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Particle Size Analysis: Leveraging New Techniques Yields Better Data

Sanjit Roy, OFI Testing Equipment Inc., Peter Bouza, Vision Analytical Inc.

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Abstract

Particle analysis is a well-known characterization practice used in many industries to understand raw materials better. Numerous particle analysis techniques have been employed in industry for decades, including drilling fluids and injection water. Most established techniques typically can offer particle size and some can provide concentration. However, most available techniques report particle size, assuming all measured particles are round or spherical. This limitation requires experienced end-user decision-making skills to determine what the particle size distribution statistics mean.

One benefit of improved electronics has been faster computing power, leading to new techniques being developed. One such technique for the analysis of particles has been Dynamic Image Analysis (DIA). DIA, a relatively new technique, relies on high-quality optics and cameras to take high-quality images of particles in motion. Once photos are taken, many shape measurements are calculated for each particle. Having statistics and shape information on each measured particle in real-time offers users a broad range of information never available before. Images of the particles also offer objective evidence of the measured particles.

This paper aims to have a basic overview of the most common particle size techniques used in the Oil and Gas industry, including Dynamic Image Analysis, and recommend how to utilize the reported data of the most common methods to make process decisions.

Introduction

Particle properties, such as size, shape, surface area, or other physical or chemical properties are necessary for accurate particle characterization. Characterization is performed using multiple commercially available techniques to measure particulate samples. Each has strengths and limitations; no universally applicable technique exists for all samples and situations.

Monitoring and controlling particle characterization properties of materials is done because it predicts behavior and performance in many application fields. By measuring these properties, one can better understand, control, and optimize the quality, functionality, and safety of products and processes involving raw material particles.

Although particle characterization is broad ranging, for the scope of this paper, we will focus on particle size analysis, particle shape, and concentration measurements. We will discuss the most common measurements and briefly explain some of the techniques and how they apply to the Oil and Gas industry with a relevant application.

Some measurements and what they represent

Particle size

Particle size is a measurement used to compare the dimensions of solid particles. Measuring particle size for raw materials is common in ensuring adequate quality control. It is also used to identify and differentiate particles. Many standard techniques perform what is known as indirect measurements, whereby the measurement is based on different physical aspects of the particles, not the size.

Such indirect measurements include how the particles scatter light intensity or their volumetric displacement. These are not direct size measurements but are correlated to particle size. These indirect measurements are then used in mathematical calculations where the particle diameter is derived. Such techniques report particle diameter data assuming particles are round or spherical. However, in practice, most industrial particles are non-spherical, and size results assuming they are spherical may miss critical information. In addition, some of these indirect techniques may ignore some particles based on the limitation of the measurement.

Direct measurement techniques on the other hand are number-based and perform measurements one particle at a time. Direct measurement techniques are typically microscopy or automated microscopy, with direct measurements of each particle.

When using particle size as a means of quality control, statistical histograms and data points are used as points of comparison. Particle size histograms are reported and can show the significance of the presence of a specific population of particles in the sample. The more common statistical data points used in industry are the D10, D50, and D90, representing the cumulative % less than particles of the specific size.

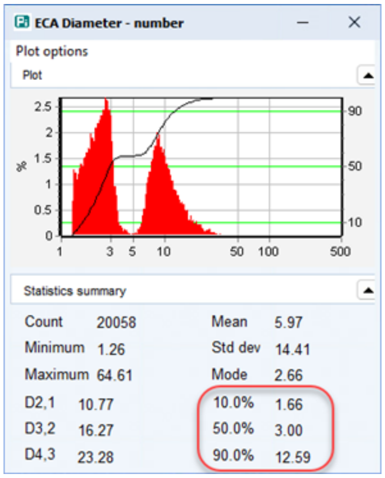


Figure 1 – Representative number-weighted particle size distribution

Particle count and concentration

Automated particle counting was made popular when it became prevalent for counting blood cells using the Coulter Counter. By counting and simultaneously performing a size measurement, the Coulter Counter was able to differentiate blood cell types by size and enumerate them which is important to diagnose patients.

Counting particles and knowing how many of any type of particle exists per unit volume, typically count/ml, has made its way past the medical field into our industry. For example, it may not be a problem if filtered drilling mud has a small presence of fine particles. However, if there is a large presence, it could then alter the flowability of the recycled drilling mud. Knowing the amount of particles present is a count and concentration measurement that can only be done with techniques able to volumetrically count particles. There are many applications where count, not just size, is an important particle characterization factor.

Particle shape

As mentioned earlier, not all particles are spherical. In fact, few are. It is also commonly known that particle shape has a direct impact on flowability, viscosity, and compaction of materials. Some think that particle shape analysis is as simple as looking at some representative images of particles. Particle images serve as good objective evidence of what your particles look like, but the ability to make many shape calculations of every particle and measuring concentration awards the user with the ability to use this information to identify issues where size-only instruments can't. In many cases, differences in shape, not size, were used to identify different lots of raw materials. Particle shape also allows users to detect and identify large particles as either bubbles, agglomerates, or contamination by using thumbnail images as evidence.

Automated particle shape analysis is a direct measurement technique performed with either dynamic or static imaging of particles. Being a number-based technique, particle shape analysis is designed to measure all particles that pass through the field of view of an image-capturing device. Commercial instrumentation for analysis of particle shape has advanced over the last 15 years due to the availability of faster computers. Automated particle shape analysis is still considered a relatively new technique that is still evolving.

Overview of popular techniques.

For this paper, we will limit the technique overview to the most common method used in the Oil and Gas industry along with Dynamic Image Analysis (DIA) which is a relatively new technique we are focusing on in this paper.

Laser Diffraction

Laser diffraction is a technique that uses a laser beam to measure the size distribution of particles in a sample. The principle, shown in Figure 1, relies on the fact that particles of different sizes scatter light at different angles. By measuring the intensity of the scattered light at various angles, a particle size distribution can be calculated using a mathematical model.

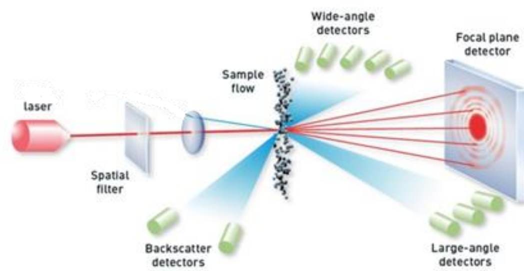


Figure 2 – Laser diffraction technique

Several physical aspects are needed to perform proper analysis using laser diffraction, including the refractive index of the particles and that of the suspension medium for the particles. These are sometimes difficult to ascertain.

Laser diffraction instrumentation can measure a wide range of particle sizes. Analysis time is fast and repeatable. However, one thing to remember is that smaller particles in the presence of larger particles are hard to detect due to their weak scattering intensity and their inability to be represented adequately in a volume-based reporting system. Also, low concentrations of particles are difficult to detect and measure due to weak signal, as are ultra-high concentrations, due to multiple scattering effects. In addition, laser diffraction will also report the size of particles assuming they are spherical regardless of their actual shape, and as we show later, few particles actually are.

Dynamic Image Analysis

Particle shape instrumentation uses Image Analysis and is either Static or Dynamic in operation. Static systems are automated microscopes that offer excellent image quality but take significant time to get representative results of many particles. Dynamic Image Analysis (DIA) systems enable the simultaneous analysis of particles while in motion, allowing the characterization of tens of thousands of particles in minutes and giving statistically representative results in a short time. It also allows detecting and characterizing rare events such as agglomerates or low quantity contamination with thumbnail images as objective evidence. Dynamic image analyzers are also flexible enough to be added as a complementary method to other measurement techniques. In addition, DIA systems can be adapted to any on-line process (Canty and Dalton, 2022).

Dynamic image analysis is a number-based technique where particles are suspended past an imaging zone, pictures are taken at high-speed, and the particles in those images are analyzed. This means particles are measured individually. There are a few other techniques in the industry that are also number-based and very suitable to give individual particle information as well as count and concentration information, which is very important. In addition, dynamic image analysis is a direct measurement technique. This means that measurements are being made of the actual particle.

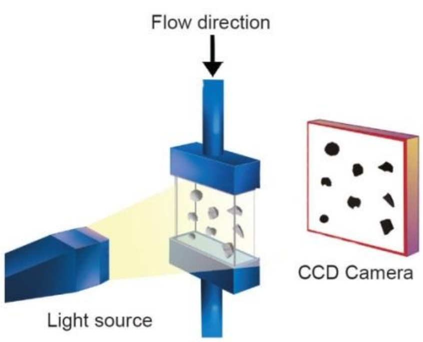


Figure 3 – Dynamic Image Analysis schematics

The ability to measure and detect individual particles and give dozens of shape measurements for every particle, including size, are a few reasons why dynamic image and particle shape analysis are becoming increasingly popular. The vast amount of data obtained for each particle is then used to classify particles and do further post-analysis processing. More of the available tools will be discussed further in the practical section of this paper.

To gain a better understanding of how dynamic image analysis works, please scan this QR-code with your phone's camera to view a live analysis of LCM.

Figure 4 – scan with your camera to see LCM video

Dynamic Image Analysis Instrumentation

Figure 5 – Picture of Dynamic Image Analyzer

Figure 5 shows an example of a dynamic image analyzer manufactured by Vision Analytical. Designed for the Oil and Gas industry, systems typically are in ruggedized cases and can operate with battery-backed power and even 5G connectivity. Systems are designed with simple interchangeable sample cells to ensure uninterrupted operation. Samples can be suspended using an external vessel and circulated through the sample cell for continuous analyses for extended periods. Samples can also be analyzed directly with collection devices such as syringes or can even be adapted to work in-line. A tablet PC is built-in for data collection, analysis and reporting purposes.

Drilling Fluid Particle Size Analysis

Knowledge of particle size and shape is of vital importance to optimally design drilling fluids. The source of particles in the fluid are either due to the drilling process itself (cuttings) or as a result of addition of products such as lost circulation or loss prevention material (LCM/LPM). Particulates added to the drilling fluid could have beneficial effects, such as encouraging the formation of filter cake on the wellbore walls to minimize fluid loss, or plugging or bridging any fractures that may be created during the drilling process. In some cases, particulates have detrimental effects, such as formation damage, excessive solids-handling at the surface, or higher fluid viscosities, etc.

Wellbore fractures could be a result of naturally occurring fractures, un-avoidable as a result of drilling, un-intentional such as excessive bottomhole pressures, intentional such as during wellbore strengthening, or any combination of the above. Particles in the fluid play an important role in preventing un-controlled growth of fractures and subsequent loss of drilling fluid to the formation.

Designing the optimum blend of loss prevention materials to minimize fluid loss into the formation depends on the size of pore spaces and/or fracture width. Various rules-based criteria, such as Ideal Packing Theory (Dick et.al., 2000, Kaeuffer, 1973), Abrams Rule (Abrams, 1977), Vickers method (Vickers et.al., 2006) etc. are commonly utilized to design optimum particle size blends. These methods rely on an optimum blend of LPM products to generate a “target” PSD. Various size measures, such as D10, D50, D90 are utilized to determine goodness of fit between the desired target and optimum blend.

The process of designing the blend and ensuring the blend matches the desired PSD relies on accurate PSD measurement on the base products as well as the final drilling fluid with the products. Currently, drilling fluid engineers on location usually have only a limited set of equipment to determine the particle size distribution of the fluid. Usually, empirical criteria on number of sacks of specific products in a given period of time is used to manage PSD during the drilling process. In some cases, manual sieve analysis of drilling fluid is utilized, but those are labor intensive, time consuming, and prone to errors such as due to product agglomeration and clumping.

In critical jobs, periodically drilling fluid samples are sent to analytical laboratories for detailed PSD analysis using any of the techniques detailed above. The associated time delay negates the value since current conditions may be totally different than when the sample was taken. The portable Dynamic Imaging tool provides a powerful and flexible alternative to rapidly determine, at the wellsite itself, the current state of the drilling fluid to determine in-time treatment necessary to achieve the optimum blend.

The following section describes measurements made on samples of oil-based drilling fluid (OBM) both with and without LPM. Measurements were performed with varying concentrations of LPM and with different types of LPM of different shapes and sizes.

Drilling Fluid with Varying Concentrations

Field Oil-Based Mud (OBM) sample was collected from a an onshore well in Texas, USA. This base drilling fluid did not contain a significant amount of LPM. The base sample was divided into 5 parts, 4 of which were blended with various concentrations of a blend of LPM. Table 1 shows the sample designations and their concentrations. All samples with LPM were blended with LPM mix of (by weight) 40% carbonate, 30% Fiber, and 30% Walnut shells. Table 1 also lists the various distribution values (“D” values) commonly used to designate particulate sizes. All fluid samples were suspended and diluted with 99% Isopropyl Alcohol (IPA). About 5 mL of the oil-based drilling fluid was agitated in a small container with the IPA before it was introduced in the circulating fluid reservoir of the device. Circulation via the built-in pump ensured the sample stayed suspended during analysis.

Fluid	Total LPM Conc (ppb)	D10 (µm)	D50 (µm)	D90 (µm)
Base	-	8.61	14.78	28.83
Base+10	10	9.30	19.05	43.94
Base+15	15	9.24	19.26	61.14
Base+20	20	8.74	17.01	39.89
Base+25	25	8.98	18.09	37.29

Table 1 – Fluid designations, LPM concentrations, and various distribution values for 5 oil-based drilling fluids with varying LPM concentrations

Fig. 6 shows the particle size distribution for the 5 fluid samples listed in Table 1. The solid line shows the PSD of the base OBM drilling fluid, while the various dashed lines show the PSD of fluids blended with varying LPM concentrations.

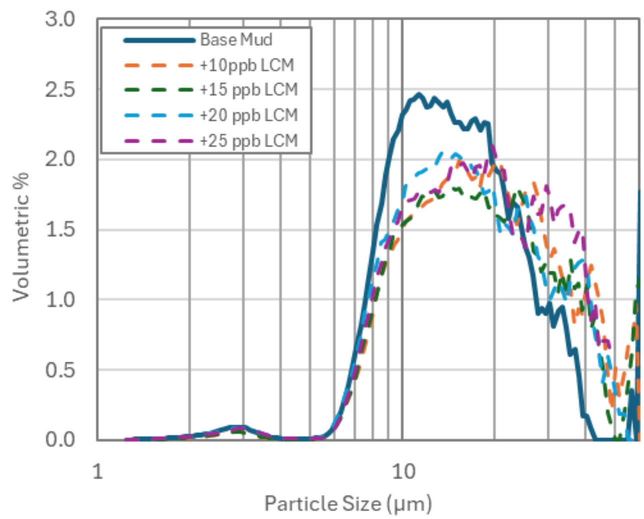


Fig. 6 – Volume-weighted particle size distribution of oil-based drilling fluid with varying LPM concentrations

Several features can be noted comparing the PSD of the base sample with the PSD of the samples with LPM. The base sample had a larger volumetric % of smaller-sized particles, while the ones with LPM had more larger-sized particles than the base sample. The PSD with the varying LPM concentrations were similar to each other since the same blend was added to all the samples. Also, all 5 samples had a smaller peak at around 3 microns. All samples in this group had a similar distribution in this range, and closer examination of the particle thumbnails (shown later in Figures 10,16) indicate that these were solid particles and not emulsion droplets. The fluid sample was collected from the field and these fine particles are drill solids (or barite) present in the fluid. While emulsion and droplet analysis is possible since particles in the 1-5 µm range are clearly observed, that topic will be discussed at a later date.

Figure 7 shows results from the same samples, but this figure shows the number-weighted distribution. The characteristic shape of the number-weighted distribution is different than the volume-weighted distribution. The number-weighted distribution shows a much higher numeric percentage of particles at the smaller size ranges, and the peak at around 2.54 µm exceeds the peak at around 9 µm. These smaller particles do not have much volumetric contribution, but numerically they are significantly higher. These results imply that fines introduced during drilling remain in the fluid.

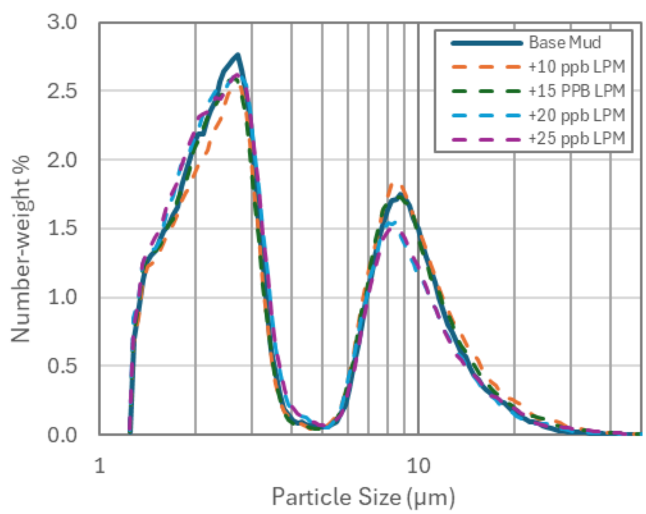


Figure 7 – Number-weighted particle size distribution of oil-based drilling fluid with varying LPM concentrations

The number-weighted distribution shows that all 5 samples (base + different LPM concentrations), as expected, have very similar distributions. The same blend of LPM at different concentrations was added to the same base drilling fluid. The dynamic image analysis clearly indicated differences between the base fluid and those with LPM. The closeness of the distribution in both volume and number-weighted analysis demonstrates instrument repeatability and consistency.

Drilling Fluid with Different Types of LPM

A series of tests were performed on a Field Oil-Based Mud (OBM) sample was collected from a an onshore well in Texas, USA. The base sample did not contain a lot of LPM. A sample of drilling fluid was divided into 5 parts, and different types of LPM were added to 4 of the samples. In all cases, a concentration of 20 ppb (by weight) LPM was added. Four different LPM were selected- calcium carbonate, walnut shells, cedar fibers, and a synthetic graphite blend. Each sample was pre-mixed thoroughly before imaging analysis was performed on each sample. Approximately 5 mL of each sample was diluted with 99% Isopropyl Alcohol (IPA) for the imaging analysis. Table 2 lists the various samples, and the corresponding distribution values from the image-based analysis.

Fluid and LPM type	D10 (µm)	D50 (µm)	D90 (µm)
Base	8.77	14.90	29.94
Base+20 ppb Nut Shell	9.54	19.81	41.87
Base+20 ppb Carbonate	10.41	23.53	48.68
Base+20 ppb Cedar	8.16	13.84	25.93
Base+20 ppb Synthetic Graphite	10.27	24.03	63.55

Table 2 – Fluid designations, various LPM types, and various distribution values for 5 oil-based drilling fluids

Figure 8 shows the particle size distribution for the 5 fluid samples listed in Table 2. The solid line shows the PSD of the base OBM drilling fluid, while the various dashed lines show the PSD of the fluids blended with different types of LPM.

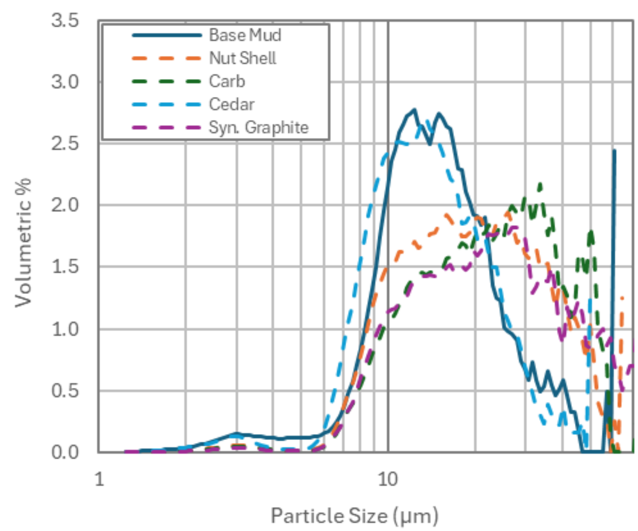


Figure 8 – Volume-weighted particle size distribution of oil-based drilling fluid with different types of LPM

Several features are immediately evident comparing the PSD with different LPM. The base drilling fluid and the sample mixed with 20 ppb Cedar fibers had very similar size distributions, while the other products had higher volumetric proportion of larger sized particles. These are reflected in the distribution values, for example the D50 of the base fluid and the one with Cedar fiber were close, while the carbonate and the graphite had higher D50 values.

Figure 9 shows results from the same samples, but this figure shows the number-weighted distribution. The characteristic shape of the number-weighted distribution is different than the volume-weighted distribution. Similar to the previous set, number-weighted distribution shows a much higher numeric percentage of particles at the smaller size ranges, and the peak at around 2.88 µm exceeds the peak at around 9-10 µm. These smaller particles do not have much volumetric contribution, but numerically they are significantly higher. While the sample with the Cedar fiber had a similar peak as the base sample at around 2.88 µm, there were a higher concentration (number) of larger particles in the sample with Cedar. The concentration (number) of larger particles was higher in the samples with Nutshell and Synthetic Graphite.

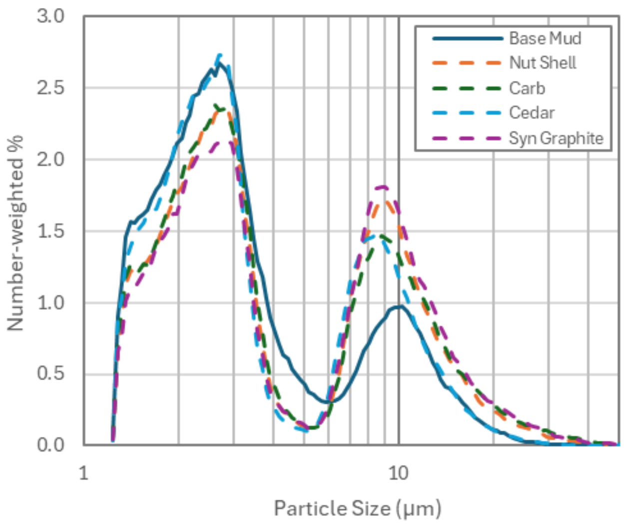


Figure 9 – Number-weighted particle size distribution of oil-based drilling fluid with different types of LPM

Practical Sample Analysis and Results

The sample was suspended and diluted with 99% Isopropyl Alcohol (IPA). This was needed to minimize presence of water given that the drilling mud was oil based. If water was present, it would appear as spherical droplets in the sample. This would impact indirect measurement techniques given that they cannot differentiate between a droplet or a particle. Direct measurement techniques such as DIA can identify bubbles / droplets and eliminate them from the results (Hoxha et.al., 2016, Canty and Dalton, 2022).

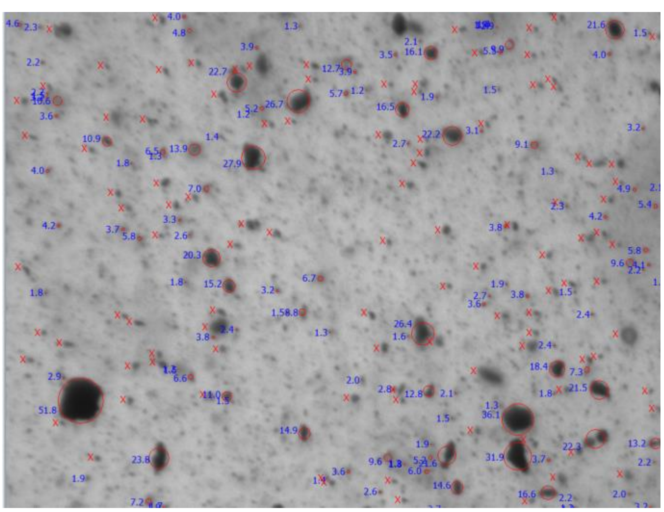


Figure 10 – drilling mud suspended in 99% IPA. Note presence of larger particles and significant amount of small particles

In Figure 10 we show an image of the sample. As can be seen, there are larger particles, however there are small particles as well. This image was taken using a DIA system during analysis.

Laser Diffraction Results

As shown in Figure 11, laser diffraction results, also known as the D[4,3] resulted in a diameter of 59.4µm. As mentioned before, the D10, D50 and D90 are shown below which is the size point at which the cumulative 10%, 50%, and 90% takes place. These additional values are commonly used to attain known size points for fines as well as the larger portion of the size distribution. Keep in mind, being an indirect measurement, this technique will report size assuming the particles are spherical or round in shape.

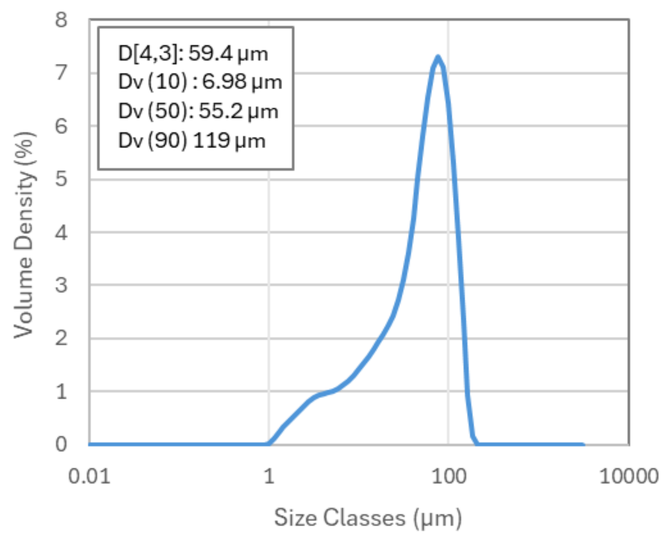


Figure 11 – Laser diffraction results

Figure 11 shows the histogram results of the analyzed sample as well as the statistical results from a laser diffraction system. As can be seen from this histogram, there is a large population of particles that are in the 90-100 µm range. Also, it can be seen that there is a small amount of samples found in the 1 to 10µm range. None the less, the significant area of particles are in the 55µm range.

Dynamic Image Analysis Results

The exact same sample, and same aliquot, was analyzed using a dynamic image analyzer. This is a direct measurement technique.

Figure 12 shows the volume weighted distribution of the sample using DIA. Although both Laser Diffraction and Dynamic Image Analysis report volume weighted size distributions, the statistical results are slightly different. The reason for this discrepancy will be discussed later.

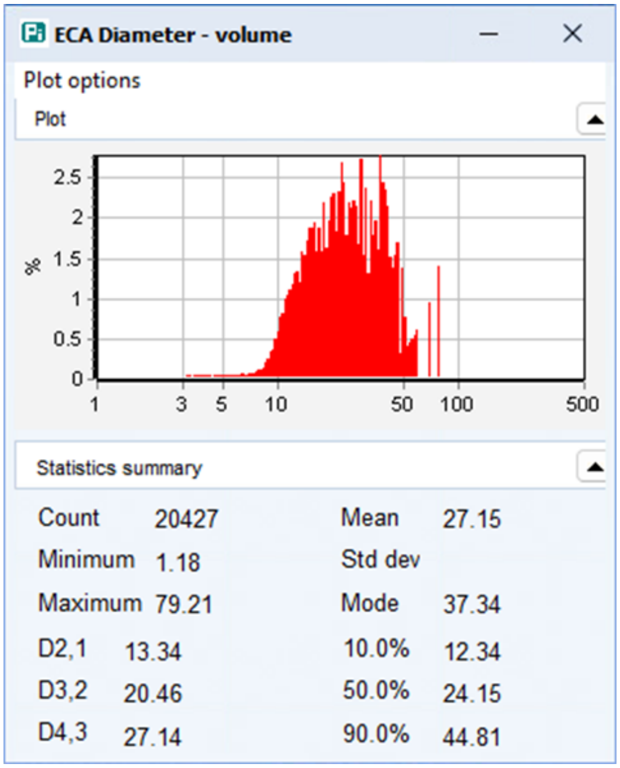


Figure 12 – Volume-weighted distributions

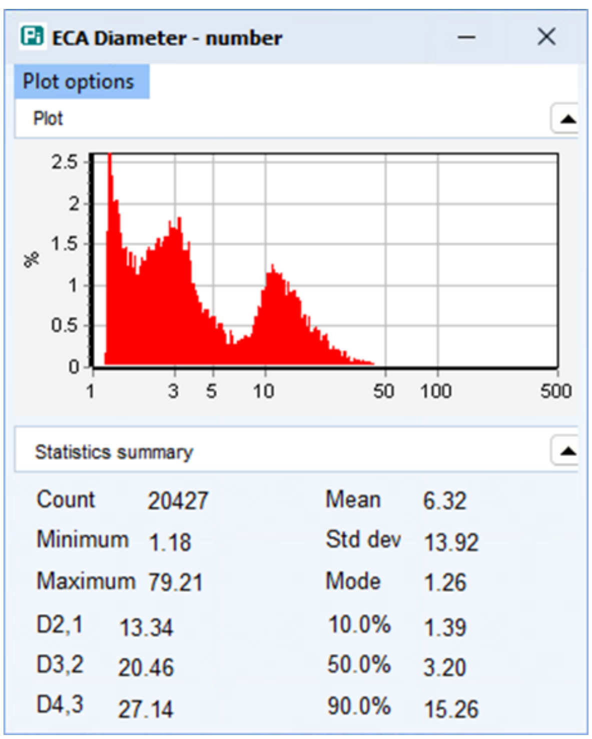


Figure 13 – Number-weighted distribution

DIA, being a number-based technique that performs analysis of each individual particle, enables reporting a number-weighted distribution of the same sample. Figure 13 shows the number-weighted distribution of this same sample. The statistics are different; however, the importance of this graph is to show the significantly high presence of small particles. This is something that was not evident in the laser diffraction results. The reason laser diffraction is unable to report the presence of these small particles is due to the fact that the scattering light of the larger particles dominate over the weak scattering of the smaller particles at higher angles. Dynamic image analysis is able to report small particles properly which can be important to your process.

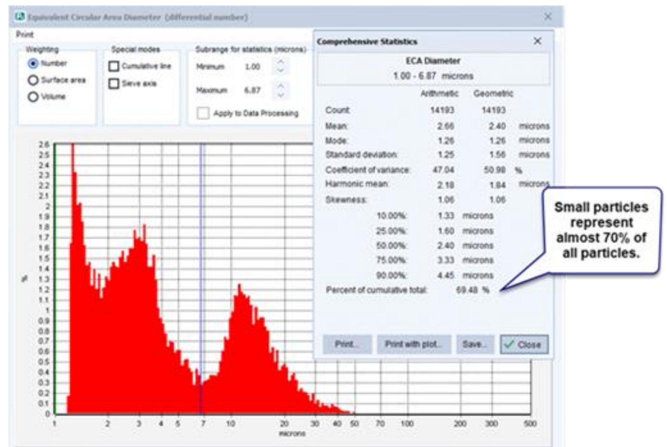


Figure 14 – DIA capable of reporting percentage of select populations in a sample

As can be seen from Figure 14, the smaller particles in the sample represent almost 70% of the entire distribution. This high presence of small particles can influence viscosity of the drilling mud. It is very important to use a technique that can properly represent the entire distribution of particles in your sample. Because dynamic analysis is a number based technique it is capable of counting particles and reporting the percentage of any sub population in your sample. Dynamic image analysis systems can also report concentration in particles per milliliter of analyzed sample.

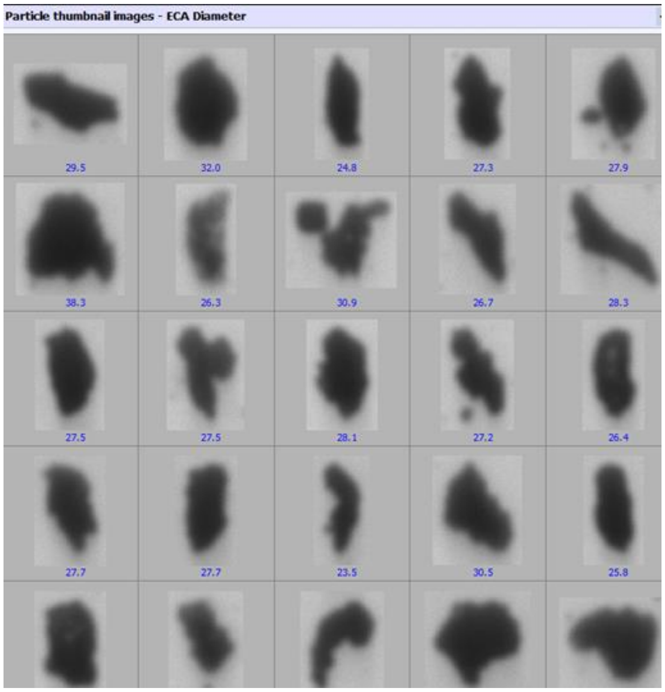


Figure 15 – Images of larger particles in sample

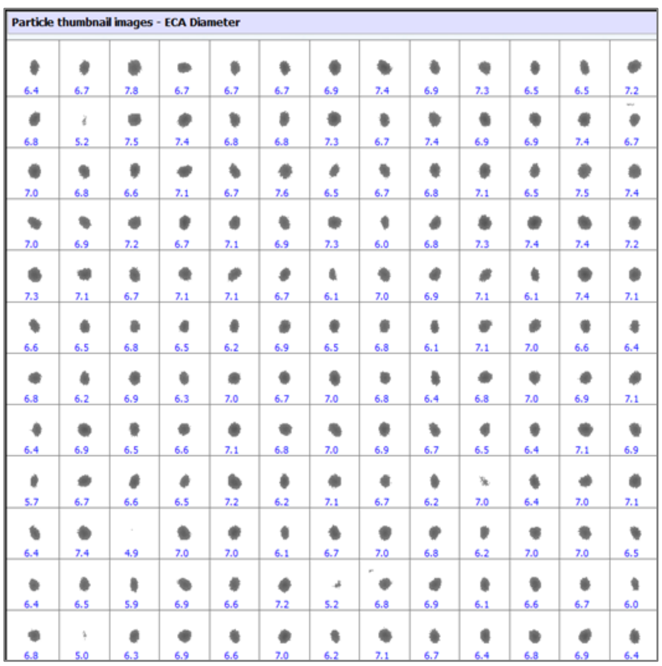
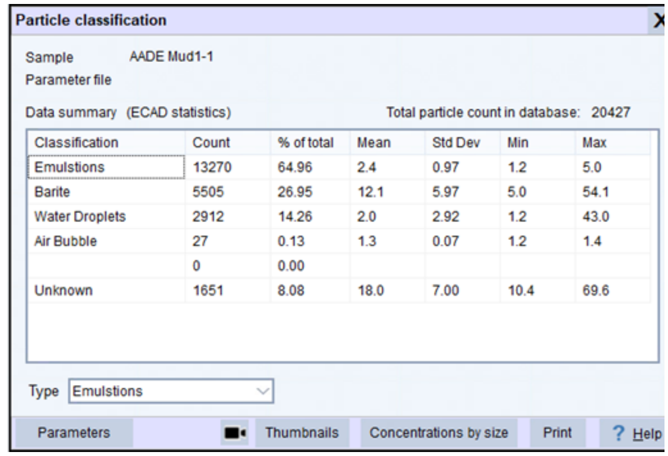


Figure 16 – Images of smaller particles in sample

Because this technique uses a camera, images of all the analyzed samples are stored. They are very useful as objective evidence that these particles do exist and what they look like. Figure 15 shows sample thumbnails of the largest particles in the sample. Note the elongated shape of most of the samples, which tend to over-estimate the size in laser-diffraction based systems which model the particles as spherical-shaped. Figure 16 shows representative thumbnails of smaller-sized particles.



Classification	Count	% of total	Mean	Std Dev	Min	Max
Emulstions	13270	64.96	2.4	0.97	1.2	5.0
Barite	5505	26.95	12.1	5.97	5.0	54.1
Water Droplets	2912	14.26	2.0	2.92	1.2	43.0
Air Bubble	27	0.13	1.3	0.07	1.2	1.4
	0	0.00				
Unknown	1651	8.08	18.0	7.00	10.4	69.6

Figure 17 – Ability to classify particles into sub-categories

Dynamic image analysis makes over 32 shape calculations for every particle. Having an abundance of information on every particle allows this technique to classify subpopulations of particles in a sample. In figure 17 shown above we are able to create subclassifications of particles by using one or several shape parameters for each category. In addition to having statistics such as what percentage of each subpopulation is in your sample, users also have the ability to view the thumbnail images of each classification as objective proof.

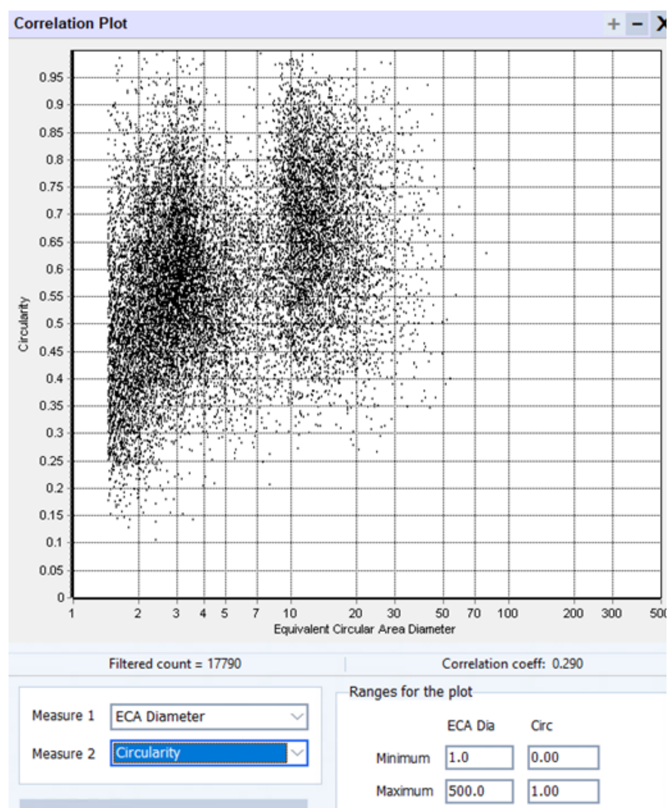


Figure 18 – Correlation Plots for finding rare event particles

Another available tool with dynamic image analysis is the correlation of particles. This tool allows users to create a scatter plot of all the analyzed particles. The plot uses any two available shape measures analyzed. This tool is very useful in identifying rare events. These can be identified easily with the different dots on the scatter plot and can be important particles such as water droplets or machine wear. As shown in Figure 18, this tool makes it easy to identify one particle in almost 18,000 analyzed particles. Note the circularity of most of the smaller particles was around 0.5-0.65, while the larger particles range from 0.6-0.8. Very few are have circularity greater than 0.9.

Differences Explained

There are two main differences in statistics that should be discussed. One difference is the variation between volume based statistics on both laser diffraction and dynamic image analysis. As shown above dynamic image analysis will report equivalent circular area diameter (a shape measure that assumes round particles) slightly smaller than equivalent spherical diameter from the laser diffraction technique which assumes the particles are spherical. Both are diameter measurements. However, when particles are irregular in shape laser diffraction will always tend to report higher results than that of any number based instrument such as dynamic image analysis. Neither technique is wrong. However, because they are different techniques it is important to understand that irregular particles will have slightly different diameter results.

The second item that needs to be explained is why dynamic image analysis was able to see the significant presence of small particles when laser diffraction was unable to detect them. Because the light energy recorded by the detector array is proportional to the volume of the particles, laser diffraction results are intrinsically volume weighted. This means that the particle size distribution represents the volume of particle material in the different size classes. The diffracted light is proportional to the particle's volume which implies that results assume particle sphericity. i.e. that the particle size result is an equivalent spherical diameter. Small particles scatter light weakly at higher angles making it harder to detect using a volume-based measurement technique.

Conclusions

Particle characterization has been an important factor in monitoring raw materials as well as investigating used materials. As technology evolved, better computing and machine vision cameras have made their way to the particle characterization world with Dynamic Image Analysis. This new technology has enabled industries to better monitor and understand their raw materials by offering not only particle size, but also particle shape, concentration, count, and classification of particles by shape. We have shown that Number-Based techniques enable the ability to analyze particles where other techniques fall short. This new technique also warrants the ability to bring lab quality measurement tools to the wellsite as well as in-line measurements for in-time analysis.

Users looking for particle shape systems typically understand particle size but feel they are missing some key information that's having an impact on their final product.

These users are looking for more information and tools to make critical decisions based on shape, not just size. Particle images alone are not enough. Size-only instruments give one shape parameter, that being size. Image analysis systems provide over 32 shape parameters. It is not the user's responsibility to figure out how to use these 32 shape parameters. It is the responsibility of the instrument manufacturer to develop tools that take advantage of all of this information and offer tools such as particle classification, particle correlation, particle concentration, shape overlays, and many more. High-quality images are always important, but post-run-processing tools create the most significant value in shape analysis.

Since technology will continue to evolve, we will stay waiting to see what other techniques emerge and leave a foot print in the world of particle characterization.

Acknowledgments

The authors will like to thank Harry Dearing and Erik Rios with Genesis Fluids for providing samples for testing. The contributions of Bryan Klumpyan and Tyrone Gonzales for help with testing is also gratefully acknowledged.

Nomenclature

DIA = *Dynamic Image Analysis*
PSA = *Particle Size Analysis*
ECAD = *Equivalent Circular Area Diameter, particle size measurement assuming particle is round (2-dimentional)*
Circularity = *Size independent shape measurement of a particle ranging from 0 to 1 where 1 is perfectly round.*

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