Method Improvement on Particle Size Distribution Analysis of Mature Fine Tailings by Laser Diffraction

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Summary

Subsampling of the oilsands and mature fine tailings (MFTs) specimens are usually performed on solid powder using either static or rotary riffles for laser diffraction particle size distribution (LD-PSD) analysis[1]. The required mass of a subsample for the laser diffraction analysis is dependent on the volume of the instrument's recirculator as well as the quantity of fines and clay size particles of the subsample being analyzed. The low volume of the recirculator and high content of clay size particles dictate less quantity of the subsample for the analysis. However, the riffles lose the capability to produce appropriate repeatability as a result of lowered justified representation of the quantity of the sample. In addition, obtaining extremely low quantities of subsample is laborious and time-intensive. This problem is more pronounced for MFTs where the fraction of clays is substantially higher compared to oilsand samples. Our in-house study on several types of MFTs revealed that the precision of findings on cumulative volumes of particles < 44 microns can be greatly improved by subsampling a larger portion of the slurry MFTs using a Lightnin impeller to homogenize the slurry at optimized conditions. The relative standard deviation percentage (%RSD) was reduced between 2-6 times for several different MFT samples, thereby demonstrating the advantage of slurry over solid subsampling for MFTs.

Introduction

Particle Size Distribution (PSD) is a fundamental physical property of sediments and rocks. It is an important indicator of quality and performance of a material. PSD is also an important parameter in the petroleum industry which affects the flow rate of hydrocarbons, recovery, quality of emulsions, processability of the extraction practices and management of tailing deposits, especially the mature fine tailings (MFTs)[1]. There are a number of methods used for PSD determination e.g., Sieve Analysis, Sedimentation Techniques (Hydrometer Method, Pipette Method, and Centrifuge Method), Image Analysis, Microscopic Techniques (Optical Microscopy, Transmission Electron Microscopy and Scanning Electron Microscopy), and Laser Diffraction. Each method has its inherent limitations, mainly due to the difficulty in defining the size of irregularly shaped particles in general and specially in the heterogeneous composition of samples common to the oilsands industry. Main advantages of the Laser Diffraction technique for PSD determination include: short time of analysis (in the order of minutes), high repeatability, small sample size (<1g), and a wide range of size fractions into which the entire range of particle sizes can be divided[2,3].

Sample Preparation and Analysis

Initially, the MFT Samples were cleaned to remove bitumen and water from the solids as the bitumen will interfere with the particle size determination. The Dean & Stark extraction method was used to clean the solid using toluene as the solvent. The reflux were continued for over 12h to ensure no residue of organics remained in the solid. Further, samples were dried at room temperature and subsequently in an oven to remove excess of toluene. The consolidated dry solid samples were disintegrated using a Hammer-Mill as the solid forms a hard clump due to the compaction of fines and clays, as well as from

the heating process used to remove the excess of toluene. XRD analysis on several different MFT samples showed that quartz, feldspars, mica/illite, and kaolinite are the major constituents followed by minor amount of siderite and pyrite.

A Beckman Coulter LS 13 320 Laser Diffraction Particle Size Analyzer was used in this study. Its measuring range is 0.4μm to 2000μm. With its PIDS (Polarization Intensity Differential Scattering) assembly, lower size limit can be extended to as low as 0.04μm. The optical bench configuration is normally equipped with a visual indicator for the proper range of a solid sample. When the obscuration is between the 8-12%, the reading is considered reliable. Below or above this range different analytical errors occur and hence reading is erroneous. Therefore, it is critical to select the proper amount of sample within the recommended range. For this comparison and statistical study, a calibrated static riffle (Sepor model) was used to divide 10 separate subsample's mass (0.08-0.11 g) for each representative MFT specimen. An appropriate amount of isopropyl alcohol was added to each sample followed by the addition of a dispersing reagent (sodium bicarbonate buffer, pH= 9.6). Samples were left overnight at room temperature, boiled for 20 minutes (while stirring) and sonicated for extra 20 minutes at a mild sonication power (60 W probe power of a sonication bath) prior to the introduction of each sample to the LD-PSD analyzer. A larger mass portion of each MFT (1.0-1.2 g) was also subsampled for the slurry subsampling method. The slurry samples (˜3L) were subjected to the same procedure, the subsampling was performed by a syringe with a long needle positioned at the impeller level after the boiling process was complete. 10 subsamples were taken and sonicated for 20 minutes at the same condition as explained above and were introduced sequentially to the LD-PSD analyzer.

Result and Discussion

Graph 1 presents the cumulative volume passing percentage of a MFT sample using two different methods including the static riffle and impeller method for the subsampling. The error bars show the error of subsampling for the 10 subsampled specimens in each method. As is visually apparent, the error bars are larger across all particle size ranges in the riffle method (left). For the cumulative volume below 44 μm, the average ± Standard Deviation (SD) is 88.85±0.54 for the impeller method and 88.60±3.08 for the static riffle method. In fact, the precision of the subsampling is improved significantly in this case as the SD lowered nearly 6 times due to the better subsampling method. However, as the amount of clay-sized contents increase in MFT samples, this effect becomes less significant since the matrix becomes more homogenous and hence subsampling error is minimal. For instance, in a MFT sample with over 99% below 44 μm, the SDs are 0.2 and 0.4 for the impeller and the riffle respectively.
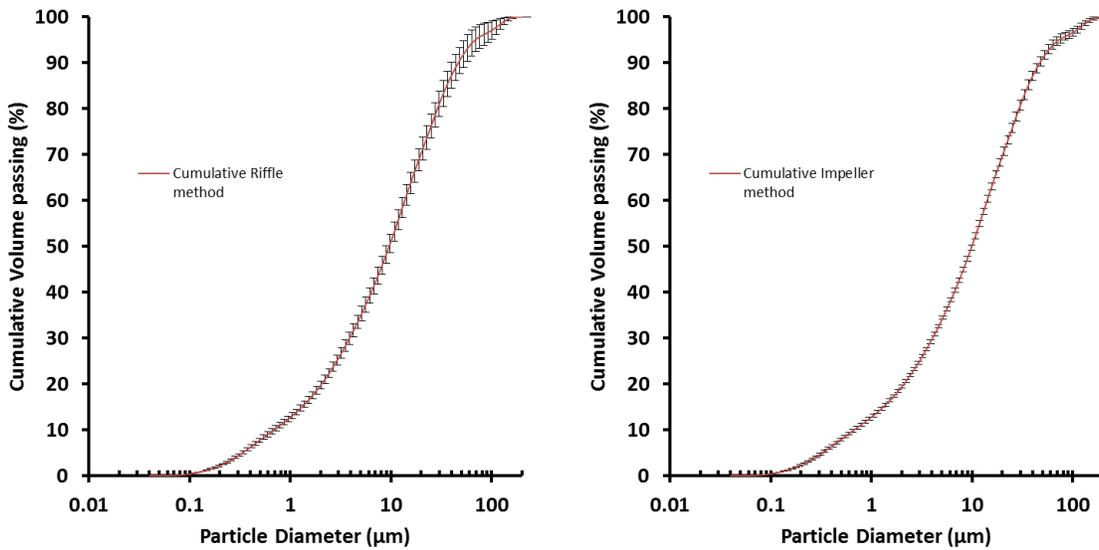
Graph 1. Right and left show the cumulative volume percentages of impeller and riffle methods respectively. Findings demonstrate the error of subsampling is much larger in the riffle method than impeller method.
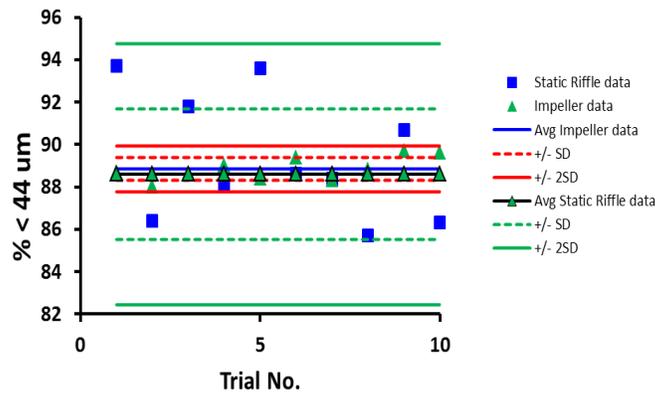
Graph 2. The actual data entries of 10 analyses for each method in addition to the average, average±SD and average±2SD are shown above. Both series were overlaid for a better visualization of the precision difference. As demonstrated the error of subsampling is much larger in the riffle method. Note that the average of both techniques are nearly identical.

Conclusions

In general, the precision of an analytical technique will always be limited by the precision inherent in each step involved when working towards the goal of precise findings. To obtain relaible results, an analyst must minimize the errors associated with each step of sample preparation up to the final analysis, especially the ones which introduce the most significant errors. When the parameters are well defined in an optical bench LD-PSD analyzer, the result of analysis will be highly precise. In reality, the steps in a sample preparation are the major source of uncertainity in PSD analysis by LD-PSD analysis. Our findings show that the standard deviation can be greatly reduced by an impeller method of subsampling from a slurry sample instead of a conventional method of rifflling of solid samples. This method is more effective when the MFT specimens become less homogenous and hence have greater probability that the static riffle will fail to provide repeatability.

References

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