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# Developing and Testing Lost Circulation Materials

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## Abstract

The development and testing of lost circulation materials in a laboratory is hampered by the scale of the tests that are possible, versus the scale of the application in the field. If actual fractures are initiated and propagated, as done in Drilling Engineering Association Project 13 in the mid-1980s, the 30-in x 30-in test blocks and equipment are on a "pilot scale" which is both expensive and difficult to manage.[1,2] If scaled down to 4-in diameter core, as was done in GPRI Project 2000 "Mitigating Lost Circulation in Synthetic-Base Fluids", the testing becomes more manageable, but still very time consuming and expensive.[3]

By combining a particle size design modeling capability with standardized lab screening tests, Halliburton has been able to design new lost circulation materials that have proven successful in the field. These standard test procedures still do not solve the "scaling" problem, but are capable of doing a good job in screening the best candidates for field evaluation. The design model and lab results will be reviewed for two different development projects, along with field application results from one of the systems.

## Background

Engineered solutions designed to improve wellbore strength and reduce drilling non-productive time due to lost circulation are directed at managing wellbore stresses (WSM). This fully engineered approach should include means to help prevent lost circulation as well as stop losses. Prevention of lost circulation by improving the wellbore strength can be accomplished by designing and applying borehole stress treatments that increase the hoop stress around the wellbore.[4,5]

Proprietary hydraulic design software (HDS) can predict the equivalent circulating density (ECD) over an interval in one module, calculate the width of a fracture that may be initiated, and select and design a proper material and particle size distribution that can efficiently prop and plug that fracture in a second module.

Specialized combinations of materials can be quickly applied to a lost circulation incident since they are "one-sack" systems that can be maintained already mixed, or mixed and applied with the rig pumps through downhole tools and the bit.

Contingency chemical sealant (CS) treatment applications are systems designed to react with the drilling fluid itself to create highly viscous and cohesive sealants in the wellbore that are then displaced into the lost circulation fractures. Drilling-fluid-reactive systems are not dependent on temperature or pressure, thus removing a significant amount of placement uncertainty present with cross-linked systems.

This combination of planning and application tools allows the operator to make decisions ahead of time during the "drilling the well on paper" phase as to which approach is the most economic: prevention or remediation.

## Overview

Lost circulation is one of the biggest contributors to drilling non-productive time (NPT), and it is the most difficult drilling segment about which to make economic decisions. Estimations of economic impact in this segment vary widely, but it is safe to say that it represents a very large portion of the total non-productive expense for drilling a well. As rig rates increase, the economic impact of NPT increases as well. Therefore, any technology that reduces drilling NPT can translate into millions of dollars in reduced drilling costs.

To address this problem, engineered solutions designed to improve wellbore strength and reduce drilling non-productive time caused by lost circulation were developed. This wellbore stress management (WSM) service provides a fully engineered approach to lost circulation problems that incorporates both unique planning software and materials.

Lost circulation planning includes both prevention and remediation methods. While it is critical that losses be stopped once they occur, it is equally important that they be prevented because problems prevented represent money never expended. One important part of the preventive plan is the design of "borehole stress treatments". The goal of these treatments is to increase the "hoop stress" in the near-wellbore region to improve the wellbore pressure containment ability.

## Design Model

The recent addition of a new module to Baroid's HDS is capable of determining the particle size distribution of any combination of individual materials that are in the data base. Based on pore size, or estimated fracture widths, the model can select the proper types and sizes of materials to plug the pores and/or an initiated fracture (**Figure 1**). For borehole stress treatments, these materials generally are selected from a full range of specialized resilient graphitic carbon and ground marble products (**Table 1**), with d50s ranging between 5 and

1200 microns.[5]

An example model solution output is shown in **Figure 2**. The d10, d50 and d90 of the solution is given, along with a composite curve showing the particle size distribution (PSD) of the mixture of materials as well as the PSD curves for the individual components. In addition, a cumulative curve is shown from which we can determine the volume of materials in the mixture that lies below that micron size by simply placing a cursor at any point along the curve.

A number of engineering scenarios can be evaluated during the planning phase for implementation during the well construction phase. These may include a pretreatment of the entire system to manage seepage and wellbore breathing issues; a sweep treatment using larger particles for potential fracture initiation in problem zones; and a treating pill to be placed across the problem interval for a borehole stress treatment and/or prior to running casing and cementing.[6] Also shown in these examples is the consideration that is given to what amount of material will be lost from the active system based on solids control screen size.

In addition, the model can be used during laboratory testing to select and design particulate systems that will be used to mitigate lost circulation. As test results are compared with specific designs, a better understanding of how to optimize the particle size distribution may be achieved.

## Laboratory Screening of Lost Circulation Materials and Systems

In most cases it is customary in the industry to test lost circulation materials in the field mud being used, or a mud that is similar to the one that will be used on an upcoming well. This is a reasonable approach for picking LCM for a specific well.

When designing a new system we believe it is better to evaluate the particulate combinations in a very simple fluid that does not contribute to stopping the lost circulation. By doing this, changes in effectiveness can more easily be related back to changes in the design that have been modeled prior to the lab tests.

### *Base Fluid*

In order to maintain the quality and consistent properties of base fluid through out a test series, it is recommended to prepare enough fluid to be used during one week of testing. The procedure for preparing our base fluid is a follows: add citric acid to the mix water to adjust the pH to 4; then add 1.75 g of a dispersible xanthan polymer for each 350 ml of mix water. Let the mixture stir for an hour. Adjust the pH back to 9 using NaOH. Add 0.5g of a biocide for each 350 ml of water and mix for an additional hour using a medium shear mixer. **Table 2** shows the nominal rheological properties for the base fluid. Initially, only 1.25 ppg of polymer was used in the base fluid, but settling of solids in the PPA tests at 250 deg F required a higher concentration.

### *Fluid Loss Tests*

Initial screening tests for lost circulation are run on a 190-micron ceramic disk in a particle plugging test apparatus (PPA) at 150 deg F (65 deg C) and 250 deg F (121 deg C) and 1000 psi pressure differential.

Those formulations selected for further study are evaluated in a second set of tests run with the base fluid weighted to 12.0 ppg to validate the effect of weighting material on the results.

After formulations for further consideration are developed, a third set of lab tests is done using either particle plugging test equipment or HPHT fluid loss cells that are fitted with metal disks in which 0.02-inch (508 microns) and 0.04-in (1016 microns) slots, respectively, are cut. These tests are also run at 150 deg F (65 deg C) and 250 deg F (121 deg C) and 1000 psi pressure differential. The weight of fluid that flows through the slots is determined at the end of the test. These tests are very short duration, with either a quick shut-off occurring or all of the fluid expelled in a matter of a few seconds time. Other slot sizes may be used for specific field-related projects.

Screening tests for designing bridging material combinations to plug formation pores are run on 35-micron ceramic disks at 150 deg F (65 deg C) and 250 deg F (121 deg C) and 1000 psi pressure differential. Confirmation data may then be run on 10-, 20-, and 190-micron disks to confirm how wide a range of pore sizes can be plugged with the formulation. Ideally, consistent results will be obtained on all three sizes. The data captured during all the tests using ceramic disks are shown in **Table 3**.

The normalized PPT (NPPT) value shown in the table is used to reduce the data to a single value for comparison purposes between samples in a single test sequence. It may not be valid to compare values between different test series. This value is based on the premise that an initial high spurt loss is positive when treating for lost circulation, but a quick shut off is desirable after the initial spurt; thus the PPT Value divided by the Spurt Loss should be a value greater than, but near, one. Additionally, a low static filtration rate is desirable such that when the PPT Value/Spurt Loss is multiplied by the Static Filtration Rate a low combined value is desirable.

Finally, a series of tests under the same conditions of temperature and pressure are run on different types of drilling fluids that are maintained as a standard. These are a 12.0 ppg dispersed water based fluid, fresh water non-dispersed fluid, salt water non-dispersed fluid and a non-aqueous fluid, respectively.

## Engineered Combinations of Lost Circulation Materials

### *High Fluid Loss Squeeze (HFLS) Lost Circulation Treatment*

By combining past experience with our ability to model the particle size distribution of lost circulation material mixtures, a unique blend containing optimized types of lost circulation materials, including a resilient graphitic carbon, formulated with an optimized particle size distribution

(OPSD) has been developed. This OPSD is demonstrated by laboratory data showing efficient sealing of 190-micron pores as well as 500- and 1000-micron slots. This effectiveness for both small and large fractures is possible due to an optimized bimodal particle size distribution (**Figure 3**) design for the suite of particle types.

The HFLS lost circulation material is effective for both large pores and fractures as shown in both particle plugging testing on fritted disks and slot tests on steel disks at 1000 psi (69.0 bar) differential (**Table 4**).

**Simplicity in treatment.** Spotting an HFLS lost circulation material pill helps save rig time and operational costs since it requires no trips out of the hole, no special pumping or mixing equipment, and no specialized spacers. The HFLS lost circulation solution can work effectively in a wide range of fluid loss situations, and can be used in all types of drilling fluids to seal off loss zones quickly and economically.

**Versatility.** If a high fluid loss squeeze is not desired, the HFLS lost circulation material can be applied by adding directly to the drilling fluid system (**Table 5**). If desired, supplemental material such as larger resilient graphitic carbon or swelling polymers can also be used with HFLS.

### *Acid-soluble Lost Circulation Materials*

Lost circulation materials that are acid-soluble and/or breakable (AS&B) for use in the reservoir are few in number. The AS lost circulation treatment designed by applying the previously discussed modeling and testing protocol is a unique blend containing optimized types and sizes of materials that are 100% acid-soluble. In addition, the lost circulation material contains an OSPAR-compliant swelling natural polymer that is acid-breakable (B).

This AS&B optimized combination is demonstrated by laboratory data showing efficient sealing of 190-micron pores as well as 500- and 1000-micron slots. This effectiveness for both small and large fractures is possible due to a combination of a bimodal particle size distribution designed for the suite of particle types. Simple weighted polymer fluids with no other additives other than weight material are used to demonstrate the effect of the optimized particle distribution only (**Table 6**). The continued effectiveness in typical drilling fluids is demonstrated in **Table 7**.

These data also demonstrate the need to apply this combination of materials as a water based pill in synthetic based fluids to attain the full benefit of the swelling polymer, since the system was not sufficiently effective in a non-aqueous fluid at 250 deg F to plug the 1016-micron slot.

For particularly difficult lost circulation incidents, such as vugular carbonates or large natural fractures, it may be more efficient to use fiber lost circulation materials as a supplement to the AS&B lost circulation material to enhance the effectiveness of the treatment. Data for an acid-soluble mineral fiber used as a supplement is shown in **Table 8**.

### *Model Design versus Test Results*

**Table 9** shows the change in results with a change in components and particle size distribution. In this case, the PSD change was modeled as a means to select the best candidate material to be used to improve the fluid loss results on a 190-micron disk after the initial formulation failed. The initial formulation contained both fiber material and sized particulates. The PSD curve is shown in **Figure 4**.

Based on the previous results for the HFLS development a formulation was proposed that reduced the fiber and replaced it with a larger size particulate material with a different shape factor than used in the first formulation. This PSD curve is shown in **Figure 5**, and the improved results in Table 9.

A second formulation was modeled that removed the remaining fiber and replaced it with a significantly larger particulate material, as shown by the PSD curve in **Figure 6**. This formulation provided the best results of all those tested (Table 9) and became the final formulation for the product. The previous test results shown in Table 8 shows that the fiber material still has the capability of contributing to a better result when used in smaller amounts with the final particulate product containing both different shapes and larger size particulates. This had to be verified by the test results since the model cannot take shape into account.

No obvious differences in the PSD curve are apparent when **Figure 7** is compared with **Figure 6**, so experience and previous testing still plays an important role in obtaining optimal results when designing lost circulation materials.

## Field Results

The HFLS has been applied on 59 wells to date (January 2008) in China, South Texas, and Louisiana Gulf Coast, with the majority of the applications successful. Where applications were unsuccessful, attempts to cure losses with other materials were not successful as well.

The AS&B has not been applied as yet (January 2008) since the final lab work has just been completed.

### *Case 1*

The operator wanted to stop losses in a severely depleted zone (Perdido A) at 10,900 ft so that the lower productive zone could be evaluated. Several conventional LCM pills had been spotted, along with three high-filtrate squeezes provided by a competitor, and two cement squeezes. A 16.0-ppg mud density was required to control the pressure in the Perdido B sand.

After washing through the final failed high-filtrate squeeze and losing complete returns, a conventional LCM pill was spotted consisting of 40 ppb of resilient graphitic carbon material, 20 ppb of GM50 sized ground marble, 20 ppb of fiber LCM, and 20 ppb of other mixed particulate material. The pipe was then pulled to the shoe while waiting on the delivery of the HFLS lost circulation material.

When the material arrived on location, the bit was run back to 10,900 ft and partial returns (90%) were established at a minimum flow rate while two 50-bbl HFLS LCM pills were prepared. After the first 50-bbl pill was mixed, it was placed in the drillstring and the second 50-bbl pill was mixed. The drillstring was continually reciprocated to avoid becoming stuck while the second pill was mixed. The second pill was then pumped and the entire 100-bbl treatment was spotted in the wellbore. The bit was pulled to the top of the pill at ±6,500 ft (above the intermediate casing shoe @ 9205 ft) and the annular preventer was closed. The pumps were brought on line slowly. Initially the pressure came up to approximately 18.0 ppg equivalent mud weight (EMW) and then broke back to 17.2 ppg EMW while squeezing 50% of the HFLS LCM pill away (50 bbl).

After 50 bbl had been forced into the formation, the pumps were shut down and pressures were monitored for the next four hours. A slight rise in pressure was noted resulting in a 17.4 ppg EMW. After waiting four hours, the annular preventer was opened and the pressure was bled off. The drillstring was run in the hole to approximately 10,700 ft. The well was displaced with a 16.0 ppg water-based fluid with full returns. The driller washed and reamed down to 11,900 ft to allow logging evaluation of the production zone.

### *Case 2*

The operator drilled a 6-3/4” production hole to a depth of 12,371 ft with a 12.8 ppg water-based fluid. After making a short trip, the operator encountered total losses while washing and reaming at 12,294 ft. The operator was not successful in recovering returns after pumping a 44-ppb pill formulated with resilient graphitic carbon LCM.

After reviewing the information, drilling fluids technical personnel recommended that the operator spot a HFLS LCM pill before resorting to a cement squeeze. The HFLS LCM pill is designed to work rapidly in severe loss situations. Under direction of Baroid personnel, the rig crew mixed a 140-bbl, 12.8 ppg pill with 110 bbl of fresh water, 0.75 ppb xanthan polymer viscosifier/suspension agent, and 50 ppb HFLS lost circulation material.

The pill was then allowed to sit in the pit while the drillstring was tripped back into open hole at 10,865 ft. Prior to pumping the LCM pill, 2 ppb of a swelling polymer lost circulation material was added to the pill. The HFLS provides a seal-and-plug function and the swelling polymer LCM provides a swell-and-plug function. Due to rig complications, only 60 bbl of the of the 140-bbl pill was spotted across the loss zones in open hole. The drillstring was then pulled above the pill and squeeze pressure was applied. The pressure was increased to 850 psi and then bled down to 175 psi and held for three hours.

After tripping back in the hole to the loss area, the operator was able to drill through the HFLS LCM pill to reach 12,371 ft, and then drilled ahead to total depth (TD) at 12,700 ft with no further losses. No special pumping equipment was required. The HFLS LCM pill was pumped with the rig pumps at full pump rates with pump pressures at 2900 psi. The pill was pumped through 3-1/2 ” drill pipe, 4-3/4” drill collars, a 4-3/4” mud motor, and a 6-3/4” bit with a 0.518 sq-in TFA.

### *Case 3*

While drilling the 6-3/4” production hole with a 13.2 ppg water-based fluid, the operator encountered total losses at 11,770 ft. They were not successful in recovering returns after staging in the hole cutting the mud weight back to 12.8 ppg and adding lost circulation materials (LCM) directly to the active system

Drilling fluids personnel recommended spotting a HFLS LCM pill for high fluid loss environments before resorting to pumping cement. The HFLS pill is a high-filtrate sized particulate pill that is designed to work quickly in severe loss situations. A 140-bbl pill weighing 12.8 ppg was mixed. The pill was then allowed to sit in the pit while the drillstring was tripped into open hole at 10,865 ft. The drillstring consisted of 3.5 inch drill pipe, 4.75 inch drill collars, 4.75 inch mud motor, and a 6.75 inch bit with a .518 sq-in TFA.

The HFLS LCM pill was pumped through the drillstring and mud motor with no adverse effects. The pill was spotted across the loss zones in the open hole. The top of the pill was above the casing shoe. The drillstring was then pulled above the pill and an 800-psi (14.5 ppg EMW) squeeze was applied on the pill for three hours. After tripping back in the hole to the loss area, the operator was able to drill through the HFLS LCM pill and on to original bit depth of 11,770 ft. Drilling resumed past 11,770 ft with no further losses.

## Summary

The use over time of a model to estimate the fracture width that may be induced in a wellbore allows a better understanding of what particle size distribution and types of materials may be most efficient.[7]

It also can illustrate how the d50 parameter is totally insufficient to describe a particle size distribution (PSD), and that, though better, knowing the d10 and d90 is still not sufficient. This is illustrated by comparing the data in **Table 10** with Figures 4, 5 and 6. The d50 for each of these systems is relatively close to one another. In addition, for two cases the d10 values are near one another. The d90 values vary the most, but again do not tell the entire story of what type of particle size distribution is present for each case. On the other hand, by viewing the entire PSD curve, a description may be made from normal, through bimodal, to a broad distribution.

## Conclusions

- The use of a design module capable of calculating full particle size distribution curves for mixtures of lost circulation materials provides an efficient means to plan and evaluate laboratory tests on LCM.
- Two new LCM systems have been designed and tested in the laboratory with the assistance from this model. Field results from application of the HFLS system designed with a bimodal PSD are very encouraging.
- The use of simple fluids to develop new LCM systems is encouraged so as to better evaluate the effects of different particle types, shapes and sizes without masking from other drilling fluid components

- Experience and knowledge of previous test results are still important when designing lost circulation materials.

## References

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## Acknowledgements

Dr Cindy Fang did the laboratory development testing for the HFLS product. Michael Vackar and Dr Vincent Yang provided the laser PSD measurements.

## Tables

**Table 1: Borehole Stress Treating Materials**

| Material | D50 (microns) |
|---|---|
| GM 1200 LCM | 1200 |
| RGC 1000 LCM | 1000 |
| GM 600 LCM | 600 |
| RGC 400 LCM | 350-400 |
| GM 150 LCM | 150 |
| RGC 100 LCM | 85-100 |
| RGC 50 LCM | 50-55 |
| GM 50 LCM | 50 |
| GM 25 LCM | 25 |
| GM 5 LCM | 5 |

**Table 2. Base Fluid Rheology**

| Rheology | 600 rpm | 300 rpm | 200 rpm | 100 rpm | 6 rpm | 3 rpm | PV | YP | n | K | $\tau_o$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Base Fluid** | 23 | 18 | 17 | 14 | 8 | 6 | 5 | 13 | | | 4.59 |

**Table 3: Data Recorded for Particle Plugging Tests On Ceramic Disks**

| PPT Data Recorded |
|---|
| 10 sec, mL |
| 1 min, mL |
| 7.5 min, mL ($V_{7.5}$) |
| 15 min, mL |
| 25 min, mL |
| 30 min, mL ($V_{30}$) |
| PPT value, mL ($2\times V_{30}$) |
| Spurt loss, mL ($2\times(V_{7.5}-(V_{30}-V_{7.5}))$ |
| Static Filtration Rate, mL ($2\times(V_{30}-V_{7.5})$)/2.739 |
| NPPT = (PPT V/SL)*SFR |

**Table 4: Fluid loss testing in a 1.25 ppb (3.6 kg/m$^3$) xanthan polymer water fluid after the addition of 50 ppb (140 kg/m$^3$) HFLS LCM.**

| Test Medium | Temperature deg F | Spurt Loss ml | PPT Value ml | Static Filtration Rate, ml | NPPT |
|---|---|---|---|---|---|
| **190 micron disk** | 150 | 68 | 76 | 1.46 | 1.63 |
| **508 micron slot** | 150 | 6.6* | NA | NA | NA |
| **1016 micron slot** | 150 | 33.8* | NA | NA | NA |
| **190 micron disk** | 250 | 88 | 112 | 4.38 | 5.57 |
| **508 micron slot** | 250 | 16.4* | NA | NA | NA |
| **1016 micron slot** | 250 | 28.5* | NA | NA | NA |

*grams ; NA = Not Applicable

**Table 5: Fluid loss @ 1000 psi differential after treatment with 50 ppb (140 kg/m$^{3)}$ HFLS LCM in 12.0 ppg drilling fluids**

| Drilling Fluid | Temperature Deg F | 508 micron slot, g | 1016 micron slot, g |
|---|---|---|---|
| Lignosulfonate Dispersed | 150 | 14.87 | 42.42 |
| Lignosulfonate Dispersed | 250 | 23.50 | 43.0 |
| 20% Salt Non-Dispersed | 150 | 4.68 | 6.69 |
| 20% Salt Non-Dispersed | 250 | 10.4 | 42.59 |
| Internal Olefin Synthetic Base Fluid | 150 | 3.44 | 43.51 |
| Internal Olefin Synthetic Base Fluid | 250 | 7.87 | 35.0 |

**Table 6 – Acid-soluble lost circulation treatment: particle plugging tests @ 1000 psi differential tested in 1.75 ppb xanthan polymer water fluid weighted to 12.0 ppg**

| Test Medium | Temperature deg F | Spurt Loss | PPT Value | Static Filtration Rate |
|---|---|---|---|---|
| **190 Micron disk** | **250** | 44.0 ml | 76.0 ml | 5.84 ml |
| **508 micron slot** | **250** | NA | 18.6 g | NA |
| **1016 micron slot** | **250** | NA | 39.2 g | NA |

*grams NA = Not Applicable

**Table 7 - Fluid loss @ 1000 psi differential after treatment with 50 ppb acid-soluble LCM in 12.0 ppg drilling fluids**

| Drilling Fluid | Temperature | 508 micron slot, g | 1016 micron slot, g |
|---|---|---|---|
| Non-Dispersed Fresh Water | 150 | 10.3 | 48.9 |
| Non-Dispersed Fresh Water | 250 | 16.3 | 49.8 |
| Non-Dispersed w/20% Salt | 150 | 4.9 | 30.0 |
| Non-Dispersed w/20% Salt | 250 | 7.0 | 56.7 |
| Synthetic Base Fluid | 150 | 0.4 | 9.5 |
| Synthetic Base Fluid | 250 * | No control* | No control* |

*requires application with a water base pill

**Table 8 - Fluid loss @ 1000 psi differential after treatment with 50 ppb AS&B lost circulation material and 5 ppb mineral fiber LCM**

| Drilling Fluid | Temperature | 508 micron slot, g | 1016 micron slot, g |
|---|---|---|---|
| 12 ppg xanthan viscosified (1.75 ppb) water-base fluid | 250 | 8.7 | 20.3 |
| Results from Table 4 with no fiber | 250 | 18.6 | 39.2 |

**Table 9: Material Evaluation: Particle plugging tests on 190 micron disk@ 150 deg F and 1000 psi differential. Tested in 1.25 ppb BARAZAN® D water-base fluid**

| Test Material | Spurt Loss, ml | PPT Value, ml | Static Filtration Rate, ml | NPPT |
|---|---|---|---|---|
| Conventional particle mix plus 28 ppb fiber | - | No control | - | NA |
| Conventional particle mix w/14 ppb fiber + 9.2 ppb GM lost circulation material | 56 | 84 | 7.7 | 11.55 |
| Conventional particle mix w/ 9.2 ppb of GM & Flake CaCO3 lost circulation materials, respectively | 71 | 81 | 1.8 | 2.05 |

**Table 10: d50 does not tell the whole story**

| Distribution | Components | D10 | D50 | D90 |
|---|---|---|---|---|
| Normal | 1 | 67 | 185 | 321 |
| Bi-Modal | 2 | 75 | 212 | 739 |
| Broad | 4 | 17 | 168 | 502 |

## Figures

**Figure 1: Near Wellbore Fracture Module**

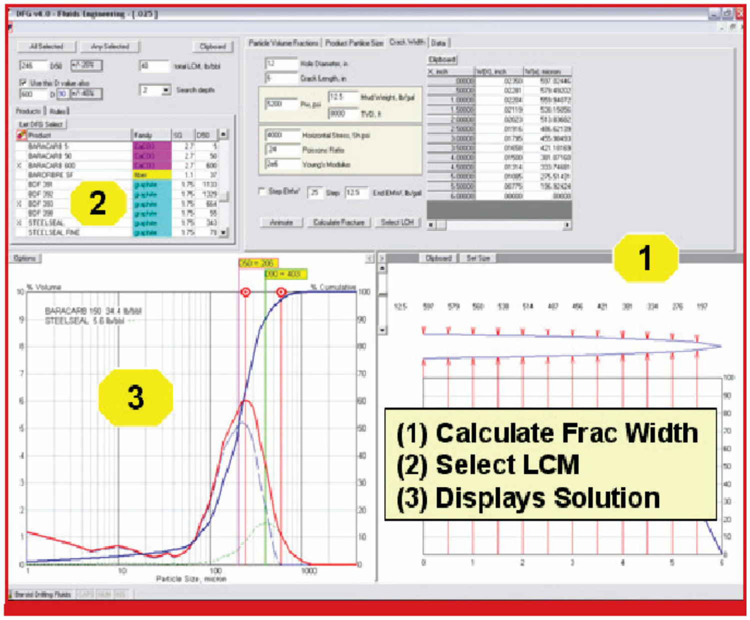


**Figure 2: LCM Three Component Particle Size Distribution**

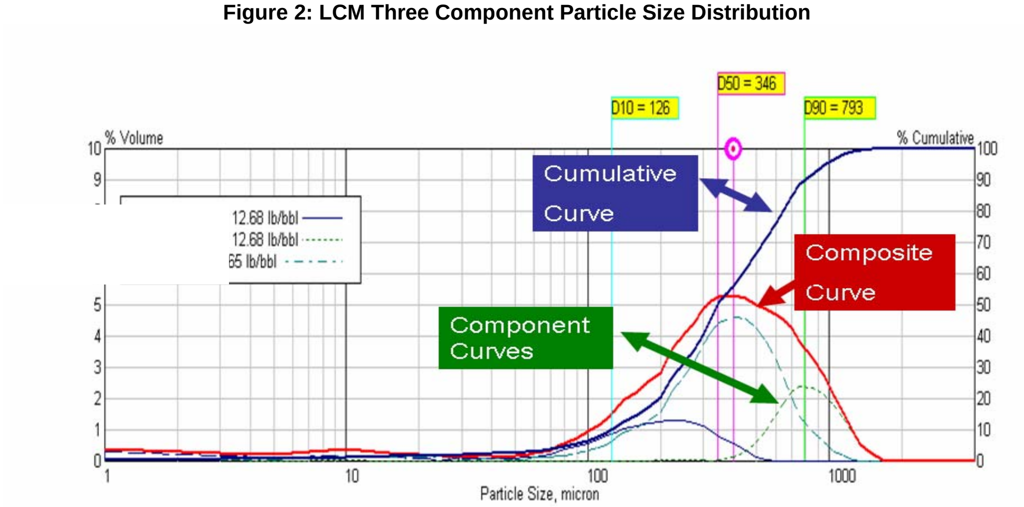

**Figure 3: Five Component Bi-Modal Distribution**

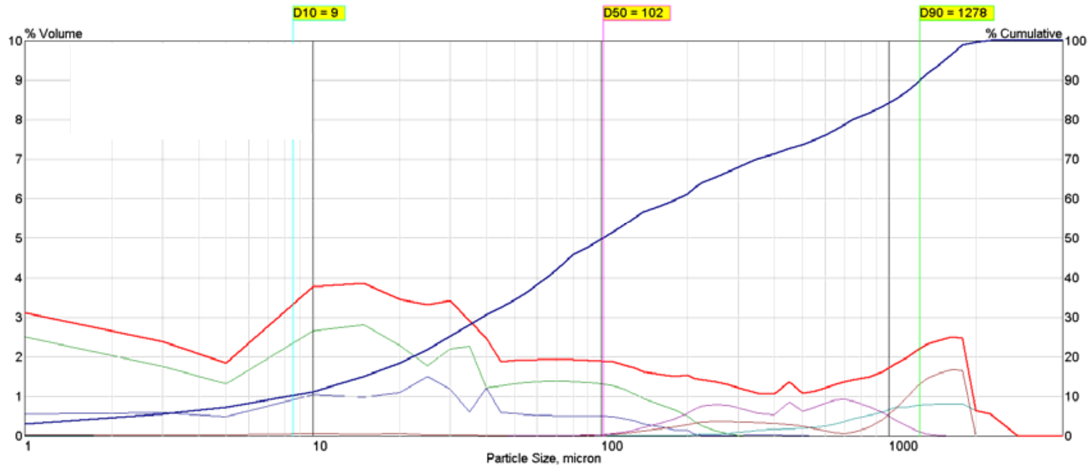


**Figure 4: Initial LCM Formulation with Particulates and Fiber**

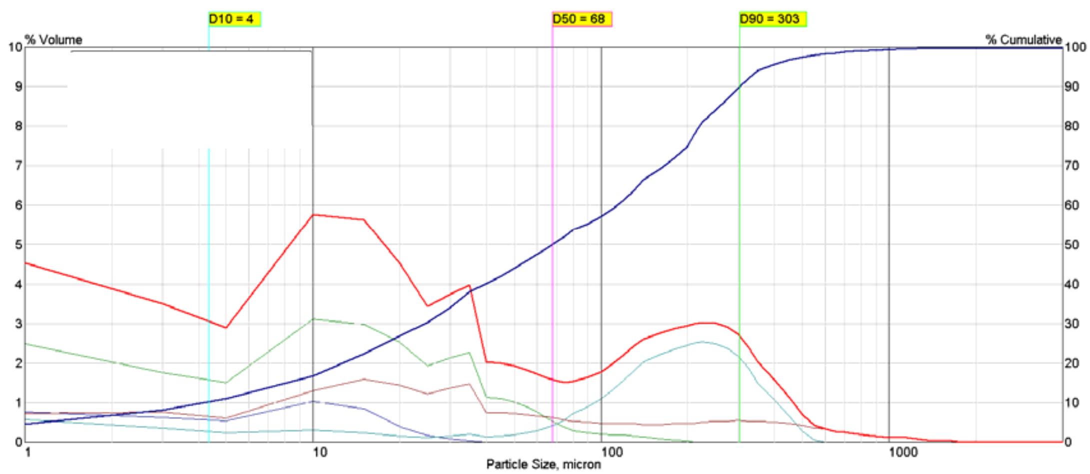


**Figure 5: LCM Formulation with Reduced Fiber and Different Shape Particulate**

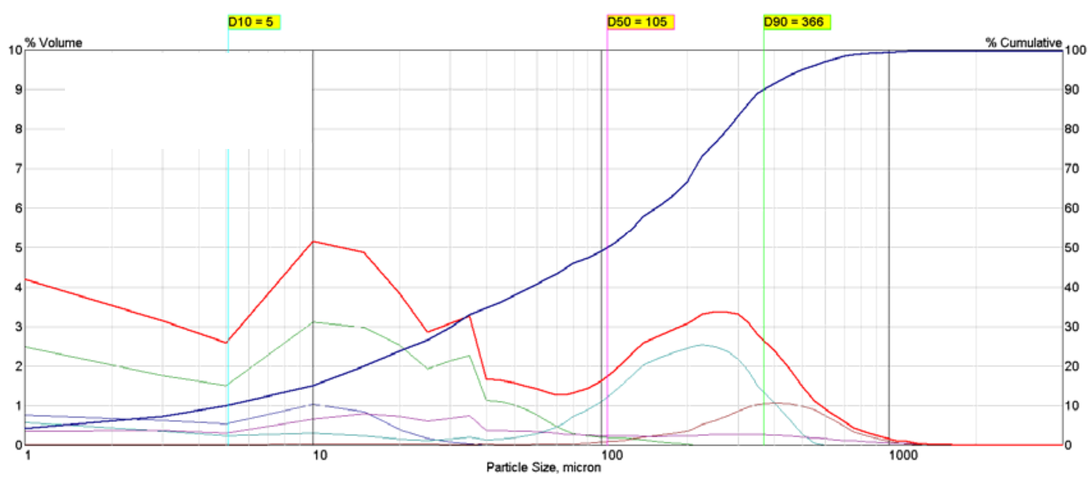

**Figure 6: Fiber Replaced by Different Shape and Larger Size Particulate**

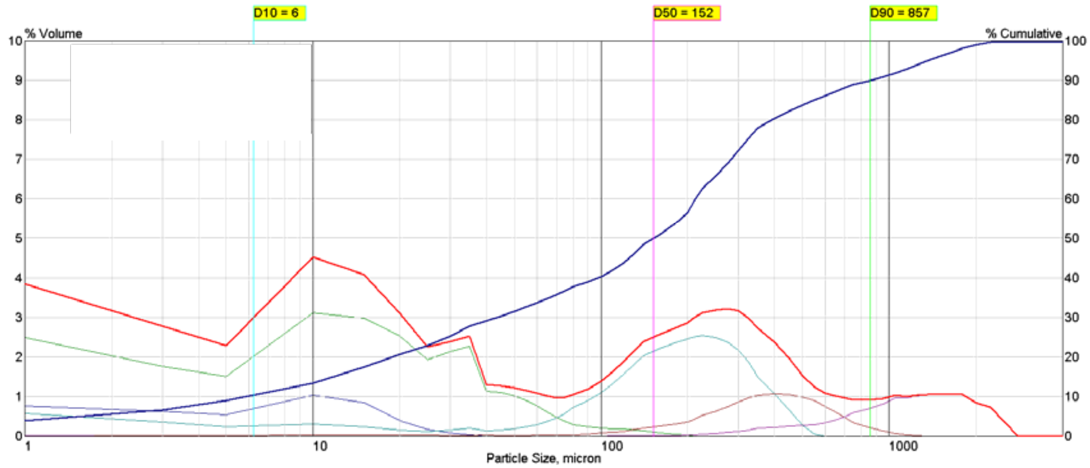


**Figure 7: Fiber Supplement Added to the Best Particulate Formulation Shows No Obvious Differences but Test Results Are Better.**

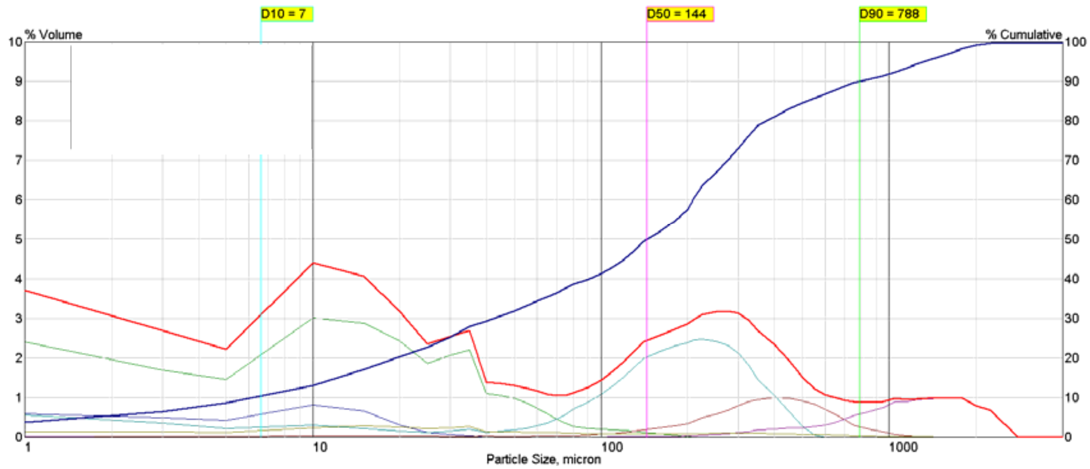


**Figure 8: One Component Normal Distribution**

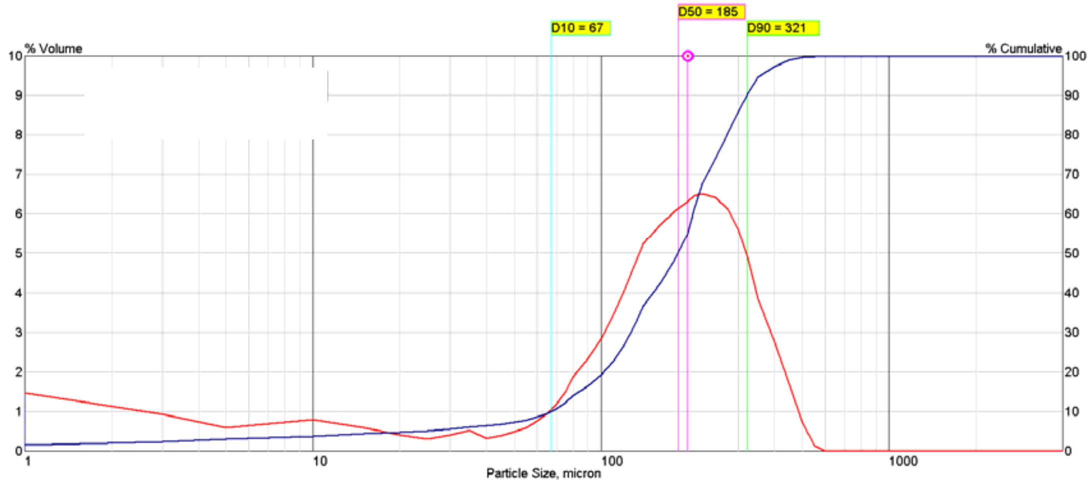

**Figure 9: Two Component Bi-Modal Distribution**

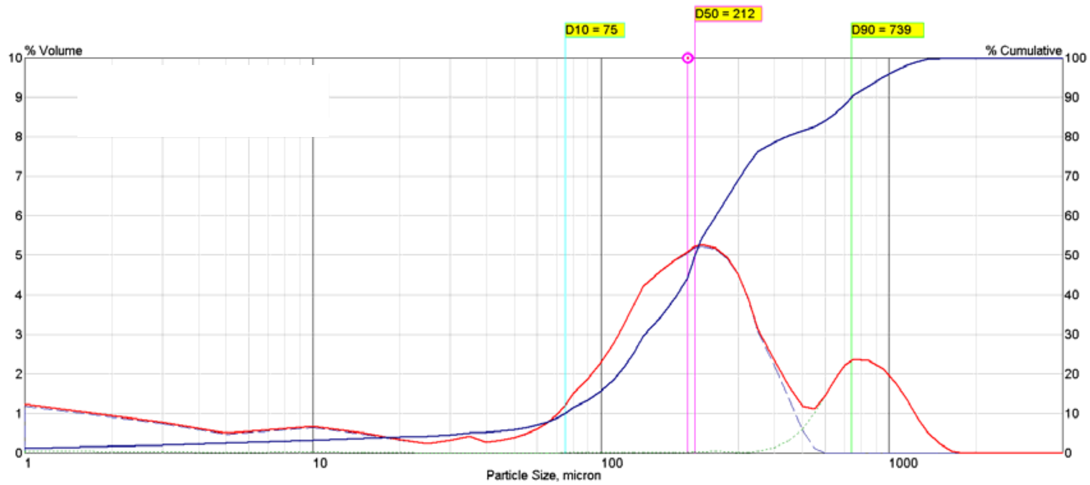


**Figure 10: Four Component Broad and Bimodal Distribution**