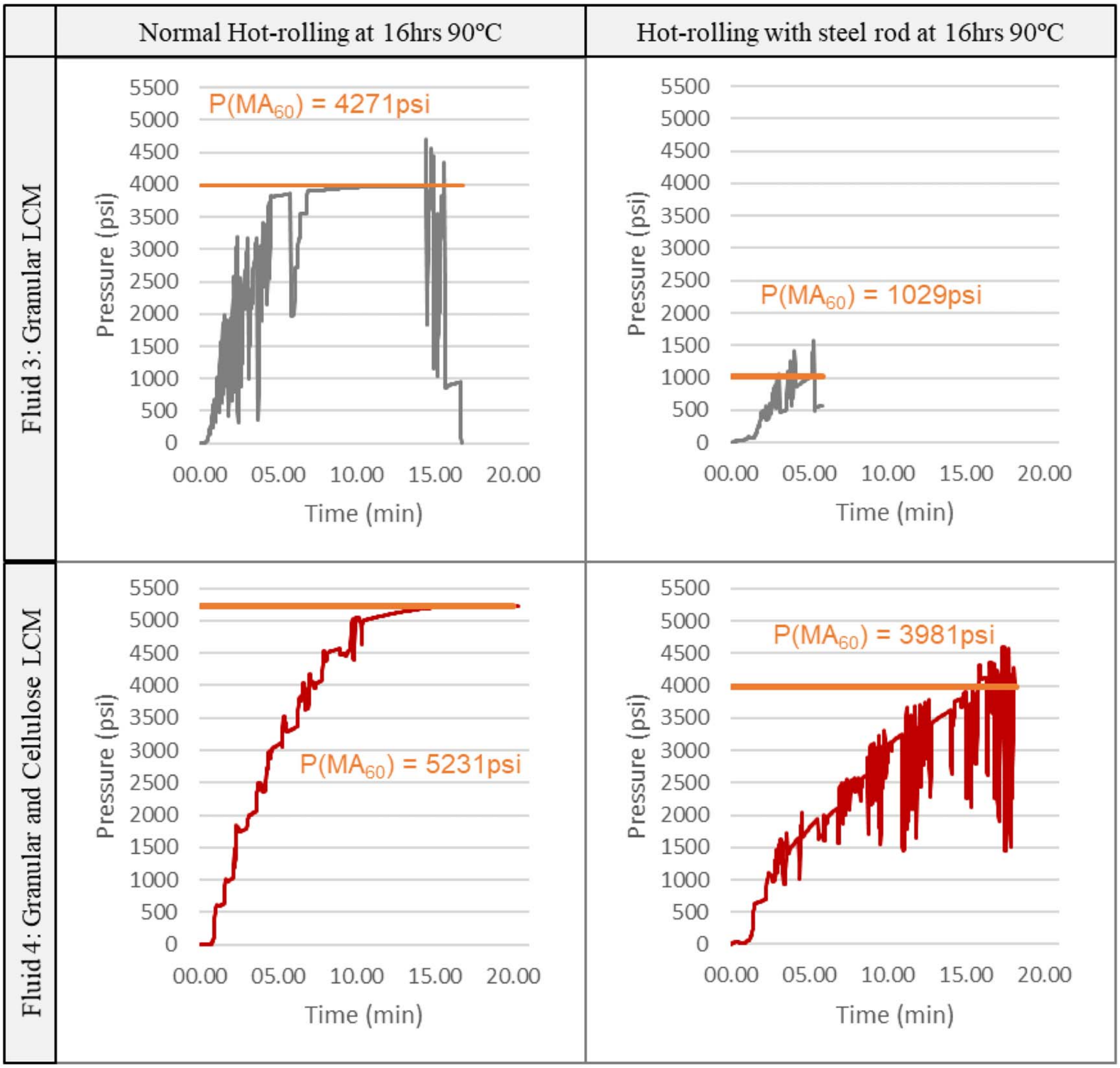

Fig. 7 Pressure charts for preventative LCM in water-based drilling fluid

Table 4 Recipes for LCM pills 1–3

Recipe for 1 L	Pill 1: 350 kg/m^3 granular LCM	Pill 2: 450 kg/m^3 granular LCM	Pill 3: 155 kg/m^3 cellulose
H_2O (g)	718.2	718.2	718.2
Na_2CO_3 (g)	0.05	0.05	0.05
NaOH (g)	0.61	0.61	0.61
Xanthan gum (g)	2.91	2.91	2.91
Poly-anionic cellulose (g)	9.71	9.71	9.71
MgO (g)	2.43	2.43	2.43
KCl (g)	42.5	42.5	42.5
Bentonite (g)	12.14	12.14	12.14
Barite (g)	408	408	408
$CaCO_3$ 150 (g)	100	100	–
$CaCO_3$ 600 (g)	100	100	–
$CaCO_3$ 1200 (g)	–	75	–
Graphite 100 (g)	100	100	–
Graphite 400 (g)	50	75	–
Cellulose LCM blend (g)	–	–	155

The tests were thereafter selected to be on either smaller or larger apertures, due to a limited supply of the oil-based field fluid. As pill 4 only achieved an SHP of 115 psi on the 1.5 mm disc, it was selected to be re-run on the 750 μm slotted disc. On this disc, the sealing pressure increased to 3788 psi. As pill 5 achieved a higher sealing pressure with PHP of 914 psi on the 1.5 mm slotted disc, it was re-tested on the 2.0 mm disc. Here the pill achieved a PHP of 129 psi. In sum, pills 4 and 5 achieved significantly lower sealing pressures when applied into the oil-based drilling fluid. The results for pill 6 were very much in line with the results of the testing in the water-based drilling fluid. The 1.5 mm and 2.0 mm slotted discs were both successfully sealed with pressures exceeding 5000 psi and the 3.5 mm disc was sealed with an SHP 2000 psi. The result on the disc with the 5.0 mm slot gave a PHP than the SHP on the 3.5 mm slot with a small margin. The pressure charts for pills 4–6 are shown in Fig. 9. It should, however, be noted that the fluid loss on the discs with large widths is erratic due to the high loss occurring once a seal is broken and that the slow buildup of hydraulic pressure may provide different results than for field conditions.

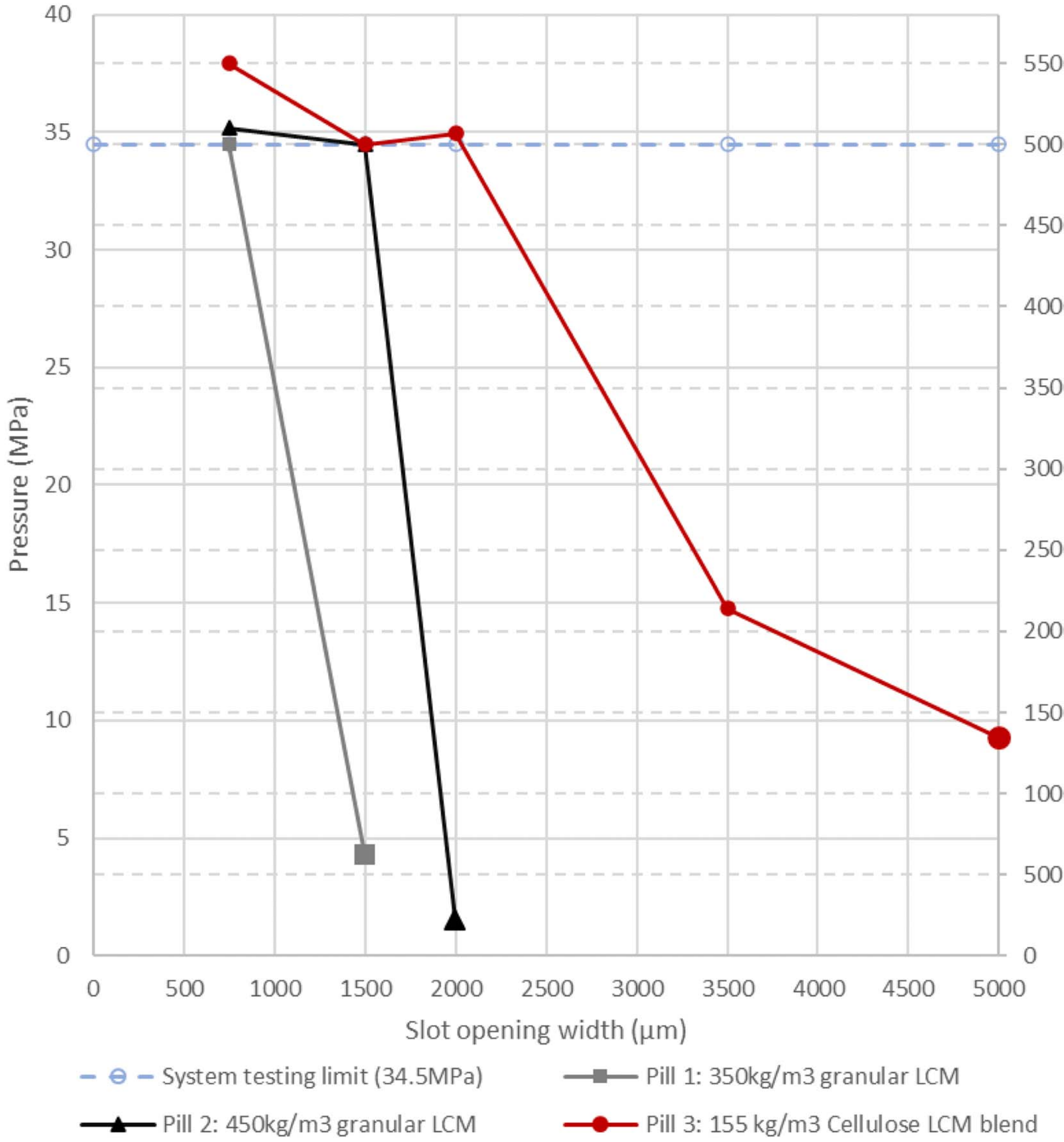

Fig. 8 Pressure charts for LCM pills in water-based drilling fluid

For the tests with cellulose-based LCM pills, the peak hold pressures exceeding 1300 psi were achieved in both oil- and water-based fluids even when the slot size was 1.5 times larger than the D_{90} value of the particles. In contrast, with the granular LCM mixtures, PHP exceeding 1000 psi was only achieved when the largest particles were around the width of the slot.

3.6 Discussion. The results of the testing of preventative LCM treatments are presented in Sec. 3.2 support the findings of Scott et al. [9] for the test conducted with conventional hot rolling, where the particle size (D_{90} or D_{99}) of the $CaCO_3$ 600, graphite 400, and the granular cellulose products was consistent with the sealing of the 500 μm slotted disc. For these tests, LCM particles with sizes equal to or slightly larger than the slot openings were present in adequate concentrations for effective sealing. The exposure to mechanical wear altered these results significantly. This shows the importance of testing fluids and LCM under conditions that replicate the mechanical wear which may be present in a specific field operation. The results differ from those of Vivas and Salehi [11], who tested thermal degradation of LCM for geothermal wells, however, without exposure to mechanical wear and without the presence of drill solids. They found granular materials to function well as LCM for 1000 μm slots and pressure up to 6.2–8.3 MPa or 900–1200 psi.

Two different methods for material degradation were used during the study. Although the PSD changes using the two different methods were not directly compared, the test results showed that both methods led to significant degradation of some materials and little degradation of others. The high-speed mixing process led to a very high degradation (99% >420 μm) of the $CaCO_3$ particles, which was a significant contrast to the resilient graphite that showed some degradation (30% > 420 μm) and the granular cellulose that showed very little degradation (5% >420 μm). The PSD

Table 5 Recipes for LCM pills 4–6

Recipe for 1 L	Pill 4: 350 kg/m^3 granular LCM	Pill 5: 450 kg/m^3 granular LCM	Pill 6: 155 kg/m^3 cellulose LCM blend
Oil-based drilling fluid (g)	1267	1267	1267
$CaCO_3$ 150 (g)	100	100	–
$CaCO_3$ 600 (g)	100	100	–
$CaCO_3$ 1200 (g)	–	75	–
Graphite 100 (g)	100	100	–
Graphite 400 (g)	50	75	–
Cellulose LCM blend (g)	–	–	155

degradation results were also consistent with the measured changes in sealing performance where the $CaCO_3$ particles were combined with either resilient graphite or granular cellulose. The combination of $CaCO_3$ and resilient graphite (fluids 1 and 3) resulted in the SHP falling by 93% in oil-based drilling fluid and 76% in the water-based drilling fluid, whereas the SHP fell only by 13% in oil-based drilling fluid and 24% in water-based drilling fluid with the $CaCO_3$ and cellulose mixture (fluids 2 and 4). For these tests with water-based fluids without degradation, it should be noted that the measured SHP for the tests was limited by the maximum test pressure.

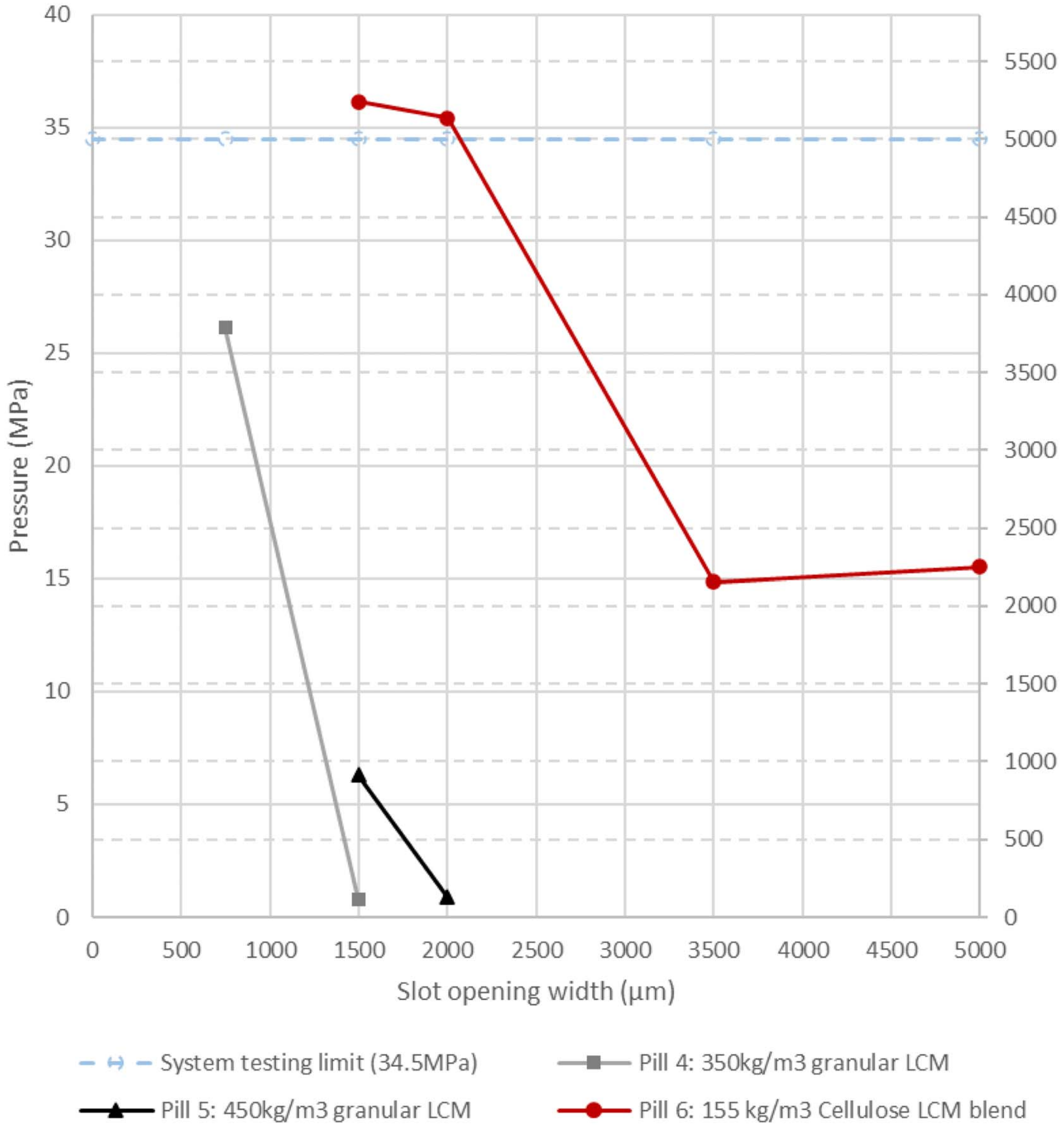

Fig. 9 Pressure charts for LCM pills in oil-based drilling fluid

The results of the degradation tests strongly indicate that ground marble or $CaCO_3$ has clear disadvantages when applied as a fracture-sealing or wellbore-strengthening material. The very high material degradation indicates that the specified product PSD is unsuitable to indicate the material's capacity to seal fractures or large pore-openings in a drilling situation where the material may be exposed to mechanical wear.

The resilient graphite showed considerably better performance than the $CaCO_3$. However, in both tests where mechanical wear had been introduced, the sealing effectiveness fell very significantly so that a high rate of product replenishment would be required to maintain a satisfactory sealing performance.

The granular cellulose particles provided the best resistance toward mechanical degradation and also provided the highest sealing pressures. It should be noted that this was the case despite the granular cellulose particles having a D_{99} value of 600 μm, which is considerably lower than the specified D_{90} values of the $CaCO_3$ of 1125 μm and the resilient graphite of 744 μm.

One likely reason for the difference in sealing strength and mechanical wear resistance of the materials is the mechanical toughness of the particles. In materials science, toughness is described as the ability of a material to absorb energy and plastically deform without fracturing. Equation (4) describes the toughness from a mechanical perspective, where σ is the stress applied, ε is the material strain, and ε_f is the strain upon failure

$$\frac{\text{Energy}}{\text{Volume}} = \int_0^{\varepsilon_f} \sigma d\varepsilon \qquad (4)$$

Toughness tests were not conducted on the materials to verify if this could be a method for differentiating the properties of materials. However, by simply grinding a sample of each material between fingers, it is clear that the $CaCO_3$ degrades very quickly, the graphite degrades much less, and the granular cellulose does not degrade noticeably. The findings related to materials degradation may also have some relevance for the tests with the LCM pills, where also the cellulose blend of pill 3 clearly outperformed the sealing capacity of the granular materials used in pills 1 and 2. For the application of LCM in a high-concentration pill, the toughness of the materials may be less relevant from a fluid circulation perspective, as the LCM particles will normally be pumped with a low flowrate to the loss zone. As such, the particles will likely not be degraded in the same manner as LCM particles that are part of the circulating system and sheared whilst being pumped through the bit. During the process of sealing a fracture, the particles will be squeezed together, and less tough particles may degrade during the sealing process.

The degradation tests identified the $CaCO_3$ as a substantially less wear-resistant material than the resilient graphite and the granular cellulose. A hypothesis is, as the seal is formed, the $CaCO_3$ particles break up to fill the voids between the more resilient graphite or granular cellulose particles. If so, this may impact the resilience of the seal toward disturbances in the wellbore relative to seals where the materials elastically adapt to create a low-permeability zone.

Significant differences were observed when applying granular LCM in oil-based fluids relative to water-based fluids. Corresponding differences were not observed when applying cellulose-based LCM materials. A reason for this difference may be related to particle–particle interaction.

In a dispersed water-based fluid, the particles will move independently upon the circulation. When a seal is created in the filter cake against a permeable formation or against a fracture, the particles will be forced together as the filter cake or seal dehydrates. With cellulose-based materials, polar interaction will occur between the cellulose particles themselves, but also between the cellulose particles and other polymers such as poly-anionic cellulose, xanthan gum, and starch. As such, there will be frictional or adhesive forces between the particles, partly like a paper manufacturing process. The filter cake will therefore be very strong and elastic.

When applying inert granular particles in a water-based drilling fluid, it is likely that there will still be present frictional- or adhesive forces between the polymer particles in a seal and that these forces enhance the seal integrity over that which might be achieved by granular particles alone, and that these forces increase as the seal is de-hydrated. The polymers will in such a situation develop an elastic filter cake. Hence, the filter cake can be structured as a separate entity and not be considered to be constructed as a formation of individual particles.

In summary, the higher the concentration of polymers and cellulose-based fibers in the filter cake, the more cohesive it will be. If cellulose-based fibers are replaced in part or in full by inert LCM, the cohesive strength of the filter cake will be reduced correspondingly.

Oil-based drilling fluids are generally considered superior to water-based drilling fluids with regards to lubricity and fluid loss in low- to medium permeability formations. Majid et al. [12] found that in water-in-oil emulsions, water forms small droplets with a size typically smaller than 5 μm in a well-sheared suspension. From a fluid loss perspective, the water may be seen as a particle suspended in the base fluid. Furthermore, Wang and Du [13] found that the D_{50} value of a certain barite powder was in the region of 15–20 μm and that the largest particles may be up to circa 75 μm. Combining the PSD of the water droplets and barite as a weighting agent, a typical oil-based drilling fluid will have a high concentration of particles. Following the Abrams Rule [14], the D_{50} value of the barite suggests that a barite-weighted fluid may effectively seal formations with pore sizes up to circa 60 μm. As a supplement to the barite particles, the high concentration of water droplets with a size <5 μm provides a very effective fine-sealing mechanism.

Unless mechanically or chemically disturbed, water droplets have a high sphericity in a water-in-oil emulsion. For formations where the barite has sufficient size to bridge the pore throats, the smaller water droplets will act as a fine sealant. No polymeric additives are used to create a long strain range elastic gel within the filter cakes.

For LCM used as part of the circulating system, other considerations should also be made with regards to impacting the overall functionality of the drilling fluid in the well. The size, shape, and adhesive forces may impact the equivalent circulating density or the formation of a filter cake on the wellbore wall. Particles that may form a cohesive network may be better suited for pill applications as any increase in fluid viscosity will be less important for such applications. In a dynamic condition, the tensile strength of the filter cake may impact its ability to withstand the erosion caused by the flow of fluid and hence provide a lower continuous fluid loss rate. In such an application, cohesive forces between LCM particles may improve the tensile strength of the filter cake and hence the wellbore stability.

The cellulose-based products used in the test are of different nature and shape. However, when comparing with the $CaCo_3$ and resilient graphite particles, it is clear that the cellulose particles have very low relative sphericity or high aspect ratio. The low sphericity of the cellulose-based particles and the polar interaction between cellulose particles under applied differential pressure may be a differentiating factor relative to the granular LCM particles.

In essence, fluid 1, consisting of a barite-weighted water-in-oil emulsion with $CaCO_3$ and resilient graphite may appear as a high concentration of medium- to high sphericity particles dispersed in a base fluid. Whenever the formation of pore throats or fractures are smaller than a critical size of the particles in the fluid, e.g., where the largest particles in the fluid are equivalent to the fracture aperture or the pore-throat size, the fluid acts very effectively to seal. However, once the pore-throat size or fracture aperture exceeds this critical particle size, the appearance of the fluid may be that of a naturally lubricating roller-bearing system. In contrast to a water-in-oil emulsion, water-based drilling fluid will have dispersed polymers that have very low sphericity and that may

Table 6 Hypothesis for mechanical interaction between particles in various fluid compositions during sealing of pore throats or fractures

	Water-in-oil emulsion	Water-based fluid with polymers for fluid loss and viscosity
Granular inert LCM particles with medium- to high sphericity	Total fluid appears as a dispersed spherical particle system with very high particle concentration and low particle-to-particle adhesive and frictional forces upon defluidization	Total fluid appears as a dispersed particle system with medium concentration of low and high sphericity particles and some particle-to-particle adhesive and frictional forces upon defluidization
Cellulose-based particles with low sphericity	Total fluid appears as a dispersed particle system with very high concentration of high sphericity particles and some low sphericity particles and with some particle-to-particle adhesive and frictional forces upon defluidization	Total fluid appears as a dispersed particle system with medium concentration of low sphericity particles and high particle-to-particle adhesive and frictional forces upon defluidization

Table 7 Hypothesis for sealing effectiveness of various fluid compositions for sealing of pore throats or fractures

	Water-in-oil emulsion	Water-based fluid with polymers for fluid loss and viscosity
Granular inert LCM particles with medium- to high sphericity	Very effective sealing up to critical pore throat or fracture size by effective particle packing Above critical pore throat or fracture size sealing ability sharply drops as the fluid behaves like a roller-bearing system	Effective sealing up to critical pore throat or fracture size by particle packing and interactive forces Above critical pore throat or fracture size sealing ability gradually drops as adhesive and frictional forces become less effective
Cellulose-based particles with low sphericity	Very effective sealing up to critical pore throat or fracture size by effective particle packing Above critical pore throat or fracture size sealing ability gradually drops as the fluid moves toward behaving like a roller-bearing system	Effective sealing up to critical pore throat or fracture size by particle packing and interactive forces Above critical pore throat or fracture size sealing ability slowly drops as adhesive and frictional forces become less effective

combine through polar molecule interaction. Also, by replacing granular inert particles in part or in full by cellulose-based particles, the polar interaction and thereby also the particle-to-particle adhesive and frictional forces are increased.

It may therefore be that the sealing mechanisms may be described as shown in Table 6 and the sealing effectiveness as shown in Table 7.

4 Conclusion

- The application of a simple moving average to identify the peak hold pressure (10 s moving average) and sustainable hold pressure (60 s moving average) provided a good and non-subjective way of measuring the pressures during LCM tests. The PHP became the most relevant metric for measuring when the LCM seal failed, whereas the SHP reflected a more reliable sealing pressure.
- The sealing effectiveness for cellulose-based LCM appeared to be reasonably similar in oil-based and water-based drilling fluids. In contrast, granular LCM was found to create stronger seals in water-based drilling fluids than in oil-based drilling fluids.
- The highest sealing pressures and lowest fluid losses were obtained when applying cellulose-based LCM. Also, the cellulose-based materials showed the ability to seal slotted discs up to 5.0 mm. In contrast, the granular LCM appeared to function very well up to certain limits. Once these limits were reached, the sealing capacity dropped sharply.
- The method for applying mechanical shear in the hot-rolling process strongly differentiated the sealing performance of the materials relative to the samples without mechanical wear. The fluid loss results of the various material classes were impacted in a way that was consistent with the PSD degradation measured using high-speed mixing tests.
- For preventative treatment of lost circulation where the fracture size is known, wear-resistant particles with a size equal to or slightly larger than the fracture size appear to be an effective treatment with a volumetric concentration of 2%.
- For LCM pill application, cellulose-based additives achieved sealing with pressure exceeding 1300 psi when the slot size was around 1.5 times the D_{90} value of the particles, whereas the granular LCM mixture only achieved high sealing pressures when the largest particles were around the slot size.
- For analyzing the effectiveness of preventative treatment of lost circulation, the drilling fluid with LCM additives should be exposed to relevant thermal and mechanical wear prior to testing.

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Conflict of Interest

There are no conflicts of interest.

Nomenclature

n = time period n
P_t = pressure at time t

Appendix

The equipment setup was as follows.

Conventional equipment used for HTHP fluid loss testing:

- Hamilton Beach Mixer
- Ohaus Pioneer Precision PX3202
- Ofite Filter Press HTHP 175 mL, Double Capped
- Ofite Viscometer model 900
- Ofite roller-oven #172-00-1-C
- Apera pH90, pH meter

Special experimental setup:

- Ohaus MB120 Moisture Analyzer
- Custom built transparent acrylic cell for enabling of reverse flow of fluid through the ceramic discs
- Festo pressure regulator LRP-1/4-2.5 and LRP-1/4-0.25
- Festo pressure sensor SPAN-P025R and SPAN-P10R
- Festo flowmeter SFAH-10U
- Nitrogen source and manifold for pressure up to 1350 psi, Ofite #171-24
- Vacuum machine, DVP EC.20-1
- Custom build permeability plugging apparatus with hydraulic pump for testing on slotted discs or ceramic discs up to 35 MPa (5076 psi)
- AEP transducers JET pressure gauge with data logger

References

[1] Alsaba, M., Nygaard, R., and Hareland, G., 2014, "Review of Lost Circulation Materials and Treatments With an Updated Classification," 2014 AADE Fluids Technical Conference and Exhibition, Houston, TX, Apr. 15–16, Paper No. AADE-14-FTCE-25.

[2] Alshubbar, G., Nygaard, R., and Jeennakorn, M., 2018, "The Effect of Wellbore Circulation on Building and LCM Bridge at the Fracture Aperture," J. Pet. Sci. Eng., **165**, pp. 550–556.

[3] Alsaba, M., Nygaard, R., Saasen, A., and Nes, O. M., 2014, "Lost Circulation Materials Capability of Sealing Wide Fractures," SPE Deepwater Drilling and Completions Conference, Sept. 10–11, Paper No. SPE-170285-MS.

[4] Khalifeh, M., Klungtvedt, K. R., Vasshus, J. K., and Saasen, A., 2019, "Drilling Fluids—Lost Circulation Treatment," SPE Norway One Day Seminar, May 14, Paper No. SPE-195609-MS.

[5] Alvi, M. A. A., Belayneh, M., Fjelde, K. K., Saasen, A., and Bandyopadhyay, S., 2021, "Effect of Hydrophobic Iron Oxide Nanoparticles on the Properties of Oil-Based Drilling Fluid," ASME J. Energy Resour. Technol., **143**(4), p. 043001.

[6] Contreras, O., Alsaba, M., Hareland, G., Hussein, M., and Nygaard, R., 2016, "Effect on Fracture Pressure by Adding Iron-Based and Calcium-Based Nanoparticles to a Nonaqueous Drilling Fluid for Permeable Formations," ASME J. Energy Resour. Technol., **138**(3), p. 032906.

[7] Klungtvedt, K. R., and Saasen, A., "The Role of Particle Size Distribution for Fluid Loss Materials on Formation of Internal Filter-Cakes and Avoiding Formation Damage," ASME 2022 41th International Conference on Ocean, Offshore and Arctic Engineering, Hamburg, Germany, June 21–30, Paper No. OMAE2022-79501.

[8] Grelland, S. S., 2021, "Analysis of Lost Circulation on the Norwegian Continental Shelf," M.Sc. thesis, University of Stavanger, Stavanger.

[9] Scott, P., Redburn, M., and Nesheim, G., 2020, "A Pragmatic Approach to Lost Circulation Treatments: What Every Drilling Engineer Should Know," 2020 AADE Fluids Technical Conference and Exhibition, Houston, TX, Apr. 14–15, Paper No. AADE-20-FTCE-062.

[10] Hoxha, B. B., Yan, L., Hale, A., and van Oort, E., 2016, "Automated Particle Size Analysis Using Advanced Analysers," 2016 AADE Fluids Technical Conference and Exhibition, Houston, TX, Apr. 12–13, Paper No. AADE-16-FTCE-78.

[11] Vivas, C., and Salehi, S., 2022, "Screening of Lost Circulation Materials for Geothermal Applications: Experimental Study at High Temperature," ASME J. Energy Resour. Technol., **144**(3), p. 033008.

[12] Majid, A. A. A., Saidian, M., Prasad, M., and Koh, C. A., 2015, "Measurement of the Water Droplet Size in Water-in-Oil Emulsions Using Low Field Nuclear Magnetic Resonance for Gas Hydrate Slurry Applications," Can. J. Chem., **93**(9), pp. 1007–1013.

[13] Wang, G., and Du, H., 2017, "Viscous Behavior and Wall Slip of Barite-Weighted Water-Based Drilling Fluids Containing a High Particle Fraction," J. Pet. Sci. Eng., **159**, pp. 773–782.

[14] Abrams, A., 1977, "Mud Design to Minimize Rock Impairment Due to Particle Invasion," J. Pet. Technol., **29**(5), pp. 586–592.