

Acid Soluble, Expandable Loss Circulation Material

Ashok Santra, Arthur Hale, Kenneth Johnson and Nicolas Osorio

Aramco Services Company: Houston Research Center, USA

Copyright 2023, AADE

This paper was prepared for presentation at the 2023 AADE National Technical Conference and Exhibition held at the Bush Convention Center, Midland, Texas, April 4-5, 2023. This conference is sponsored by the American Association of Drilling Engineers. The information presented in this paper does not reflect any position, claim or endorsement made or implied by the American Association of Drilling Engineers, their officers or members. Questions concerning the content of this paper should be directed to the individual(s) listed as author(s) of this work.

Abstract

Loss of circulation during drilling or cementing operations is a serious problem that is responsible for non-productive time (NPT) during drilling and cementing. This work will focus on a new technology to remediate severe lost circulation. We have developed two different Lost circulation material (LCM) setttable fluid systems that can cover downhole applications up to temperatures of 300°F. The setttable LCM fluid generates nitrogen gas in-situ above 120F resulting in expansion and filling the natural and/or induced fractures. The fast-setting nature of the setttable pill develops hard seal within a few hours after placement.

Laboratory testing confirms the different level of expansion at different temperatures and pressures conditions wherein the rate of gas generation is faster at higher temperature. The higher the confining pressure the lower the expansion, whereas the temperature favors the expansion. It has also been confirmed that the hardened LCM fluid is 100% acid soluble in 15% HCl. To further confirm if the setttable pill can seal a sandstone fracture, an experiment was conducted where a 3.55 mm simulated fracture confined under 3000 psi was plugged after placement of the LCM fluid across the simulated fracture. The setttable LCM fluid entered the fracture and after several hours for the fluid to harden, the fracture was sealed with enough structural integrity to withstand several hundred psi differential pressure.

The expandable nature of this acid soluble fast setting fluid pill systems is novel and unique being able to expand even under down-hole pressure conditions, to effectively cure major losses at application temperatures up to 300F.

Introduction

Fluid losses into the formation during drilling and/or cementing operations caused by high permeable formations, induced or natural fractures is a challenging and costly affair for the operators due to (1) loss of expensive fluids, (2) large amounts of non-productive time (NPT) and (3) inadequate zonal isolation needing future remedial work. Estimated cost associated with loss circulation events can be multi billion dollars annually in NPT, additives and other drilling operation related expenses (Alshubbar et al., 2018). Equivalent

circulating density (ECD) of the fluids exceeding the fracture gradient of formations causes induced fracture, whereas, natural fractures are to either unconsolidated or cavernous formations. Particulate based Loss Circulation Materials (LCM) has been used for decades that forms mechanical plug to cure the losses, wherein the plugging efficiency is dependent on the size, shape, distribution and mechanical properties of the materials used (Whitfill et al., 2006; Chellappah et al. 2018, Ramirez, et al., 2009 and Mehrabian et al., 2017). Severity of fluid loss has been categorized as a function of loss rate as shown in Table 1 below (Nayberg and Petty 1986).

Table 1: Loss type vs loss rate

Type of Loss	Loss Rate (bbl/hr)	Traditional Remediation
Seepage	1 to 10	Particulate based LCM (conventional)
Partial	10 to 100	Particulate based LCM (conventional)
Severe	>100 but some return	Specially designed
Total	>500 to no return	Specially designed or none

Some of the very commonly used particulate based LCMs used in the industry are shown in Figure 1, whereas during a particular application combination of more than one LCMs and various grades are used to obtain most effective plugging. Usually various types of waste and inexpensive materials are used for the applications.

Laboratory optimization along with analytical modelling do a fairly good job (Chellappah et al., 2018 and Mehrabian et al., 2017) prescribing an effective solution for seepage and partial loss scenarios. However, when it comes to curing severe losses none of these approaches work effectively, because of the size, shape and tortuosity of the openings in the formation. Therefore, when the fracture sizes (either induced or natural) are so large, to achieve minimum flow resistance the volumes and concentrations necessary of LCMs that can be pumped may

be relatively too small is size to effectively seal the fractures. To overcome such limitations, we have developed setttable fluid that can generate nitrogen gas in-situ to provide effective volume expansion depending upon downhole pressure. In addition the expandable and setttable fluid can be used in combination with optimized blend of LCMs (Figure 1) for curing severe losses.

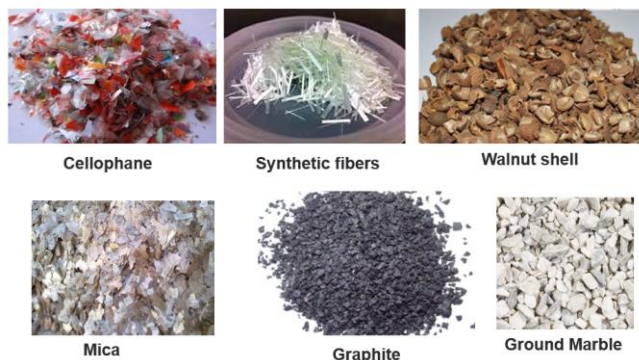


Figure 1: Typical LCM materials used in the industry

Materials

Sorel Cement (Sorel 1967) based setttable fluids with enhanced acid solubility have been developed by using materials like CaCO₃ (Santra et al. 2009, Seymour et al. 2013). Main ingredients for such magnesium-oxy-chloride based setttable fluids are MgO, MgCl₂.6H₂O and water. Type of MgO can be either light burned, hard burned or dead burned to control the reaction rate as function of temperature. Set cement forms the following crystal phases based on the application temperatures (Chau et al., 2008 and Cole et al., 1955):

- (a) Temperature below 100°C (212F):
 $Mg(OH)_2 + 3Mg(OH)_2.MgCl_2.8H_2O$ (3-phase) +
 $5Mg(OH)_2.MgCl_2.8H_2O$ (5-phase)
- (b) Temperature above 100°C (212F):
 $2Mg(OH)_2.MgCl_2.4H_2O$ (2-phase) and/or
 $9Mg(OH)_2.MgCl_2.5H_2O$ (9-phase)

Crystal phases were characterized by X-ray Diffraction (XRD) analysis.

Xanthan gum has been used as a viscosifier or suspending aid. Organic nitrogen gas generating material has been used to produce N₂ gas in-situ. A typical cement foamer has been used to stabilize thus generated nitrogen gas in the fluid. Inorganic phosphate or borate can be used to control the pump time of the fluid pill.

Experimental and Application Procedures

STEP 1: Adjustment of Thickening Time using Slurry A:

It is important to make sure that the amount and type of retarder is properly optimized to ensure that the fluid has adequate pump time by running several Thickening Time experiment using HTHP consistometer and following API RP 10B guidelines. Consequence of a shorter than required pump time will result in premature setting of the fluid during pumping whereas an longer than required pump time will cause unseetable LCM pill, both these scenarios will result unsuccessful treatment. Typical slurry designs and retarder sensitivity is shown below in Table 2 and 3:

Table 2: Typical slurry-A design for adjusting Thickening Time test

Materials	Weight
Water	423gm
Retarder	12gm
MgCl ₂ , 6H ₂ O	250gm
Xanthan gum (viscosifier)	4.5gm
MgO	300gm

Table 3: Thickening Time test parameters and results different BHCT

BHCT	BHP	Ramp time	Thickening Time	Fluid time at BHCT
110°C (230F)	8000psi	80min	2:24	54min
126.7°C (260F)	8000psi	60min	1:36	36 min
149°C (300F)	8000psi	60min	1:13	13 min

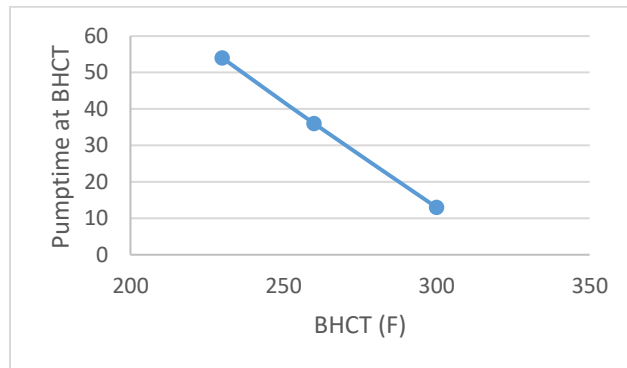


Figure 2: Temperature vs Thickening Time at different bottomhole circulating temperature (BHCT)

As shown in Table 3 and Figure 2, Thickening Time can be optimized by adjusting the amount of retarder up to a maximum of 10% by weight of MgO. It is noteworthy to mention here that the retarder response of the settable fluid developed here is extremely linear unlike regular Portland cement based fluids. Figure 3 demonstrating the linear response of the retarder.

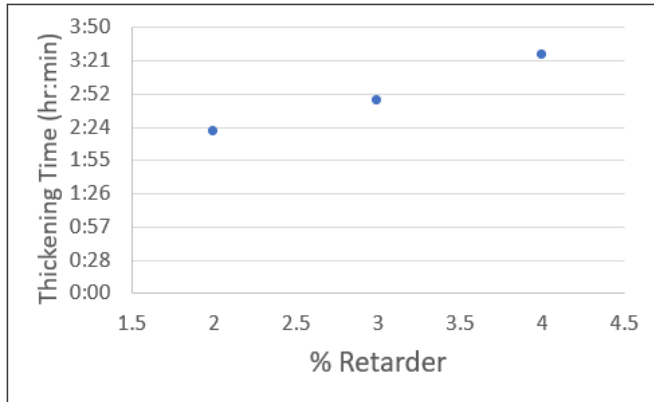


Figure 3: % Retarder vs Thickening Time at bottomhole circulating temperature (BHCT) 200 °F, 3000psi

Slurry A mixing procedure:

- Add 423 gm water in a wearing blender
- add 6gm retarder and mix for 5min at 2000RPM, make sure it is fully dissolved
- add 250gm MgCl₂ and mix for 5min at 2000RPM, make sure it is fully dissolved
- add 4.5gm xanthan gum and mix for 5 min at 2000RPM
- add 300gm MgO and mix 2min at 2000RPM

STEP 2: Storable slurry B preparation procedure:

Once the amount of retarder has been determined from STEP 1, a storable slurry B (as seen in Figure 4) can be prepared with the following typical composition ratio and mixing procedure:

- add 600 gm water
- 16.8gm retarder and mix for 2min at 2000RPM
- add 357gm MgCl₂.6H₂O and mix for 10min at 2000RPM
- add 7gm xanthan gum and mix for 10 min at 2000RPM
- add 17gm nitrogen generating additive and mix for 10min at 2000RPM
- measure pH with a meter, make sure that the pH is close or lower than 3.5

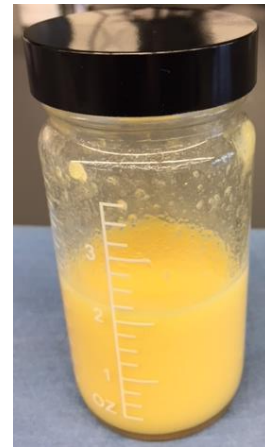


Figure 4: Typical color of Slurry B

STEP 3: Confirmation of in-situ volume expansion:

Volume expansion test is carried out after mixing required amount of MgO and foamer with Slurry B. First, take 15ml Slurry B in a graduated tube (as shown in Figure 5), add 6.4gm MgO and shake to mix well for about 60 sec, then the foamer is added and gently blended with a spatula to avoid immediate foaming with air. Thus mixed samples are capped and then placed in a preheated water bath. Volume expansion as a function of real times are noted.

Table 4: Formulation for expansion test

	48.9 °C (120F), water bath	60 °C (140F), water bath
Slurry - B	15ml	15ml
MgO	6.4gm	6.4gm
Foamer	0.3ml	0.3ml

Figure 5 below demonstrate the time dependent volume changes at two different temperatures. A control sample was prepared without adding any nitrogen gas generating additive.

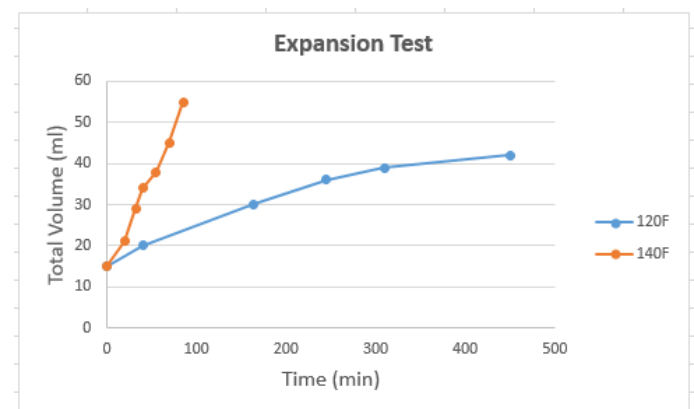


Figure 5: Total volume plots as function to real time

AADE-23-NTCE-049

As seen from Figure 5, the rate of expansion is highly temperature dependent and increases with the increase in temperature. A close to 300% final volume expansion was observed, however, actual downhole volume expansion will vary the following way:

- Directly proportional to the amount of gas generating agent used
- Directly proportional to the downhole application temperature
- Inversely proportional to the downhole application pressure

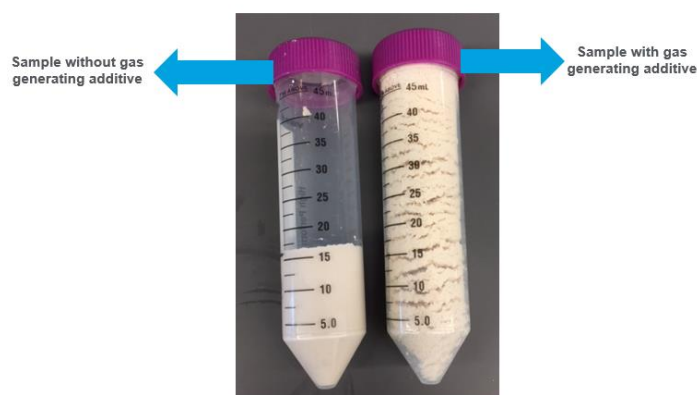


Figure 6: Picture of an unexpanded vs expanded set sample after 16hrs at 60° C (140F), water bath

Field Application Procedure

Slurry B can be premixed and stored for 2-3 days and brought to the location for use. When needed required amount of MgO can be blended and pumped immediately to minimize preparation time.

Compressive strength development:

Ultrasonic cement analyzer test result demonstrated the fast-setting nature of the LCM pill, a ~90% of total compressive strength was developed within 5 hours of the test at 120 °F, 1000psi.

Acid solubility:

Acid solubility of the set cement with or without using gas generating additive was carried out with 1cm x 1cm x 1cm set cement cube hanging in with a plastic thread in a beaker with 1 liter 15% HCl. Sample was periodically take out from the beaker, washed in tap water, dried and then weight was taken. Under ambient conditions it took about 80 min to dissolve the cube completely without any residue left. The rate of dissolution will depend upon the wellbore temperature (faster

at higher temperature) and the acid contact surface area. Higher porosity/permeability due to foaming will help the diffusion of acid phase in to the set cement which is the rate limiting step in this reaction kinetics.

Fracture sealing test:

We conducted simulated fracture sealing test using sandstone rock cores were conducted at 120° F and 1000psi. Fracture width of 3.55mm was used for the test, as shown in **Figure 7**. Expandable LCM fluid pill was placed in the fracture and the remaining fluid was displaced with higher density brine from the borehole. Fluid placed in the fracture was cures at 120° F, 1000psi for 16 hours before running fracture sealing test as function of differential pressure. Results demonstrate that a sealing capability of over 900psi differential pressure was observed (**Figure 8**), this is significant.

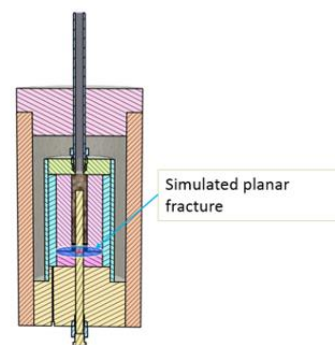


Figure 7: Simulated fracture sealing setup

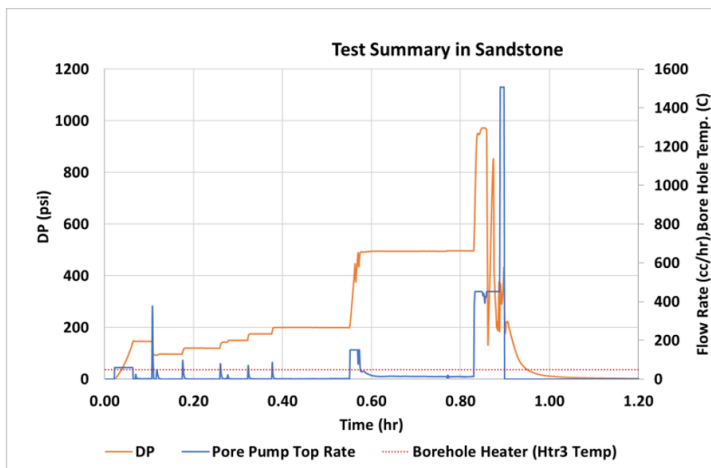


Figure 8: Fracture sealing efficacy test

Conclusions:

- Sorel cement based settable, acid soluble LCM fluid pill developed in this work is a viable technology for curing severe losses at bottomhole temperature up to 149° C (300° F).
- Desired pump time can be obtained by adjusting the type and amount of retarder used in the pill design.
- Pill developed adequate compressive strength wherein ~90% of the strength was developed within 5 hours of the placement, confirming the fast setting nature of the pill compared to regular Portland cement.
- Set mass is completely soluble in 15% HCl.
- Simulated fracture sealing capability of over 900psi differential pressure was observed under simulated condition.

References

- Alshubbar, G., Nygaard, R., Jeennakorn, M. 2018. The effect of wellbore circulation on building an LCM bridge at the fracture aperture. *J. Petrol. Sci. Eng.* **165**: 550–556. <https://doi.org/10.1016/j.petrol.2018.02.034>.
- Chau, C. K., Li, Z. 2008. Microstructures of magnesium oxychloride Sorel cement. *Adv. Cement Res.* **20**: 85-92. <https://doi.org/10.1680/adcr.2008.20.2.85>.
- Chellappah, K., Majidi, R., Aston, M. Cook, J. 2018. A practical model for wellbore strengthening. Presented at IADC/SPE Drilling Conference and Exhibition, Fort Worth, Texas, USA, 6-8 March. SPE-189589-MS. <https://doi.org/10.2118/189589-MS>.
- [Cole, W. F., Demediuk, T. 1995. X-Ray, Thermal, and Dehydration Studies on Magnesium-Oxychlorides. Austral.](#)

[J. Chem. 8: 234-251.](#)

Mehrabian, A., Savari, S., Whitfill, D. Abousleiman, Y., 2017. Geomechanics of wellbore strengthening revisited: a combined theoretical and experimental approach with field case studies. Presented at SPE/IADC Drilling Conference and Exhibition, The Hague, The Netherlands, 14-16 March. SPE-184609-MS. <https://doi.org/10.2118/184609-MS>.

Nayberg, T. M., Petty, B. R., 1986. Laboratory Study of Lost Circulation Materials for Use in Oil-Base Drilling Muds. Presented at Deep Drilling and Production Symposium, Amarillo, Texas, USA, April 6-8. SPE-14995-MS. <https://doi.org/10.2118/14995-MS>.

Repetto, C., Moroni, N., Pilia, L., Santra A. Successful Application of Acid Soluble Plugs in Open Hole Slotted Liner Completion, presented at the SPE Oil and gas conference and exhibition held in Mubai, India, 20-22 January 2010. SPE 129159-MS. <https://doi.org/10.2118/129159-MS>

Ramirez, H., Santra A., Martinez, C. Ramos, X., Gas-Migration Control and Mechanical Properties Improvement with a Right Angle-Set Slurry Design to Be Applied In a Production Cementing-Job for Ecuador presented at Latin American and Caribbean Petroleum Engineering Conference held in Cartagena de Indias, Colombia, 31 May-3 June, 2009, SPE 123085-MS, <https://doi.org/10.2118/123085-MS>

[Santra, A., Liang, F., Fitzgerald, R. 2009. Sorel cement for HPHT downhole applications. Presented at SPE International Symposium on Oilfield Chemistry, The Woodlands, Texas, USA, 20-22 April. SPE-121102-MS. <https://doi.org/10.2118/121102-MS>.](#)

Seymour, B., Santra, A. 2013. Detailed laboratory investigation of acid soluble cements as solution for lost circulation across the producing zones. Presented at SPE/IADC Middle East Drilling Technology Conference & Exhibition, Dubai, UAE, 7-9 October. SPE-166804-MS. <https://doi.org/10.2118/166804-MS>.

[Sorel, S.1867. On a new magnesium cement, Comptes Rendus-Academie des Scances. Hebdomadaires, 65: 102-104.](#)

Whitfill, D., Thaemlitz, C., Jamison, D., Wang, H. 2006. [New Design Models and Materials Provide Engineered Solutions to Lost Circulation. Presented at SPE Russian Oil and Gas Technical Conference and Exhibition, Moscow, Russia, 3-6 October. SPE-101693-MS. <https://doi.org/10.2118/101693-MS>.](#)