

## Shear Degradability of Granular Lost Circulation Materials

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### Abstract

This report summarizes the results of a laboratory study undertaken to document and better understand the physical degradation experienced by lost circulation materials (LCMs) in a circulating drilling fluid during drilling operations. In this study, 15 conventional “granular” LCMs used to remediate and prevent lost circulation were subjected to shear degradation tests, and the effect of shear on the particle size distribution (PSD) was measured. Four chemical families of LCMs were studied: marble (metamorphosed high-purity limestone); carbon (graphite and petroleum coke); a tough, short-filament fiber; and pecan nut shells. All of these had aspect ratios of length/width/breadth  $\sim 1/1/1$ , hence were classified as granular.

The results indicate that, while the carbon-based products are more resistant than the marble-based products to shear degradation, neither one is particularly long-lasting, especially compared to the fibers and nut shells, which exhibited little or no change in their PSDs. An exception is a high-resiliency carbon-based LCM, which showed much greater resistance to shear degradation than the other carbon-based products, though slightly lower than the fiber and nut shell products.

Both the carbon- and marble-based products exhibited greater resistance to shear degradation with decreasing particle size, and the trend appeared to be stronger for the carbon than for the marble product line. A fine-mesh marble exhibited unusually high sensitivity to shear, which was not consistent with that trend; this may not be surprising, though, considering that it is sourced differently than the other marble products and is ground in a manner that may make it more susceptible to fracturing. Neither the fiber nor the nut shell demonstrated a clear size dependence of shear resistance based on particle size.

### Introduction

Continuous wellbore strengthening treatments generally require maintenance of a specific size distribution of the lost circulation material (LCM) in the whole mud. Unfortunately, these LCM are susceptible to comminution (size degradation) as they circulate through the bit and wellbore, which results in the particle size distribution (PSD) of the LCM becoming finer as drilling proceeds. Consequently, maintenance of a target PSD requires continuous addition of coarser particulates and removal of the fines. Although some studies of LCM degradability have been carried out in the past, these looked at

only a few LCM and then only at total recovery of material after brief shearing of LCM suspended in a drilling fluid. Since size of the LCM is a key property that controls its ability to seal and strengthen fractures, it is critical to measure the effect of shear on the full PSD of the material. Furthermore, we still have little understanding of the effects of size and composition of the LCM on its degradability. Work is required to quantify these properties so that we may provide some guidance on the types, sizes and amounts of LCM that are employed in the initial and subsequent maintenance treatments of the drilling fluid.

In this study, a simple shear test was used to examine the relative susceptibility to shear of various types and sizes of M-I SWACO granular LCMs, and the effects of the shearing process on the materials' full PSDs were measured before and after shearing. A total of 15 LCMs from four chemical families of products were tested.

### Experimental Approach

Granular materials that are used as LCMs cover a range of median particle size,  $d_{50}$ , from 2 to 2500  $\mu\text{m}$ . For wellbore strengthening treatments, this is typically adjusted upward, perhaps to a range of 40 to 2500+  $\mu\text{m}$ . To address that application, shear tests were carried out with representative samples of granular LCM with  $d_{50} \geq 40 \mu\text{m}$ . These materials included

Marble 40, 250, 500, 600, 750, 1400, 2500  
Graphite 350  
Graphite High-Resiliency (GHR) 500  
Graphite/Petroleum Coke Blend (GPCB) 150, 750  
Fiber 100, 300  
Nut Shell 450, 1400

where the number affixed to the product type corresponds to the nominal  $d_{50}$  of the material. Three methods were examined to determine their suitability for determining PSDs of the LCMs: Image Analysis (IA), Laser Diffraction or Light Scattering (LLS) and Dry Sieve Analysis (DSA).<sup>1</sup> The latter most closely resembles the methodology used to classify LCMs during preparation and is used to establish approximate product specifications of these materials. IA, which is typically carried out via contour analysis of photomicrographic images of the material, has the advantages of providing information on the morphology of the material

and of providing direct observation of particle size. However, it is tedious to use for characterization of materials with broad distributions of particle sizes. Nevertheless, the 2-dimensional PSD generated by IA, when converted to a standard volumetric PSD, serves as an excellent reference by which to compare other techniques. LLS requires suspension of the particles in a liquid medium and may require dilution and perhaps sonication or other technique to prevent agglomeration. With input on the refractive index of the material, most LLS instruments can provide rapid quantitative PSDs over the range 1 to 2000  $\mu\text{m}$ . DSA, whether through use of stacked sieves or individual sieve, can provide quantitative mass/volume PSDs for particles > 20  $\mu\text{m}$  in size, though some types of fine particles may require treatment with a flow additive to neutralize electrostatic charges.

Previous studies have suggested that the PSDs generated by DSA for spherical materials match PSD curves obtained via IA fairly well over the size range 100 to 2000  $\mu\text{m}$ . The same may be said for LLS vs IA for particle sizes in the range 1 to 50  $\mu\text{m}$ . Larger particles present various challenges for LLS, particularly settling, uncertainties due to the small sample size, and limitations of the equivalent sphere model. Fig. 1 demonstrates how the  $d_{50}$  of Marble samples determined via LLS varies from that determined via DSA. This plot of  $d_{50}(\text{LLS})$  vs  $d_{50}(\text{DS})$  shows clearly that as particle size increases, LLS generates increasingly larger values of  $d_{50}$  than does DSA.

The foregoing discussion suggests that accurate and reproducible PSDs of granular materials with  $d_{50} \geq 100 \mu\text{m}$  may be obtained using DSA; for smaller particulates, LLS is probably the better choice. Consequently, the PSDs of all the LCMs save Marble 40 were measured with DSA, using a Hosokawa Jet Air Sieve Analyzer. Ten sieves were used in each case, with the mid-range sieve (5<sup>th</sup> or 6<sup>th</sup> sieve) chosen so as to have an opening size approximating the  $d_{50}$ . The PSD of Marble 40 was measured in water using a Beckman Coulter laser diffraction system.

For the Shear Tests, all of the LCM samples were first dried overnight in a static oven at 105°C. Most of the LCMs lost < 2% by weight, which was attributed to moisture. Only the Fiber products lost a significant amount: 7% for Fiber 300 and 10% for Fiber 100. The shear tests were conducted with a 3-speed Waring blender on a speed setting of “high”. For each test, 25 g of the LCM was first dispersed in 1 lab equivalent bbl of Houston tap water, and then sheared for 5 min. A duplicate sample was sheared for 10 min. The choices of 5 and 10 min were made based on preliminary Shear Tests with Marble 250 to generate differentiable PSDs. Two of the series of shear tests (Marble 250 and 500) were repeated to ensure reproducibility of the test results.

After shearing, all solids in the blender vessel were decanted, along with washings of residue in the vessel, through a 400-mesh screen. The resulting cake was scraped gently onto a watch glass, dried overnight, and the contents weighed. The PSD of the material recovered from the 5- and 10-min shear tests was measured in the same manner as the

initial sample, using the same set of sieves.

All of the PSDs were corrected for the material lost through the 400-mesh screen, i.e. < 38  $\mu\text{m}$ . The cumulative PSDs for the initial, 5-min and 10-min shear test samples were plotted and estimates were made by eye of the  $d_{10}$ ,  $d_{50}$  and  $d_{90}$  for each PSD curve.

## Results

The effects of shear degradation on the PSDs of the granular LCMs are plotted as bar charts in the form of “Resistance to Shear” for 5 and 10 min of shear using  $d_{50}$  (Fig. 2) and  $d_{90}$  (Fig. 3). Resistance to Shear is defined here as:

$$100 * d_s^t / d_s^0$$

where  $d_s$  is the particle size ( $d_{50}$  or  $d_{90}$ ) and the superscript  $t$  is the duration of the shear test (5 or 10 min).

For trend analysis, the data were re-plotted in various ways. The effects of the initial  $d_{50}$  and  $d_{90}$ , respectively, on the Resistance to Shear are plotted in Figs. 4 and 5, respectively, which show, though not very clearly, that Resistance to Shear increases with decreasing initial particle size. The large scatter in these composite plots appears to be at least partly associated with effects related to the type of particulate. When the results are grouped by chemical family, much clearer trends are evident.

The marble products as a group show significantly less Resistance to Shear than any of the other groups (Figs. 6 and 7), and the Resistance to Shear clearly increases with decreasing initial particle size. Marble 40 appears to be an outlier. It shows low Resistance to Shear, particularly in the  $d_{90}$  correlation, in spite of its relatively small initial size. The authors speculate that this is related to the grinding method: Marble 40 probably is generated using a Raymond® roller mill, whereas Marble 250 and larger sizes of marble are generated using screen rock techniques with stacked screens.<sup>2</sup> The roller mill is thought to generate much higher grinding and impact forces than the screen rock techniques, which is attested to by striations and other imperfections on the surfaces of Marble 40 particles, and we speculate that particles of Marble 40 possess microfractures that make them more friable than the larger marble products. The apparent outlier status of Marble 250 that is observed in the  $d_{50}$  correlation is a mystery and actually may be an illusion created by the presumed flatness of the curves drawn through the data. Indeed, the  $d_{50}$  correlation of Resistance to Shear vs initial size looks to be much weaker than the  $d_{90}$  correlation. In the latter, the data for Marble 250 are not obviously outliers, and the curves in that size range may easily be re-drawn with a steeper slope to include those data.

Figs. 8 and 9 show the initial particle size correlations obtained for the graphite-based products. These show a similar but steeper trend of Resistance to Shear with initial  $d_{50}$  and  $d_{90}$  compared to the marble products, and clearly all of the graphites exhibit greater Resistance to Shear than the marbles. However, GHR 500 shows an exceptionally high Resistance to Shear. This may be a by-product of its high resiliency,

although the authors think it is more likely due to a tougher skin being formed on the product compared with the other carbon-based LCMs; according to the manufacturer, GHR 500 is made by a process that results in a more uniform and tougher outer shell than the other carbon-based LCMs.

Of the products examined in this study, the cellulosic Fiber and Nut Shell products proved to be the most resistant to shear forces. This is demonstrated in **Figs. 10 and 11**, which show the effect of initial particle size on Resistance to Shear after 10 min for all four LCM families, namely Marble, Graphite, Fiber and Nut Shell. The Resistance to Shear increases in the order

Marble < Graphite < Fiber and Nut Shell

The particle size trend lines for the Nut Shell and Fiber products appear to run in the opposite direction to those observed for the Marble and Graphite products, i.e. Resistance to Shear decreases with decreasing initial particle size. However, the trends are weak and may be attributable to uncertainties in the test results. This is particularly true for the Fiber products. When dried after a shear test, recovered Fiber formed a mat that had to be broken up by gentle manual manipulation. This may have caused some comminution of the material and skewed the PSD to lower values.

The results observed in this work have been verified qualitatively in previous studies. In one study, the LCM was first sieved with a 60-mesh (250  $\mu\text{m}$ ) screen, dispersed in a liquid or drilling fluid, sheared for various periods of time with a Silverson L5M Lab Mixer rotating at 7000 rpm, and the amount of dried material recovered on the 60-mesh screen was measured and compared with the initial amount.<sup>3</sup> The total amount of degradation observed for four LCMs after 15 min was as follows: Marble 250 - 90.4%, GPCB 150 - 28.1%, Graphite 350 - 19.9% and Fiber (no grade specified) - 7.3%. The findings presented here agree qualitatively with those results.

### Field Studies

When the whole fluid system is treated with LCM, it may prove economical to configure the solids control equipment (SCE) to retain the LCM. However, field studies indicate that the PSD of the LCM shifts downward with increasing residence time in the mud while drilling, indicating physical degradation of the material as it circulates with the mud. In recent drilling operations in which the fluids were treated with blends of Marble and Graphite, a large fraction of the recovered Marble was found to have been degraded in size; the recovered Graphite had also degraded, but much less so.<sup>4</sup> Other authors<sup>5</sup> recently opined, "calcium carbonate could be expected to degrade more rapidly than most graphite products, and some fibers may not degrade at all." Some confirmation of this claim has come from recent tests conducted with a triple deck shaker designed to separate LCM from cuttings and fines and return the LCM to the active system. Those tests demonstrated that comminution of Marble and Graphite occurs downhole during normal drilling operations.<sup>6</sup>

In the field studies described above, degradation of the particulates has been attributed to shear at the bit, impact on the wellbore walls, and grinding between pipe and openhole/casing. However, forces capable of tearing apart particulates also exist in other places on the rig. More than a decade ago, a study was conducted to examine the effect of rigsite pumping and separation equipment on comminution of Marble 40 that was used to weight up a reservoir drilling fluid.<sup>7</sup> It became evident that, independent of its travel through the drillpipe and annulus, Marble was easily comminuted even by the surface hardware, which led to reduction in median particle size to as low as 5  $\mu\text{m}$ . Regardless of the mechanism by which LCM physically degrade, the results of field studies consistently indicate that both Marble (calcium carbonate) and synthetic graphite comminute rapidly, and that even after one circulation a significant fraction of the Marble is smaller in size; the synthetic graphite, while generally more resistant than Marble to comminution, nevertheless is also susceptible to shear degradation, resulting in a similar but smaller downward shift in the PSD.

### Maintenance Requirements at the Rigsite

Comminution of the LCM requires continuous addition of larger LCM to compensate for the material that becomes degraded in the course of drilling. Although rigsite requirements for a prescribed PSD of the LCM blend necessitate development of a project-specific maintenance schedule, the results presented here indicate that maintenance of a prescribed PSD with marble and/or carbon-based materials will require larger inventories than with Fiber and Nut Shells.

Rigsite devices used to monitor the PSD of particulates in drilling fluids, such as stacked wet sieves and laser reflectance, measure particle size but provide no information about chemical identity of the particulates. Similarly, algorithms used to calculate the required amounts of each LCM are based strictly on size, and a default position is that maintenance additions are carried out with only the largest particulates, independent of type.

The SCE system includes shakers and driers with screens that remove cuttings larger than 3 mm in average diameter and fines between 75 and 120  $\mu\text{m}$ , while capturing and returning LCM (particulates between 120 and 3000  $\mu\text{m}$ ) to the active system. A centrifuge is often used to limit the concentration of ultrafines (< 75  $\mu\text{m}$ ). The SCE is configured so as to maximize removal of unwanted drilled cuttings and fines and retention of the LCM, again without specific consideration of the nature of the LCM. Experience, though, dictates including some types of materials, such as graphite, regardless of the PSD requirements, because graphite may improve the tightness or integrity of the LCM bridge.

### Conclusions

Although the authors are not aware of any systematic study of the effect of circulation while drilling on the integrity of LCM, it is widely believed that carbonates, including marble,

physically degrade more rapidly than carbon-based LCM (graphite, coke), and that nut shells are the most resistant to comminution. The simple shear tests conducted in this study likely do not simulate exactly the forces experienced by LCMs downhole, but we expect that these tests can provide an idea of the relative susceptibility of the LCM to comminution under downhole conditions. The results obtained here for four families of granular LCMs bear this out. It was found that Resistance to Shear increases in the order

$$\text{Marble} < \text{Graphite} < \text{Fiber and Nut Shell}$$

with the exception of GHR 150, which in these tests proved to be nearly as resistant to shear forces as the Fiber and Nut Shell products. A fairly strong dependence of Resistance to Shear on initial particle size was found for the other members of the Graphite family, while the relationship was weaker for the Marble family and little or no clear relationship on initial particle size was found for Nut Shell and Fiber.

On the basis of the observed susceptibility of each LCM to degradation by shear forces, it may be concluded that GHR 500, Nut Shell 450 and 1400, and Fiber 100 and 300 are less shear-degradable than the other comparatively sized granular LCMs examined in this study.

## Acknowledgments

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## Nomenclature

- $d_i$  = Maximum particle size for  $i\%$  of the particulate population. For example,  $d_{90}$  is the maximum diameter of 90% of the particulates, i.e. 90% of the particulates are of a size equal to or less than  $d_{90}$ .
- $D_i$  = Maximum particle size for  $i\%$  of the particulate population that will pass through a shaker screen or

sieve. Typically screen designations are made with reference to  $D_{100}$  (in  $\mu\text{m}$ ) or API number: <sup>8</sup> 100% of spherical standard particles ( $\text{Al}_2\text{O}_3$  or glass) smaller than this size will pass through, and 100% of such particles larger than this size will be retained.

*GHR* = Graphite of High Resiliency, where Resiliency is defined as the % Axial Recovery of a confined pellet of the subject material after momentary exposure to 10,000 psig.

*GPCB* = Graphite/Petroleum Coke Blend

*LCM* = Lost Circulation Material

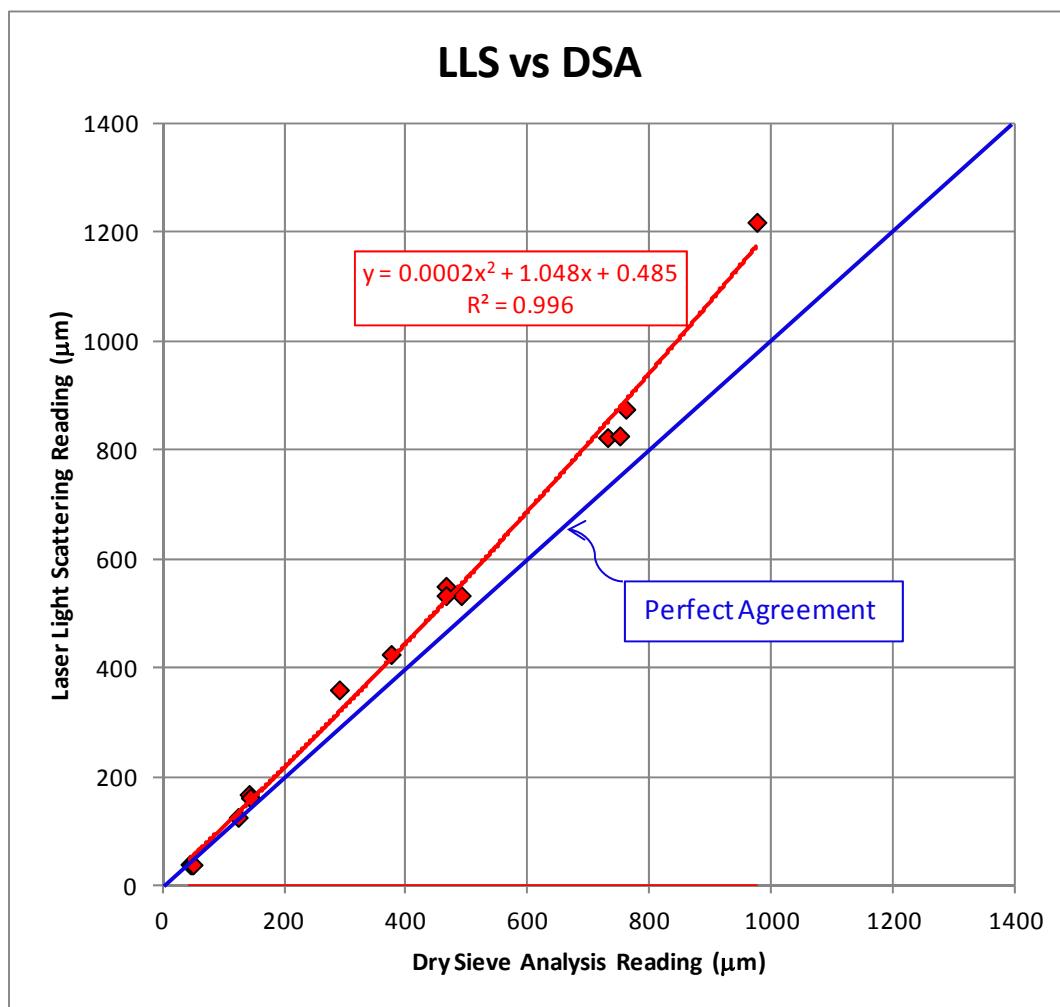
*LGS* = Low-Gravity Solids, i.e. particulates with density less than that of weighting material ( $\sim 4.2 \text{ g/cm}^3$ )

*PSD* = Particle Size Distribution

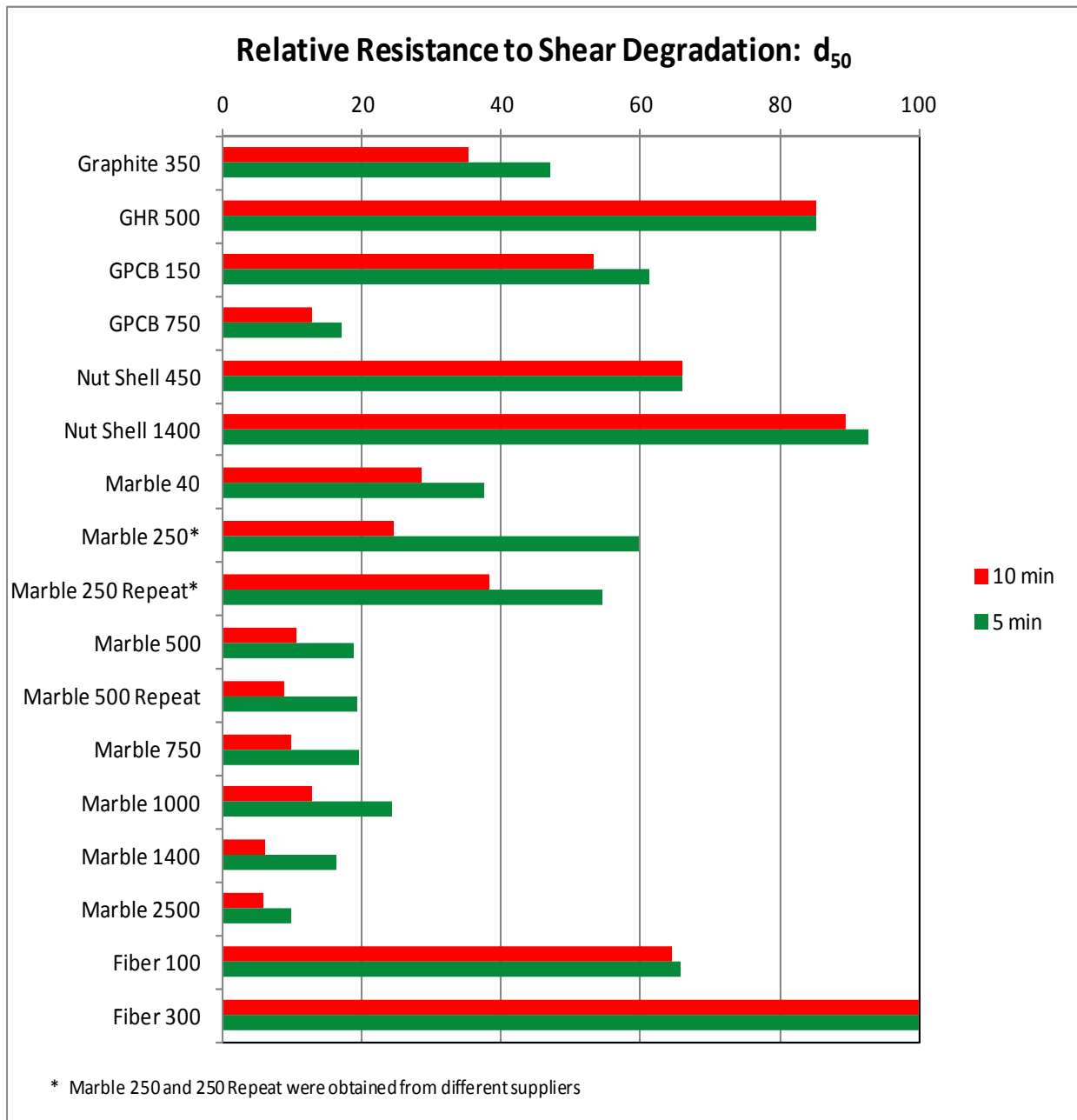
*SCE* = Solids Control Equipment

## References

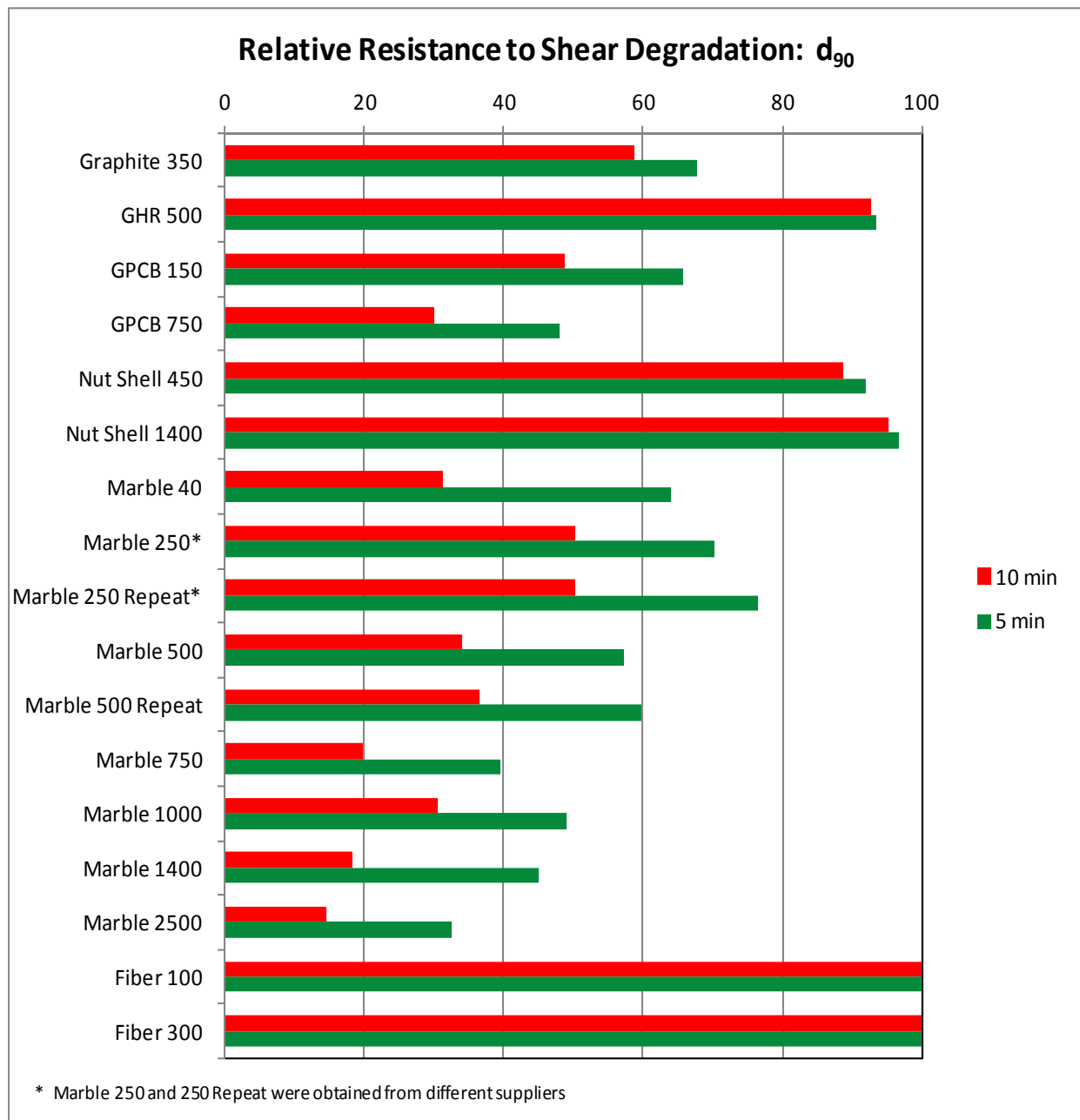
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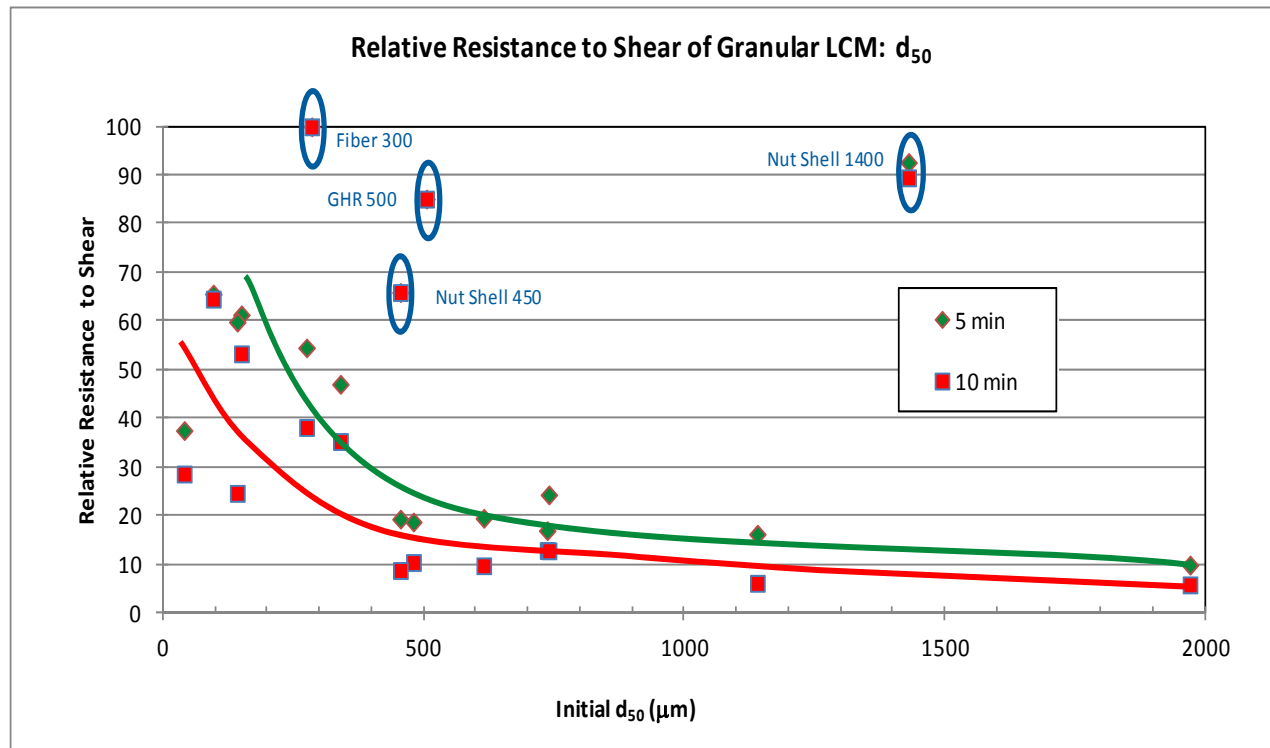
**Figure 1 – Correlation between Laser Light Scattering (LLS) and Dry Sieve Analysis (DSA)**  
Marble samples with  $d_{50}$  ranging from 40 to 1000  $\mu\text{m}$



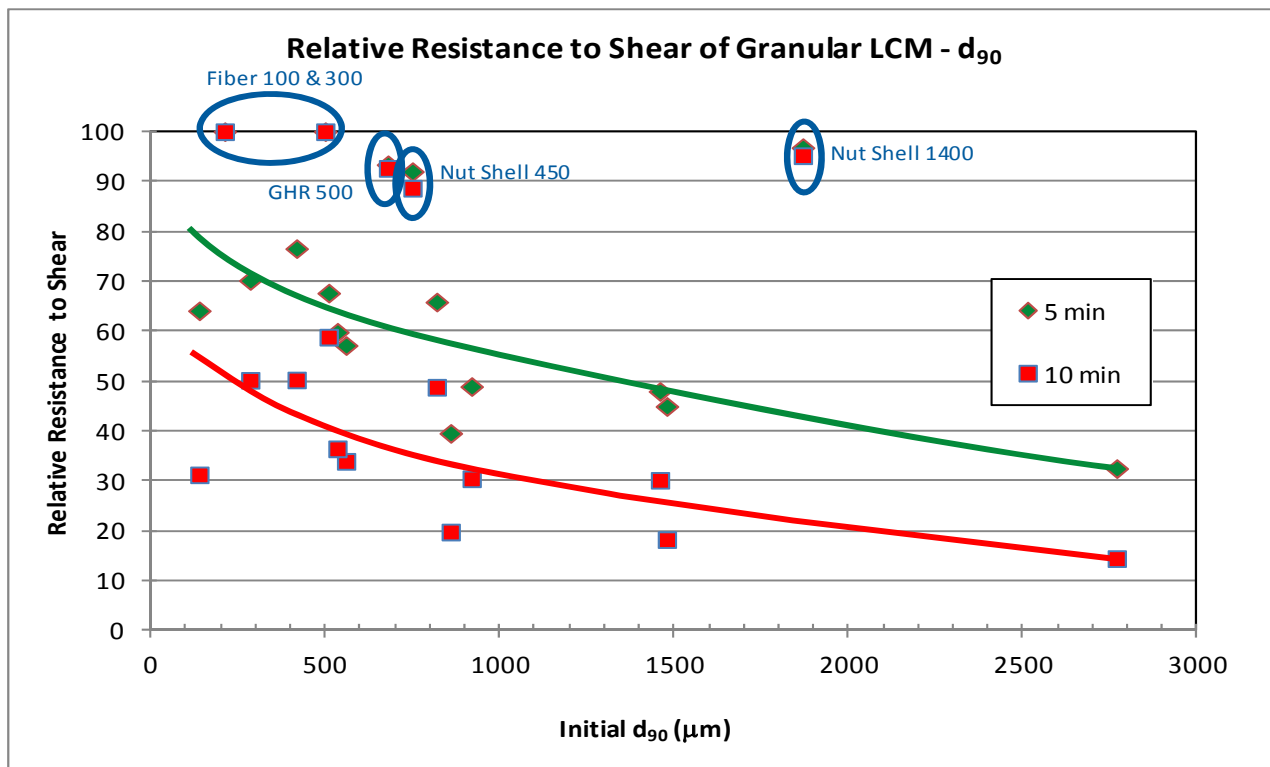
**Figure 2 – Resistance to Shear (effect of shear on  $d_{50}$ ) of Granular LCMs**



**Figure 3 – Resistance to Shear (effect of shear on  $d_{90}$ ) of Granular LCMs**

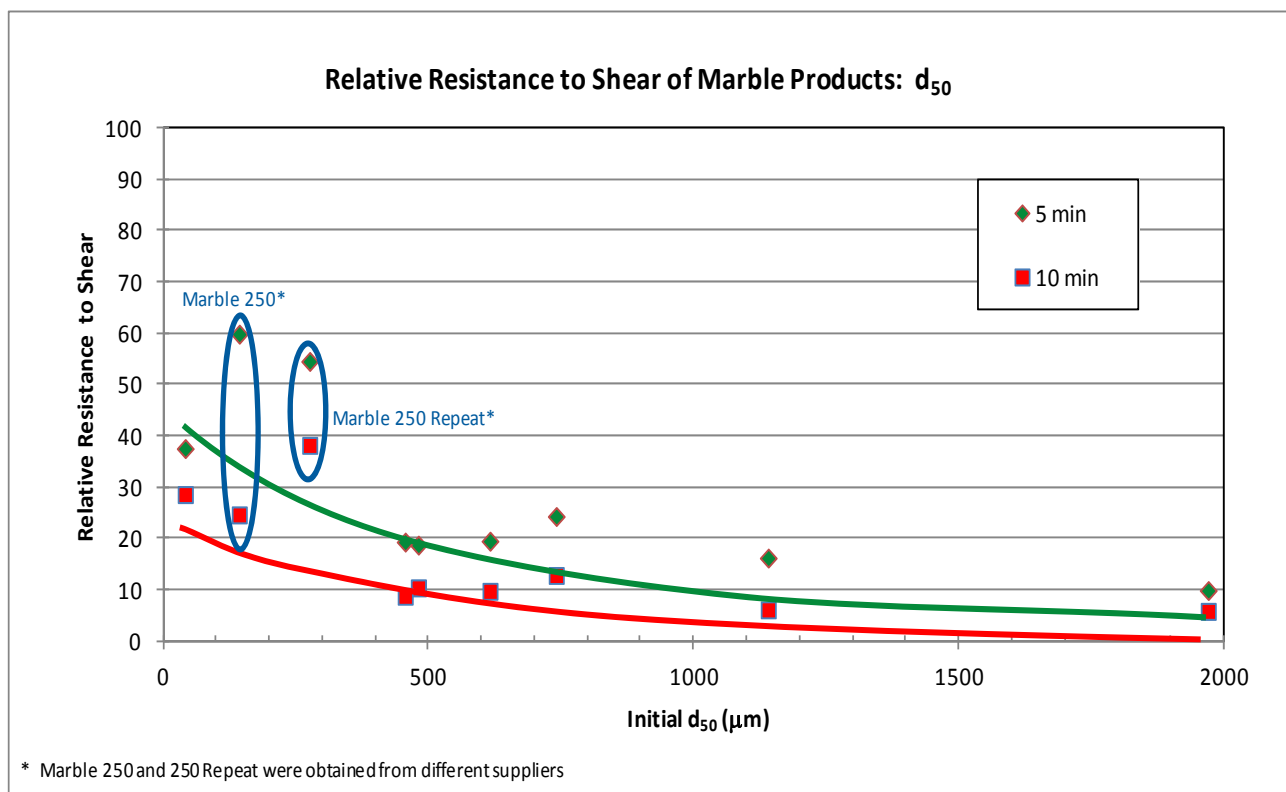


**Figure 4 – Effect of Initial Median Particle Size ( $d_{50}$ ) on Resistance to Shear of Granular LCMs**

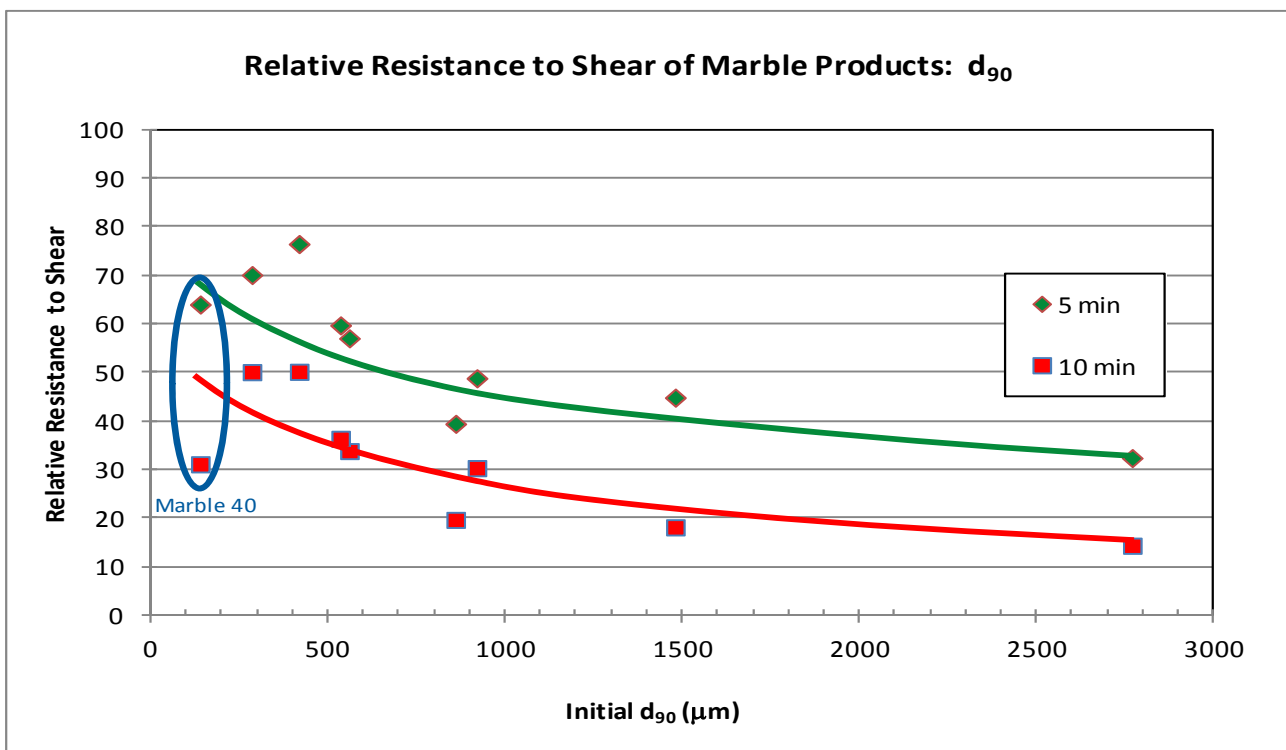


**Figure 5 – Effect of Initial  $d_{90}$  on Resistance to Shear of Granular LCMs**

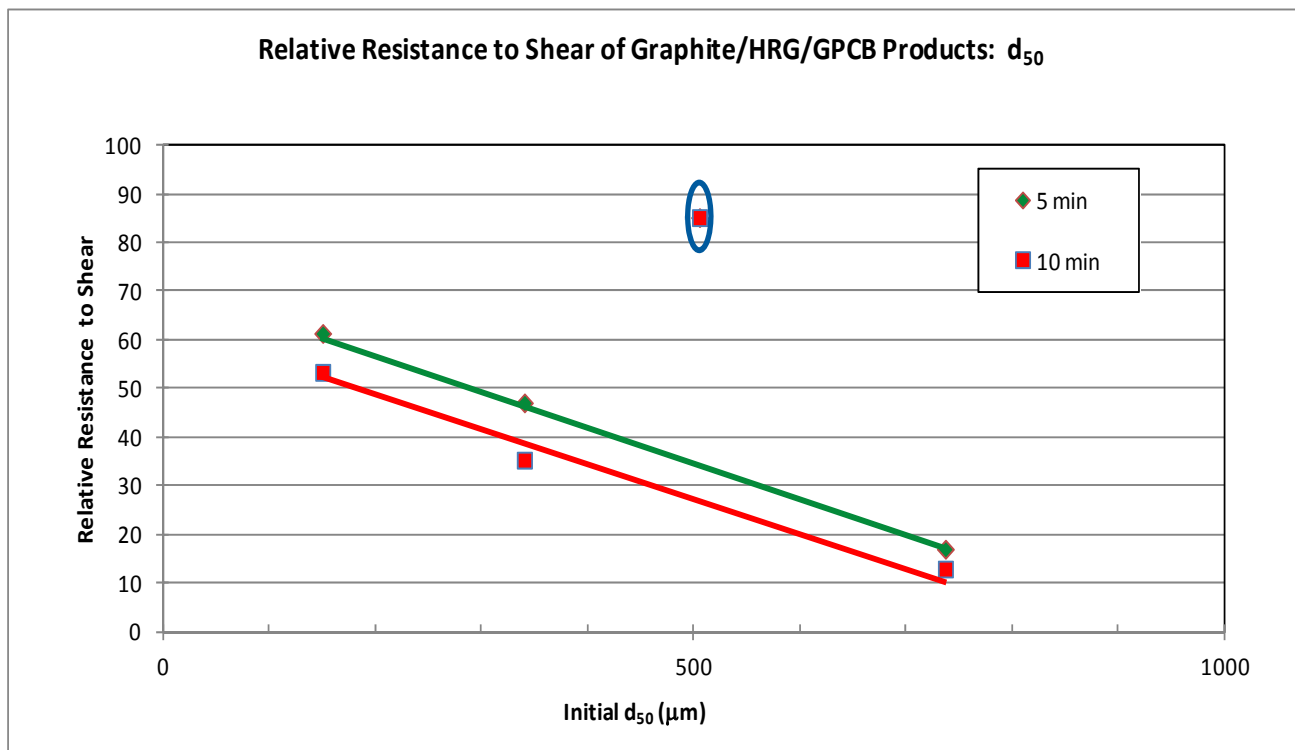




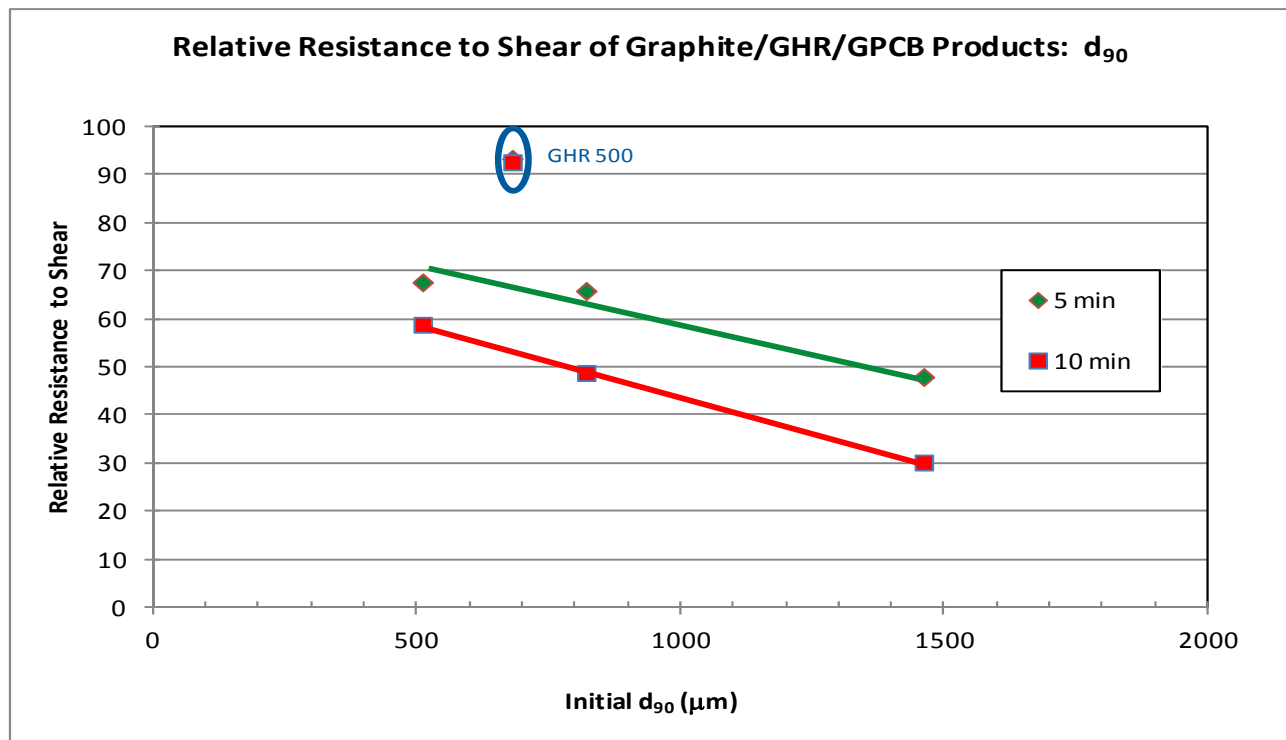
**Figure 6 – Effect of Initial  $d_{50}$  on Resistance to Shear of Marble Products**



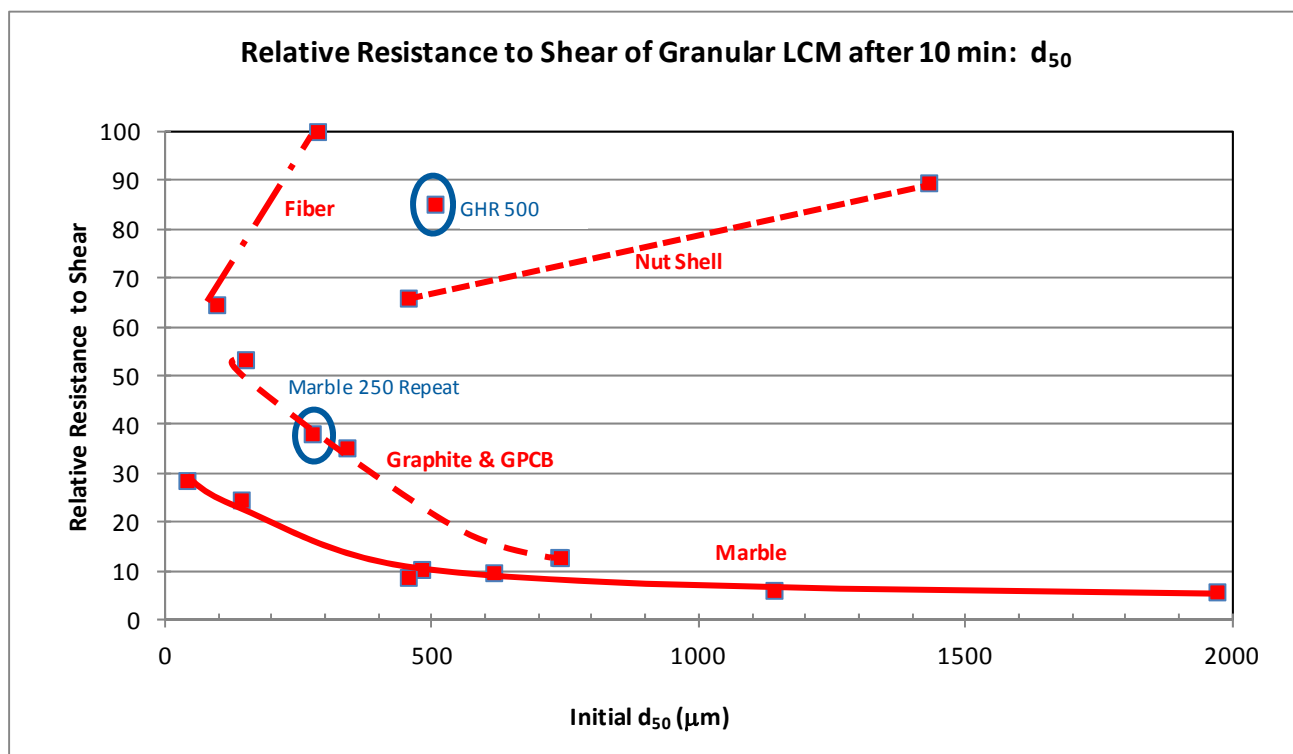
**Figure 7 – Effect of Initial  $d_{90}$  on Resistance to Shear of Marble Products**



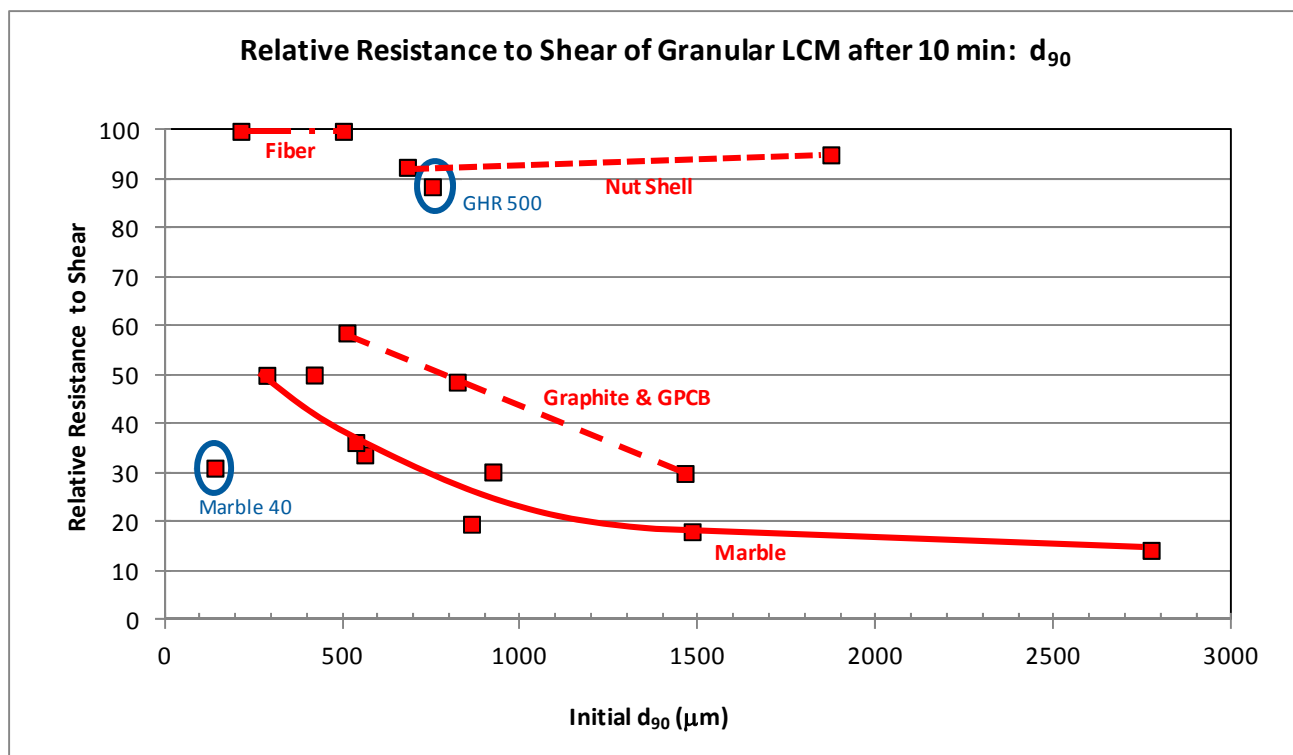
**Figure 8 – Effect of Initial  $d_{50}$  on Resistance to Shear of Graphite Products**



**Figure 9 – Effect of Initial  $d_{90}$  on Resistance to Shear of Graphite Products**



**Figure 10 – Effect of Initial  $d_{50}$  on Resistance to Shear (10 min) of LCM Families of Products**



**Figure 11 – Effect of Initial  $d_{90}$  on Resistance to Shear (10 min) of LCM Families of Products**