PROCEEDINGS, 48th Workshop on Geothermal Reservoir Engineering
Stanford University, Stanford, California, February 6-8, 2023
SGP-TR-224

Thermal degradation and mixture properties of materials used for lost circulation management

William Kibikas[1,*], Chun Chang[2], Stephen J. Bauer[1], Seiji Nakagawa[2], Patrick Dobson[2], Timothy Kneafsey[2], Abraham Samuel[3]

[1]Sandia National Laboratories, Albuquerque, NM 87185

[2]Lawrence Berkeley National Laboratory, Berkeley, CA 94720

[3]Geothermal Resource Group Inc., Palm Desert, California, USA

*Corresponding author contact info: wmkibik@sandia.gov

Keywords: lost circulation materials; degradation; viscosity; high temperature

ABSTRACT

Loss of drilling fluids is a common problem that represents a significant cost to geothermal projects and delays continued development of geothermal resources. Various materials have been identified or developed which, when mixed with drilling fluids, are known to mitigate lost circulation to varying degrees. Several questions remain unresolved with regards to how the different lost circulation materials will behave, and particularly how they thermally degrade, under different conditions and time frames. To address these issues, we conducted several tests of various lost circulation materials previously used in geothermal drilling operations, measuring how, when mixed with fluids and heated, the materials degrade and affect the viscosity of fluids. Degradation tests were run from 20 to 250 °C, measuring mass loss, water loss, and gas release during degradation of the materials. Viscosity tests were run showing the apparent viscosities with increasing shear rate and temperature of the material mixtures. Together, the results permit quantification of how thermally induced changes in the materials are likely to affect the behavior of drilling fluids in-situ and how long they can reasonably act to seal lost circulation zones.

1. INTRODUCTION

A common problem during drilling operations in the subsurface is the partial or total loss of drilling fluids into highly fractured zones. This loss represents a non-trivial economic cost added to any project, as well as introducing potential environmental and safety issues (Lowry et al., 2022, Winn et al. 2023). Invasion of drilling fluids into the surrounding formations can also produce permeability damage, productivity decline, wellbore stability and other complications that can hinder operations.

High fluid loss during drilling can occur through a number of vectors, including in naturally fractured formations, cavernous or karstic formations, highly permeable zones, or areas where drilling has induced fracturing. Lost circulation can be even more challenging with geothermal well. Factors such as high downhole temperature, large fracture zones, and relatively low fracture gradients are common in geothermal drilling conditions and contribute to the high observed costs of lost circulation treatment (Mohamed et al. 2022). Due to the prevalence of lost circulation in drilling, methods have been employed for more than a century to reduce lost circulation during drilling, either preventing fluid loss from occurring or correcting zones of fluid loss after loss has occurred (Alsaba et al. 2014, Saleh et al. 2020).

Corrective methods are often employed due to the high variability of loss circulation zones and the need for flexible solutions during drilling. For this approach, lost circulation materials (LCM) are added to the drilling fluid (either continuously or in concentrated pills or sweeps), which then ideally will act to plug zones of fluid loss as drilling continues. A wide array of materials has been used in drilling, often depending upon the degree or type of loss occurring, and the efficacy of different materials is debated. For partial losses, granular, fibrous, and flaky LCM are often employed depending on both economic and local conditions (Alsaba et al., 2014). For more severe loss zones, settable polymers are often preferred due to their viscous and cohesive properties.

An issue with employing such LCM is the extreme conditions that necessitated for geothermal wells. In geothermal systems, temperatures are expected to exceed 200 °C, and as technologies improve the pressure-temperature conditions for drilling are expected to continue to increase as more geothermal resources become economically viable (Saleh et al. 2020). However, for many LCM, whether natural or artificial, it is not well understood how they will behave at such extreme conditions. Many commonly employed materials were designed for lower pressure-temperature conditions that occur in oil and gas wells. A concern exists that certain materials will not respond well to such high temperatures, meaning that their efficacy in sealing lost circulation zones is questionable.

This work seeks to address questions about the properties of a number of commonly employed LCM, both natural and artificial, and how they change with conditions relevant to geothermal systems. Thermal degradation of LCM was conducted by heating the materials in pressure vessels to a suite of temperatures and recording the loss of mass of both the fluids and LCM. Microscopic analysis of materials was used to show how degradation altered geometry and structure of each material. Viscosity tests were conducted to measure the response of different LCM mixtures to both increased shearing and heating. These results were used to evaluate what characteristics would be desirable for an LCM in drilling geothermal wells.

2. MATERIALS AND EXPERIMENTAL METHODS

2.1 Lost circulation materials

A large array of different LCM exists and are currently used in drilling operations. For this work, five common particulate materials were selected for testing, based on their prominent use in geothermal drilling operations (Figure 1). Additionally, a suite of specialized gellants developed for sealing significant loss zones was also tested (WCA). A list of all the materials examined and their respective properties can be seen in Table 1.

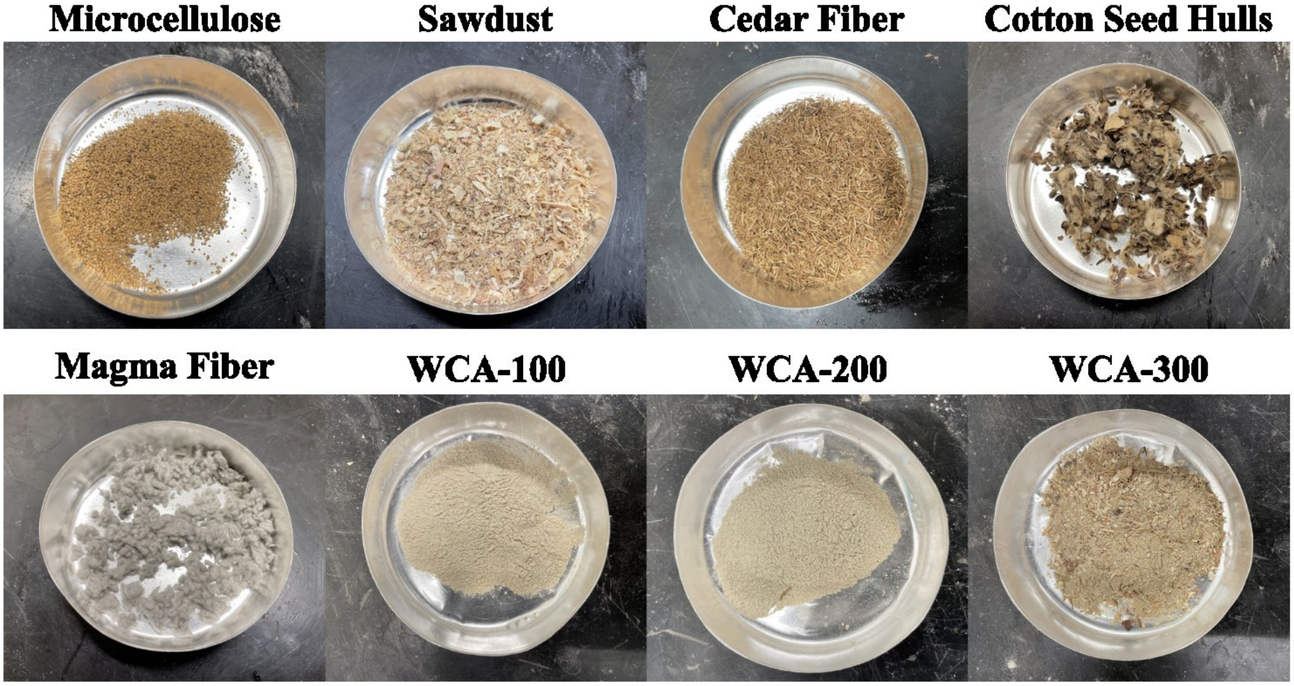


Figure 1: Undegraded LCMs prior to mixing with fluids or heating.

The classification utilized by Caenn et al (2011) is adopted here and used to categorize the materials. Granular materials are typically rounded and stiffer particulates than the other categories with varying grain sizes. Flaky materials are flat with larger surface areas and are ideal for forming a mesh. Fibrous materials are more flexible but are more degradable than other groups. Slurries are products designed to harden with time and are widely variable in their composition, though they are employed conjunctively with other LCM types (Alsaba et al., 2014).

Table 1: LCM evaluated here along with relevant properties

LCM	Density (g/cm3)	Type
Microcellulose	1.60	Granular
Sawdust	0.21	Flaky/Fibrous
Cedar Fiber	0.35	Fibrous
Cotton Seed Hulls	0.19	Fibrous
Magma Fiber	2.60	Fibrous

WCA-100	1.20	Slurry
WCA-200	1.20	Slurry
WCA-300	0.33	Slurry

Micronized cellulose (i.e., microcellulose) is granular LCM. The grains are well rounded and sorted, with an average diameter of 1 mm. Sawdust employed here is both flaky and fibrous, with particles being either elongated chunks or flatter, rounded slivers and exhibit a large variation in diameters. The cedar fibers are elongated particulates that can vary highly in both length and width. The cotton seed hulls and magma fiber are both fine fibrous materials existing primarily as clumps, with the cottonseed hulls being natural products while the magma fiber is an artificial product created for lost circulation plugging. The WCA-100/200/300 are artificial gellants provided by North Star Energy LLC. The WCA-100/200 products are a fine powder that acts as a gellant and are each mixed separately with the additive WCA-300 to form a polymer.

2.2 Degradation tests

The thermal degradation of each LCM was conducted by mixing the LCM with water with specific weight percentages. The mass of both the LCM and water was measured prior to testing then placed in stainless steel cups (Figure 2). Only one material was tested at any given time to avoid issues of mixing occurring during heating. The LCM mixtures (one cup per experiment) were then sealed in a Parr pressure vessel and placed in an oven. The mixtures were then heated at temperatures from 90 to 250 ˚C for durations ranging from 1 to 45 days.

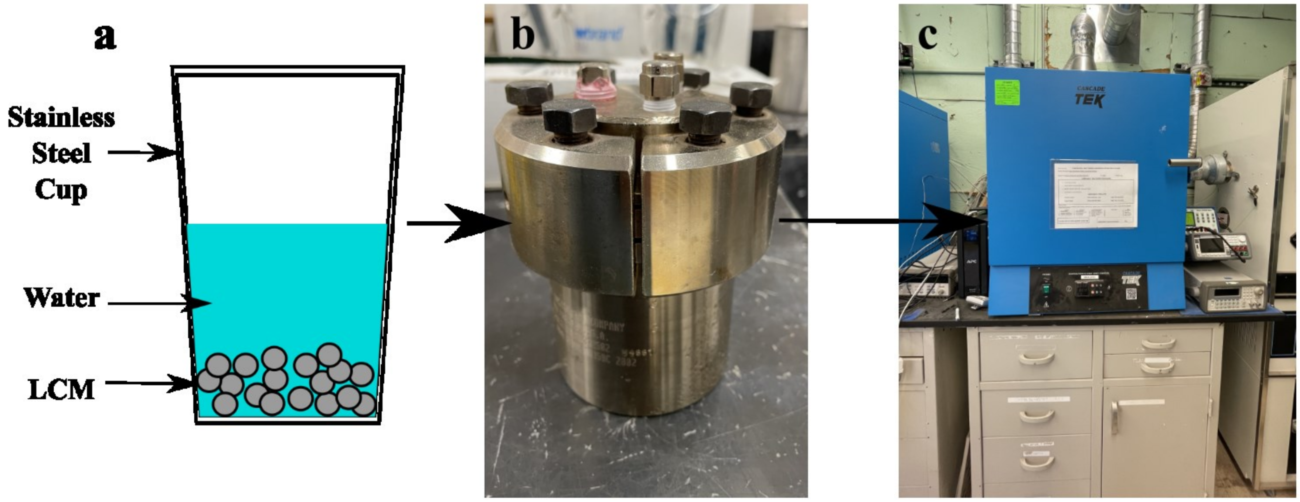


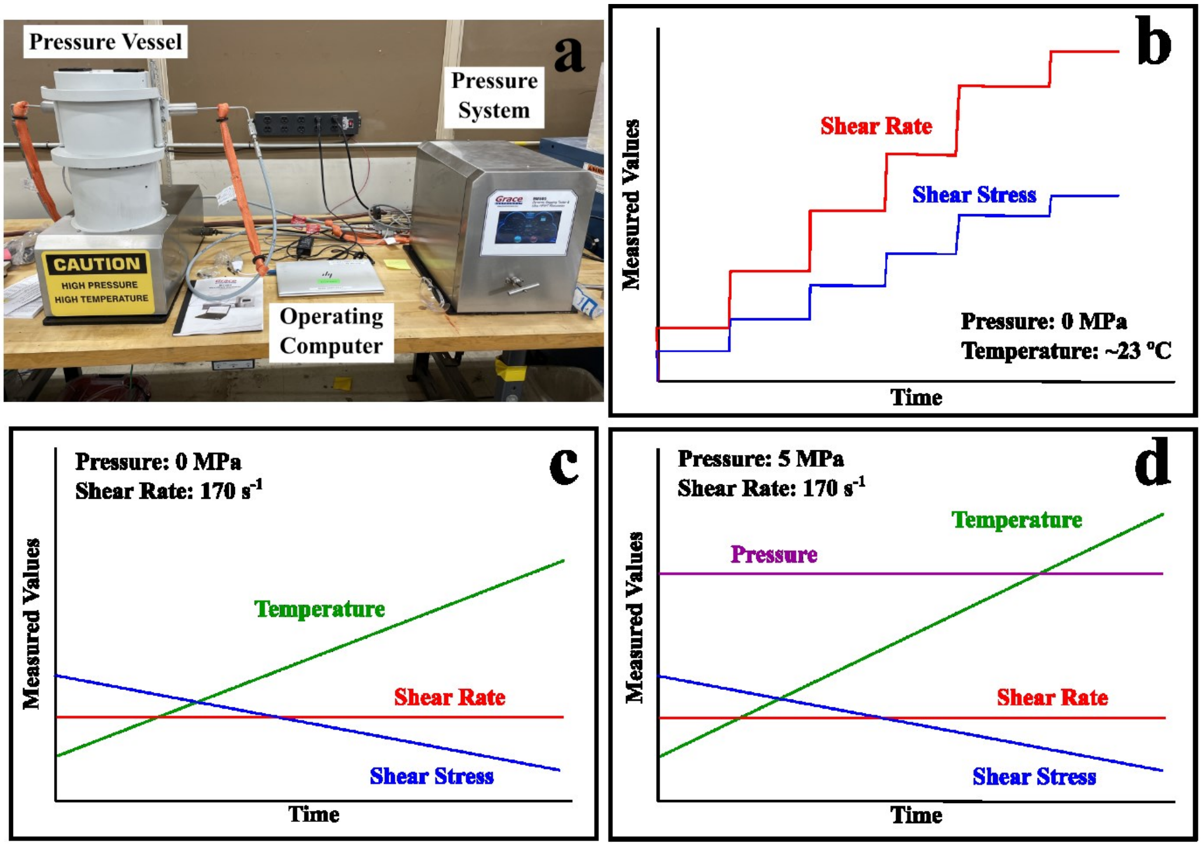
Figure 2: Test setup for degradation tests showing: a) container with both LCM and water; b) the pressure vessel used to seal the LCM mixture during heating; c) the high temperature oven used to heat the samples.

Once testing was completed the ovens were shut down, with both the oven and vessel allowed to return to room temperature to avoid issues of steam generation. Once at ambient conditions, LCM mixtures were removed from the oven and the pressure vessel. The weight of the mixture in each cup was then measured to find the amount of total mass lost (i.e., both LCM and water). The mixtures were then heated to 60 ˚C in a drying oven to vaporize the remaining water in the cups and leave only the LCM. The dried LCM was then weighed to show the total mass lost due to degradation.

2.3 Viscosity tests

The viscosity of the LCM mixtures at various stages of degradation was studied using the M7500 rheometer shown in Figure 3. The microcellulose, sawdust, cedar fiber, cotton seed hulls, and magma fiber were prepared by mixing the LCM with water at proportions of 1.5% LCM and 98.5% water. These weight % correspond ~5 lb of LCM per bbl of water during lost circulation treatment. In drilling, additives are often mixed to increase the viscosity of the fluid. Here, either 0.15% (i.e., 0.5 lb/bbl) of xanthan gum or 9% (i.e., 35 lb/bbl) of bentonite was added to the mixtures as well. The WCA mixtures however utilized different weight ratios. For the WCA mixtures, the

WCA-100/200 gellants were added with ratios of 15 lb/bbl (LCM to water) while the WCA-300 was mixed at a ratio of 40 lb/bbl. A magnetic stirrer was used to mix each test sample for a period of 16-24 hours.



see

Figure 3: M7500 rheometer system used to test viscosity of mixtures (a), and the three test setups used for measuring the viscosity of each LCM mixture: increasing shear rate test at room temperature (b); increasing temperature up to 90 ˚C with constant shear rate (c); increasing temperature up to 200 ˚C with constant pressure and shear rate (d).

Examples of LCM mixtures before testing are shown in Figure 4. Once stirring was finished, the samples were added to the pressure vessel and sealed to begin testing. Three types of tests were run. Initially the samples were sheared at increasing rates under ambient conditions to measure the viscosities of the LCM mixtures. The second test type involved shearing each sample with a constant rate (~100 rpm or 170 s^{-1}) while increasing temperature to 90 ˚C at rates of 1-2 ˚C/min. The increasing temperature tests were conducted to show how the LCM mixtures would thermally degrade while constantly agitated. The third test was similar to the second type, except that the samples were pressurized to 5 MPa (725 psi) and while shearing at 100 rpm the temperatures were then increased to 200 ˚C. The thermal degradation tests were only conducted once with each LCM mixture, so different samples were used for the 90 and 200 ˚C degradation tests.

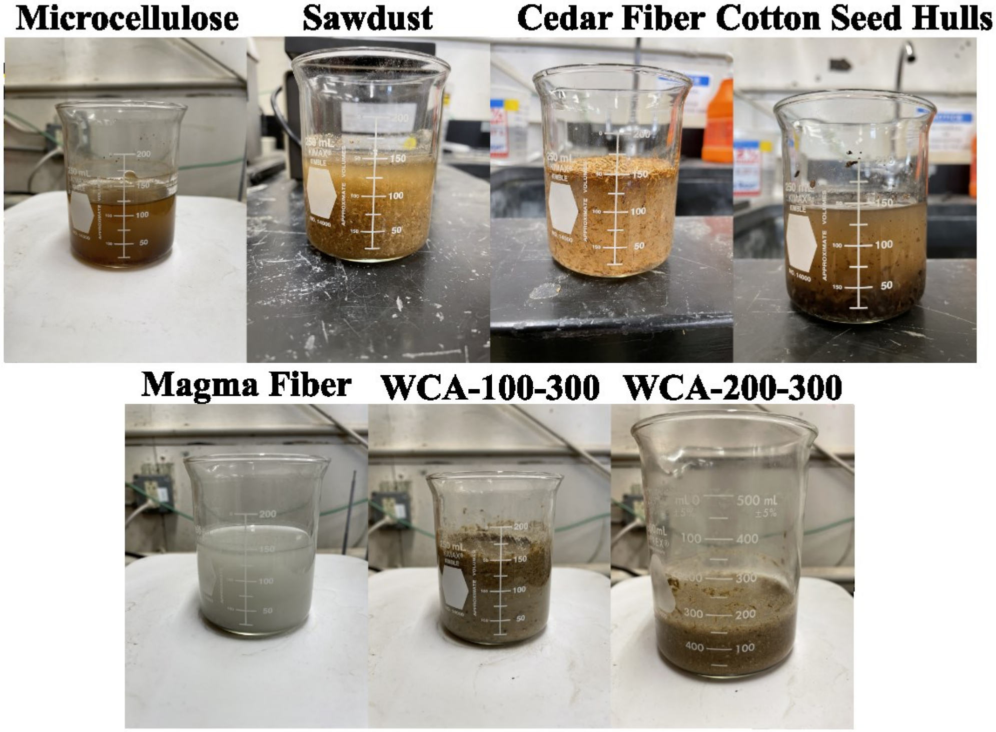


Figure 4: Examples of mixtures prior to testing, with the microcellulose, sawdust, cedar fiber, cotton seed hulls, and magma fiber being mixed with xanthan gum. The WCA mixtures were only mixed with the two components and water.

3 EXPERIMENTAL RESULTS

3.1 Degradation tests

The mass of the initial LCM in each mixture varied initially from 5 to 50%. Figure 5 shows how the fraction of mass remaining for each LCM when heated to 90, 120, 200, and 250 ˚C varies with the duration of heating. At 90 ˚C, very little mass loss is observed no matter the duration of heating. At 120 ˚C, above the normal boiling temperature for water, much greater mass loss is observed in all the materials than at 90 ˚C. This trend continues for the tests at 200 and 250 ˚C, where the most obvious transition in the material appearances occurs (Figure 6). The most significant mass loss is observed in the first 24 hours, as LCM mass loss only increases by 5-30% with greater durations of heating (Figure 5).

On the basis of mass loss, magma fiber exhibits the least degradation due to temperature and time heated. Only at 250 ˚C is significant mass lost observed in the magma fiber mixtures, and even then it is much less than any of the other materials. Most of the materials only exhibit ~10% mass loss at most when heated to 90 ˚C. At 120 and 200 ˚C, the most significant mass loss occurs for the microcellulose, sawdust and cotton seed hulls, between 20-50% after 1 day of heating. The WCA mixtures exhibit fairly similar trends in mass loss, more than the magma fiber but less than microcellulose, sawdust and cotton seed hull mixtures. Cedar fiber seems to degrade less than any of the materials except the magma fiber up to 200 ˚C. Once each material is heated to 250 ˚C, the materials tested all exhibit at least 50% mass loss after 1 day of heating, with the exception of the magma fiber. Much less variance in the mass loss is observed at this temperature than compared to the lower conditions.

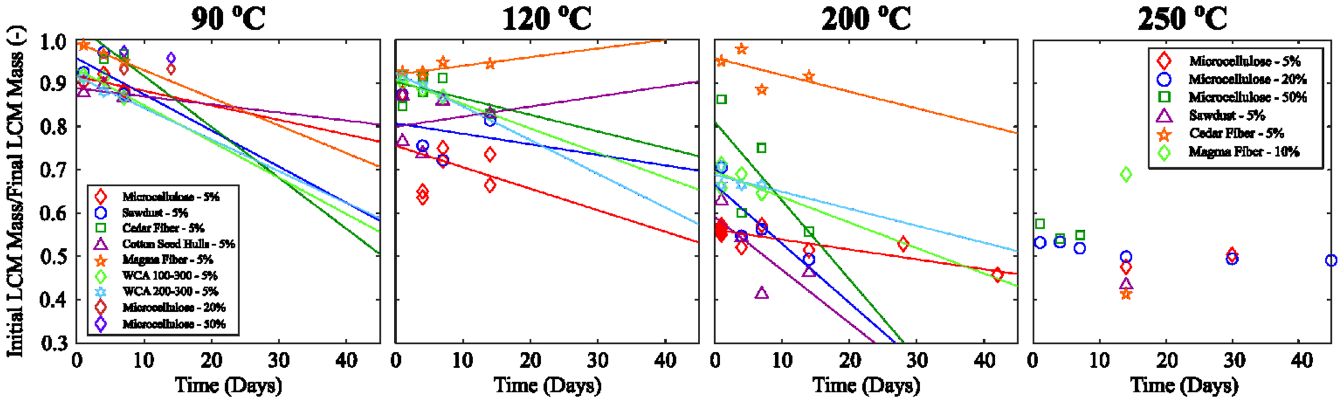


Figure 5: Mass fraction of each LCM remaining after thermal degradation plotted against heating duration. The legend for 90 ˚C applies to both the 120 and 200 ˚C, while the legend for 250 ˚C is different. The weight % of the LCM in each mixture is listed in the legends, varying from 5 to 50%.

Figure 6 shows how the LCM mixtures change after 7 days when heated to various temperatures. None of the materials show significant alteration at 90 ˚C, save perhaps the sawdust and cedar fiber which are biodegradable materials. At 120 ˚C, the materials begin to darken, and in the case of microcellulose, form a mesh-like material at the surface of the mixture. At 200 ˚C, the LCM mixtures show the largest qualitative change. All LCM save for the magma fiber become completely blackened and brittle, and the materials can be easily disintegrated. The sawdust, cedar fiber and especially the microcellulose form a blackened mass that coalesces at the top of the water, though the cedar fiber shows only slight example of this change. The cotton seed hulls completely lose cohesion as a fibrous materials, leaving only blackened particles. The WCA mixtures both become a hardened material that adheres to the container, similar to a dried mud layer, with very little fluid remaining. The magma fiber shows by far the least variation as only a small browning of the LCM can be observed after more than 7 days of heating at 200 ˚C.

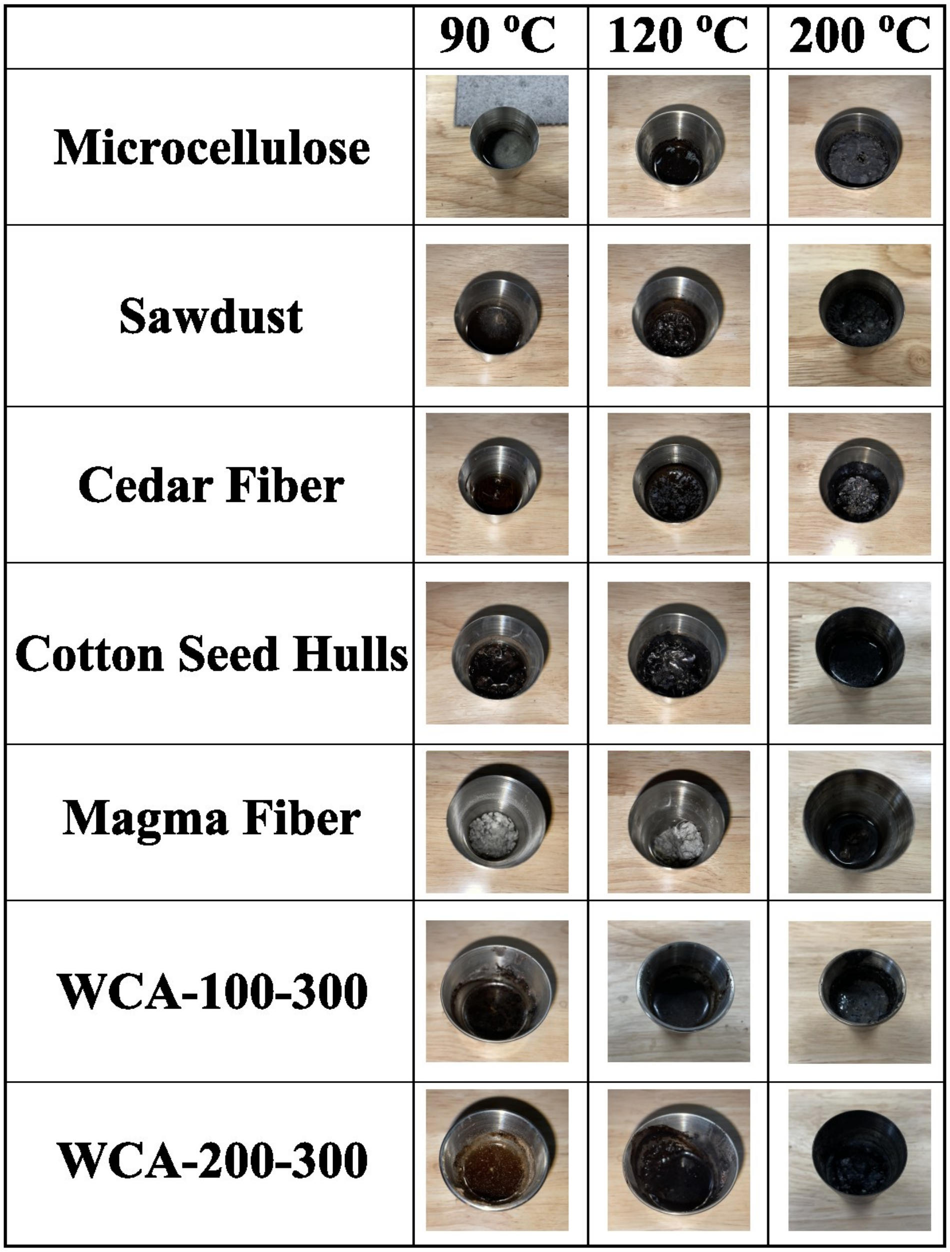


Figure 6: Images of degraded LCM mixtures after 7 days of heating at 90, 120, and 200 ˚C.

3.2 Viscosity tests

Viscosities of the LCM mixtures at room temperature and varying shear rates are shown in Figure 7. For the xanthan gum mixtures, the viscosities approach 2-10 mPa*s, while the bentonite mixtures approach 20-30 mPa*s with increasing shear rate. Interestingly, the WCA-100/200/300 mixtures (Figure 7a) have significantly higher viscosities than any of the normal LCM materials. Generally, the LCM mixtures tend to cluster around the values observed in the mixtures with only water and xanthan gum/bentonite.

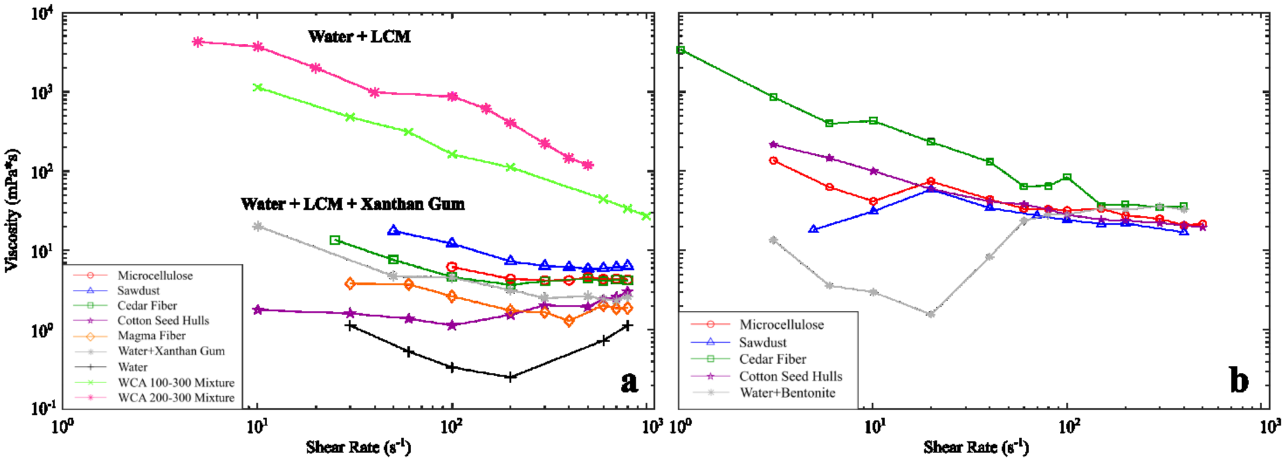


Figure 7: Viscosity of mixtures at room temperature with increasing shear rate: a) Xanthan gum-LCM mixtures and WCA-100/200/300 mixtures; b) bentonite-LCM mixtures.

The thermal degradation of the mixtures is shown in Figure 8. Temperature was increased from 25 to 90 ˚C at ambient pressure, while the mixtures were sheared at a constant rate of 170 s^{-1} (or 100 rpm). Generally, the viscosity of the xanthan gum and bentonite mixtures tended to decrease with continued heating and shearing. The viscosities of the bentonite mixtures were generally higher, but interestingly enough when temperature approached 90 ˚C some of the mixtures exhibited a slight increase in viscosity. The WCA-100/200/300 mixtures are by far the most unaffected by the increase in temperatures; though WCA-200-300 mixtures have higher viscosities (see also Figure 7), both exhibit near constant viscosities clustered around 90 and 110 mPa*s for WCA-100-200 and WCA-200-300, respectively.

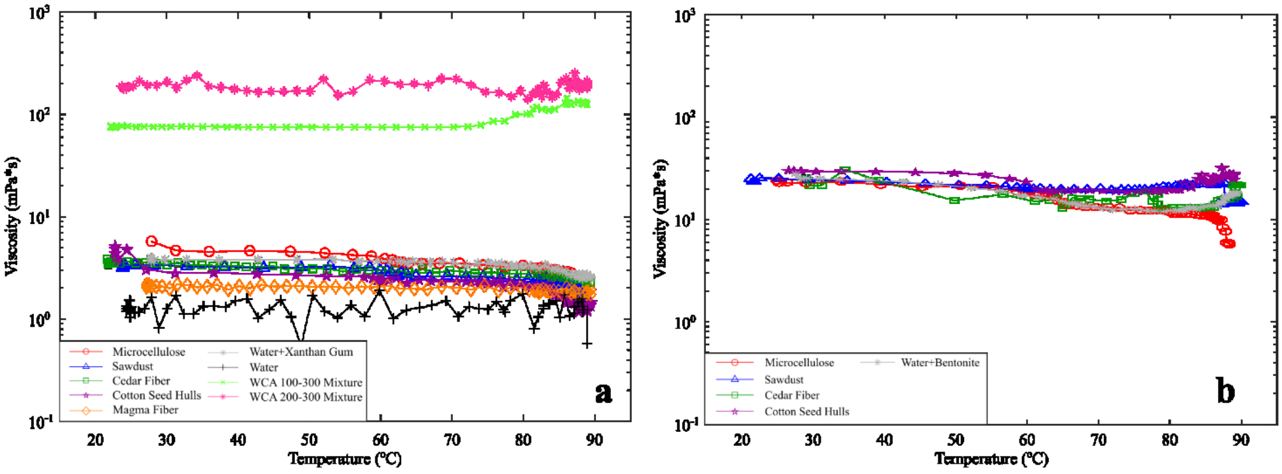


Figure 8: Degradation of viscosity with increasing temperature at a constant shear rate (170 s^{-1}): a) Xanthan gum-LCM mixtures and WCA-100/200/300 mixtures; b) bentonite-LCM mixtures.

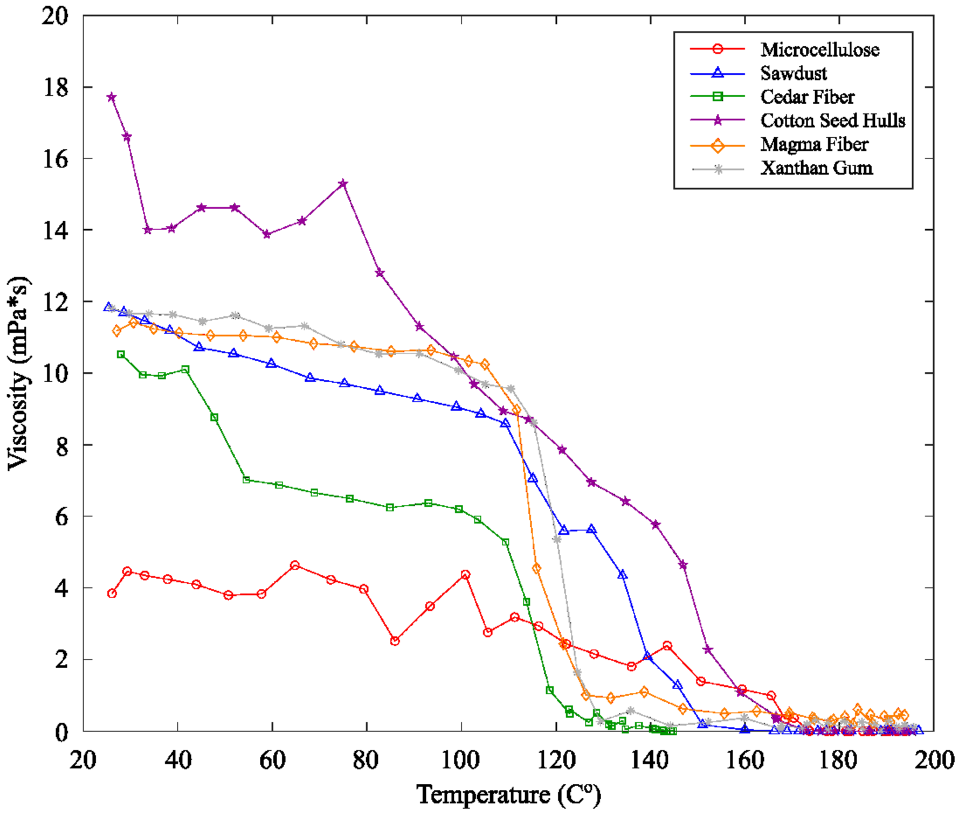


Figure 9: Degradation of viscosity with increasing temperature at a constant shear rate (170 s^{-1}) for xanthan gum mixtures at 5 MPa confining pressure.

Figure 9 shows how the viscosity degrades when temperature is increased to 200 ˚C under constant shearing. Initially all of the materials exhibit slightly decreasing viscosity values as temperature is increased. However, between 110-160 ˚C, the viscosity of all the xanthan gum mixtures drops precipitously to near 0, remaining low until reaching 200 ˚C.

4 ANALYSIS AND DISCUSSION

4.1 Temporal and thermal degradation of lost circulation materials

The degradation of each LCM is quantitatively and qualitatively different, as shown in Figure 5 and 6. However, there are general trends that can be observed. First, the largest loss in LCM mass occurs very quickly during heating, within the first 24 hours. The reduction in mass appears to be exponential with time for the materials, with the possible exception of the magma fiber which may degrade linearly with time. Second, there is a clear transition in material degradation between 90 and 120 ˚C as well as between 200 and 250 ˚C (Figure 5,6). Above 90 ˚C different LCM start to change character and lose their undegraded characteristics, and duration of heating has more of an effect on the heated LCM. Above 200 ˚C, the different LCM degrade in a similar manner (except for magma fiber), suggesting that the temperature is sufficient to eliminate much of the variations between LCM that produce different particulates when heated at lower temperatures. Third, the LCM mixtures will become less viscous when continuously sheared and/or heated (except for the WCA mixtures) (Figure 8).

Based on the degradation tests, magma fiber is by far the least degraded by temperature while microcellulose, sawdust and cotton seed hulls degrade the most. The WCA and cedar fiber mixtures are between these two extremes in terms of mass loss (Figure 5). The effect of time on degradation is not as easily shown, but the major material change to each LCM occurs very quickly and that further heating does not lead to major alteration.

The viscosity tests with each LCM indicate that the microcellulose, sawdust, cedar fiber, cotton seed hulls, and magma fiber do not significantly affect the observed viscosity (Figure 7,8). Rather the viscosities themselves are due to the additive (i.e., xanthan gum and bentonite) properties and weight % in each mixture. Indeed, this is why in Figure 9 all the viscosities degrade rapidly between 110 and 160 ˚C despite the different LCM added to each. Xanthan gum, when mixed with water, forms a polymer (Figure 4) that can suspend the particles. However, above 90 ˚C the polymer breaks down and the mixture degrades quickly such that the viscosity of the mixture diminishes to near 0 (Vivas and Salehi, 2021). The exception to this is the WCA mixtures, which have much higher viscosities that do not degrade when heated to 90 ˚C. The gellant (WCA-100 or -200) appears to be the main control of the viscosity, rather than the additive WCA-300, which is composed of a mixture of particulates including microcellulose, shredded paper, and wood chips (Figure 4). Interestingly, the gellant material added also seems to have a chemical role. Magma fiber may break down little due to heating, but when mixed at room temperature with bentonite the particles appear to dissolve almost completely save some thin fibers scattered throughout the mixture. Thus, it must be understood that not only the in-situ conditions important for degradation, but also the chemical interactions that each LCM is expected to experience both due to the drilling fluids and the subsurface fluids.

4.2 Identifying ideal LCM characteristics

The characteristics of an ideal LCM are dependent upon the specific locality where lost circulation treatment is needed. Different lost circulation zones have different characteristics, such as the type of opening fluids are escaping through, the average aperture of the fractures, and the in-situ conditions at the zone. This is why there exists such a swathe of materials used for treatment, as no single material will fit all circumstances, and why many companies often mix multiple LCM types together to combine particulate properties when sealing lost circulation zones (Alsaba et al., 2014).

Based on our experiments, the particulates used do not significantly alter the viscosity of the LCM mixtures. However, they do degrade differently. If a permanent seal is desired, magma fiber is the ideal material as it will degrade the least due to temperature. Because it is an acid-soluble material, careful consideration of the chemical interactions must be done before employing its use. However, it is possible that a material that is slightly degradable may be desired as well, as the particulates will be able to fit into loss circulation zone fractures or openings more easily if they are slightly degraded by the temperature conditions. Cedar fiber would thus be ideal as it only experiences a small degree of degradation compared to other materials, though its elongated shape may make it difficult to seal thinner fractures. Fibrous materials, like magma or cedar fiber, tend to form a “mat-like” bridge in loss zones that allows smaller particles to escape through while constraining larger particulates (Nayberg, 1987). This would then make them ideal for many different loss zones when mixed with smaller particulates such as microcellulose or sawdust, which could seal fractures and pack the gaps where the fibrous materials are unable to seal. Additionally, microcellulose and sawdust appear to form a rigid mesh structure when they are thermally degraded at high temperatures and might thus act as good to seal the gaps left by larger LCM (Figure 6). An ideal characteristic then would be a mixture of not only LCM type (i.e., granular, flaky, fibrous) and size, but also of degradation degree, as this would allow for long term modulation of lost circulation zones rather than permanent seals. For treating lost circulation zones within the geothermal reservoir, it is desirable to employ LCM that will temporarily halt the loss of drilling fluid when permeable fractures are encountered, but will thermally degrade with time, allowing these fractures to regain their permeability (either as feed zones in a production well or outflow features in an injection well).

5 CONCLUSIONS

The thermal degradation of several different materials used in lost circulation treatments was studied using heating and viscosity testing. The organic LCM tend to degrade the most at all temperatures (ex., 30-60% mass loss at 200 ˚C), while the magma fiber tends to degrade the least (ex. 10-15% mass loss at 200 ˚C). In terms of viscosity, the LCM do not significantly affect the viscosity of the fluids, which depend primarily on what additives are used in each mixture to suspend the particulates. Constant shearing up to 90 and 200 ˚C of the LCM mixtures indicates that, except for the WCA mixtures which remain constant, the viscosity of the mixtures will decrease with both continuous shearing/agitation as well as temperature. The different patterns of degradation among the LCM types suggest that: 1) LCM should be selected not only based on shape and size, but also by how much they degrade at a given condition; 2) the individual LCM will not significantly affect the viscosity of the mixture unless a large enough concentration is used; 3) LCM selected for use within the reservoir interval of a well should degrade either thermally or chemically so that the plugged fractures can recover their permeability over time. The use of two or more LCM types may also be able to take advantage of the different degradation behaviors in sealing lost circulation zones.

ACKNOWLEDGEMENTS

The authors would like to thank John Tuttle and Ron Tait of Sinclair Well Productions for providing the microcellulose, sawdust, cedar fiber, cotton seed hulls, and magma fiber utilized in this study. The authors would also like to thank Lyndon Chandarjit and Henry Lopez at North Star Energy Products for providing the WCA-100/200/300 materials utilized in this study. We would also like to thank Mathew Ingraham for conducting an internal review of this manuscript for Sandia National Laboratories.

This work was supported by the U.S. Department of Energy, Office of Energy Efficiency and Renewable Energy (EERE), Geothermal Technologies Office (GTO), under Award Number DE-AC02-05CH11231 with LBNL and contract DE-NA-0003525 with Sandia National Laboratories. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy’s National Nuclear Security Administration under contract DE-NA-0003525. This manuscript describes objective technical results and analysis. Any subjective views or opinions that might be expressed in the paper do not necessarily represent the views of the U.S. Department of Energy or the United States Government. SAND2023-11309A

REFERENCES

Alsaba, M., Nygaard, R., Hareland, G., and Contreras, O.: Review of lost circulation materials and treatments with an updated classification. *AADE National Technical Conference and Exhibition, Houston, TX* (2014, April).

Caenn, R., Darley, H.C., and Gray, G.R.: Composition and properties of drilling and completion fluids. Gulf professional publishing, (2011).

Lowry, T., Winn, C., Dobson, P., Samuel, A., Kneafsey, T., Bauer, S., and Ulrich, C.: Examining the Monetary and Time Costs of Lost Circulation. (2022).

Mohamed, A., Salehi, S., Ahmed, R., and Li, G.: Experimental study on rheological and settling properties of shape memory polymer for fracture sealing in geothermal formations. *Journal of Petroleum Science and Engineering*, *208*, (2020), 109535.

Nayberg, T.M.: Laboratory study of lost circulation materials for use in both oil-based and water-based drilling muds. *SPE Drilling Engineering*, *2*(03), (1987), 229-236.

Saleh, F., Teodoriu, C., Salehi, S., and Ezeakacha, C.: Geothermal Drilling: A Review of Drilling Challenges with Mud Design and Lost Circulation Problem. *Proceedings of 45th Annual Stanford Geothermal Workshop, Stanford University, Stanford, CA*, (2020).

Vivas, C., and Salehi, S.: Rheological investigation of effect of high temperature on geothermal drilling fluids additives and lost circulation materials. *Geothermics*, *96*, (2021), 102219.

Winn, C., Dobson, P., Ulrich, C., Kneafsey, T., Lowry, T. S., Akerley, J., Delwiche, B., Samuel, A., and Bauer, S.: Context and mitigation of lost circulation during geothermal drilling in diverse geologic settings. *Geothermics*, 108, (2023), 102630.