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Comparison of particle size distributions obtained by laser diffraction and sieve-hydrometer methods for oil sand tailings

Darren Senft, Srbojib Masala and Reza Nik
Klohn Crippen Berger, Calgary, Alberta, Canada
Scott Martens, Gennaro Esposito and Tim Eaton
Shell Canada Energy, Calgary, Alberta, Canada



ABSTRACT

The paper presents a comparison of the results of about 600 Particle Size Distribution (PSD) tests on tailings from the Shell Canada Muskeg River Mine site. PSD test methods included Laser Diffraction (LD), Clean Sieve/Hydrometer (CH) and Dispersed Dirty Sieve/Hydrometer (DDH). The LD and CH tests were conducted on bitumen-free samples obtained by the Dean-Stark method. In the DDH tests, the bitumen was not removed from the samples prior to the hydrometer test. The results obtained by different testing methods are compared and the comparisons required for conversion of the PSD parameters from one testing method to the other are calculated. Possible causes for differences in the results are discussed, the primary one being presence of bitumen in oil sands tailings. Recommendations and conclusions are made for application of the PSD standard methods specific for oil sands tailings.

PRESENTACIONES TÉCNICAS

Este artículo presenta la comparación de resultados de cerca de 600 ensayos de Distribución Granulométrica (PSD, Distribución de Tamaño de Grano) realizados en muestras de residuos de minería de las arenas del petróleo en el proyecto Muskeg River Mine de la compañía Shell en Canadá; estos incluyen ensayos de Difracción Laser (LD), Granulometría por lavado e Hidrometría (CH) y Granulometría por dispersión e Hidrometría (DDH). Los ensayos de LD y CH se realizaron en muestras limpias de betún (petróleo) obtenidas por el método Dean-Stark. En los ensayos DDH, a las muestras no se les había removido el betún previo a la hidrometría. Los resultados que se obtuvieron por los diferentes ensayos son comparados y se calcularon las correlaciones necesarias para obtener los parámetros de conversión de distribución granulométrica (PSD) de un método a otro. Se analizan las posibles causas de las diferencias entre los resultados, considerando en primer lugar la ubicuidad del betún en las arenas del petróleo y los varios procesos requeridos para la preparación de las muestras. Se presentan recomendaciones para el desarrollo de métodos normalizados, específicos para los residuos de minería de las arenas del petróleo.

1 INTRODUCTION

Particle size distribution (PSD) is one of the basic properties in soil mechanics. It is used for soil classifications and in various comparisons to estimate other soil properties. Several parameters can be derived from particle gradation curves, including: the median particle size D_{50} ; the effective size D_{10} ; coefficients of curvature and uniformity C_c and C_u ; fines content FC; and clay content. These parameters, commonly used as descriptors for soil classifications, are intimately associated with typical, or expected, soil behavior: magnitude of settlement, deformation rate in consolidation, available strength under loading, and similar.

In the oil sands mining industry, PSD plays a similar role. It serves to broadly classify tailings into fine and coarse fractions, with somewhat indistinct boundaries depending on the operator. Similar parameters derived from a PSD curve serve as common descriptors of tailings products and directly point toward specific properties and behavior in mining applications.

Fines content and clay content are the two most often used PSD parameters. In geotechnics, fines are defined as particles smaller than 0.074 mm, while in oil

sands mining the threshold value is 0.044 mm. Therefore, a fraction between 44 and 74 microns, which is considered (coarser) silt in soil mechanics, is classified as sand in mining. Clay particles are those smaller than 0.002 mm both in mining and geotechnical engineering.

Common methods for PSD determination in soil mechanics are sieving, for particles larger than 74 microns, and hydrometer sedimentation for particles smaller than 74 microns. The sieve-hydrometer (SH) test method is specified by ASTM.

Both sieving and hydrometer are generically manual techniques, require relatively large amounts of material for testing, and may be slow and expensive for certain mining applications. This is perhaps the main reason why the oil sands industry selected Laser Diffraction (LD) as a preferred method for PSD determination, first for ore characterization, then for tailings as well. As a relatively new method, with a wide variety of testing equipment and procedures, LD is still under increased scrutiny of both scientists and practitioners. Different equipment and various preparation and testing procedures make comparison of the results and standardization difficult. In addition, the interpretation is based on a number of simplifying assumptions; in

essence, LD does not measure particles sizes, but obtains them as a product of physical analogies, with some underlying assumptions (light scattering patterns around spherical particles). This is a similar situation as with the hydrometer, where the interpretation is enabled by the assumption of spherical shape of particles, which is generally not the case. Therefore, calibration of LD by comparison with other test methods is essential. LD and SH do not produce the same PSD curves, and no general correlations have been developed, not surprising with the variety of soil gradations. However, some material or site specific relationships have been successfully established between various PSD testing methods.

An additional stimulus for this study was the recent legislation for the oil sands industry in Alberta, Directive 074 (ERCB, 2009). This directive prescribes sieve and hydrometer methods as the requirements for legislated reporting of oil sands tailings properties. However, it does allow for the use of other PSD methods if their correlation with SH can be satisfactorily established, primarily for the fines content in 44-micron basis (FC_{44}). This is the main reason Shell began a comprehensive testing program to develop a correlation between PSD parameters obtained by the two methods. This will allow the use of the more expedient LD technique in everyday work. For ERCB annual reporting the LD parameters can be converted to the SH values.

PSD testing of oil sands tailings is complicated by the presence of bitumen, which is variable in its composition and properties. Various adaptations of existing standards exist; they differ in: whether bitumen is removed or not; if it is, how it is performed (e.g. by Dean-Stark, cold wash, etc.); if it is left in a sample, how it is treated for testing (dispersed or not), etc.

This paper presents the result of PSD testing of samples of tailings taken from Shell Canada Energy's Muskeg River Mine (SCE MRM) tailings ponds during a regular pond survey in the fall of 2009. The various sampling methods used are described, including their advantages and drawbacks. Comparisons of PSD parameters of different methods are presented. Comparisons are also presented of different definitions of PSD parameters (e.g. FC_{44} and FC_{74}) for the same testing method. Suggestions are made for improvement of certain aspects in testing. Shell Canada continues to refine tailings sampling and testing procedures to support mineral mass balance regulatory reporting and development of new tailings technologies aimed at reducing fluid tailings inventories and producing 'trafficable' tailings deposits.

3 PREVIOUS WORK

Comparative testing of PSD determination methods at Shell commenced with start-up of the MRM operation and the first pond surveys in 2003. An MRRT report (2003) makes a comparison of the LD and SH results, particularly the fines contents in the 44-micron basis. They find that for the tailings with high fines content ($FC_{44} > 80\%$), the LD method provides lower fines than

the sieve method. However, for coarser tailings ($FC_{44} < 60\%$), the LD method results in higher fines than the sieve method. These findings agree with our results.

From 2004 to 2009 Sethi was using LD and SH methods for PSD analysis of bitumen-free samples from the MRM tailings pond (Sethi, 2006). The LD equipment Sethi used was different from MRRT's, and Sethi's sample preparation technique was focused on dispersion of the finest fractions. Differences between LD and SH methods were noted, but not analyzed.

A comprehensive comparative study of LD and SH was performed by Mahood et al (2009) on 290 samples of Shell's ore, with fines content in the 44-micron basis up to 90%. In preparation, all samples went through Dean-Stark bitumen extraction and later disaggregation of lumps by pestle and mortar. LD was completed using a single laser diffraction machine and the same set of operating procedures/parameters. SH was conducted at one laboratory using the ASTM procedure. Therefore, the inter-laboratory variability was eliminated from the results. The results of the LD and SH were compared at discrete points along the PSD curve. They show comparable fines contents FC_{44} and FC_{74} (the 1:1 relationship with the data dispersion of about $\pm 10\%$), but also a tendency for LD to overestimate finest fractions, including the clay content. Curving trends were visible on all plots for fractions smaller than 44 microns, similar to MRRT findings in 2003. LD was providing higher fines than SH, except at the 2 micron level (clay size) where LD heavily underestimated the clay content.

The above methods were slightly different than the methods used in the presented testing.

4 SCOPE OF PSD TESTING IN 2009

In preparation for tailings regulatory reporting beginning in September 2010, a field testing and sampling program was completed at MRM in the fall of 2009. Laboratory determination of PSD of collected samples included the following methods: Laser Diffraction (LD), three variants of Sieve Hydrometer (SH) method, and Scanning Electron Microscope (SEM). The two most important tasks were:

- establishing Standard Work Procedures for tailings sampling and testing procedures for future programs at MRM and Jackpine Mine (JPM);
- establishing SH - LD comparisons (with and without hydrocarbons present) for fine tailings.

4.1 Sampling

A total of 587 samples from 64 sites were tested for PSD during the MRM pond survey in 2009 (KCB, 2010b). Samples were collected from the pond or beach surface to native ground in both fluid and solid tailings, in intervals ranging from 0.25 m to 1.0 m. Spatial distribution of samples was intended to provide representative values for the pond geotechnical model, not to cover the spectrum of possible gradations of

deposited tailings. This accounts for the variable coverage of tailings gradations in the 2009 PSD data set.

Depending on the depth and material type to be sampled, a number of different sampling tools were used. These are summarized in the following sections, and detailed in the TDP Procedures Manual document (KCB, 2010a). The first three samplers are standard equipment of Geoforte, Edmonton, AB; the other two are sampling tools of Conetec, Vancouver, BC.

4.1.1 Suction Sampler

The suction sampler was used to sample fluid tailings (FT) from the surface of the pond down to a maximum depth of 3.5 m, recovering a 4–5 litre sample (according to Directive 074, 'fluid tailings' are tailings that have an undrained strength less than 5 kPa).

4.1.2 Fluid Piston Sampler

The fluid sampler was used to sample FT and soft fine tailings/sand mixtures. The sampler operates with the help of pressurized nitrogen inside the sample cylinder. The gas keeps the piston on the bottom of the sampler until the sampler is at the desired elevation. Once the sample depth is reached, the gas pressure is released and hydrostatic pressure at the bottom of the sampler forces material into the sampler. It is noted that the sample is collected from a fixed point in the pond.

4.1.3 Cyre Soft Tailings Sampler

The Cyre sampler was used to collect samples of denser fine tailings (over 25% - 30% solids), fine tailings / sand mixes, and asphaltenes. The sample size is 1 m long and 37 mm in diameter. This sampler also operates with the help of pressurized nitrogen inside the sample cylinder, which keeps the piston at the bottom of the sampler. Once the sampler is in position the piston is kept at a fixed elevation while the gas pressure is released and the sampler cylinder is pushed over the sample. Therefore, this sampler does not suck tailings and provides a lower level of disturbance than the suction of fluid piston samplers above.

4.1.4 Wireline Fluid Sampler

The Wireline Fluid Sampler is similar in design to the Geoforte Fluid Piston Sampler. This sampler is comprised of two parts; a lower portion consisting of a cylindrical chamber, which collects the sample, and an upper weight which keeps the sampler in an upright position as it is lowered to a sample depth via a wireline attached to a winch on the boat. The sampler uses a compressed gas source to keep the sample chamber closed until the desired sample depth is reached, then the gas is turned off. As the gas is expelled, the piston inside sample chamber rises, and the chamber is filled with FT. Once on surface, gas pressure is used to extrude the sample.

4.1.5 Sonic Sampler

Sonic sampling was required to collect samples from the beach locations and below pond bottom, as the other samplers were not capable of penetrating coarse (sandy) tailings. Sonic sampling was conducted with a regular drill rig fitted with a sonic drill head and a piston type sampler. The sampler was capable of collecting a 50 mm diameter sample in 2 m lengths.

The sonic drill head uses a mechanically induced vibration that is transferred directly to the sample rods and the sampler. The vibrations induce a very fine layer of liquefied soil surrounding the sampler cylinder and the rods, reducing both rod shaft friction and tip resistance. The vibrations are also used to extrude the sample from the cylinder. This repeated vibration causes a high level of disturbance in the samples.

5 REPEATABILITY OF PSD TESTING

Before the testing program started, a control series of three blind duplicate samples was delivered to the contributing laboratories for assessment of the repeatability of their work. The results showed occasional large differences in the corresponding data points for identical samples, which suggested the need for a tighter control over testing procedures. It was not clear at the time whether the problem was in the test performance or poor sub-sampling for the control series. The sub-sampling procedures were subsequently improved, as mentioned in section 6. However, due to time constraints a second control test for repeatability was not conducted.

6 PREPARATION – SUB-SAMPLING

When more than one type of test was to be conducted on a sample, it was necessary to divide it into a number of sub-samples. Sub-sampling had to be completed in such a way that each sub-sample had representative characteristics close to those of the source tailings sample. Depending on the fines content of the tailings sample, two sub-sampling methods were used during the 2009 program.

6.1 Standard Sub-Sampling

This method is used for the non-segregating tailings samples in which the sand-to-fines ratio (SFR) is low and the material has reasonable consistency to prevent instant segregation of the coarse particles. Initially a spoon/spatula is used to stir the contents of the sample container, mixing the thin and thick components. Next, a kitchen mixer (with two mixing blades) is used to thoroughly homogenize the sample for about one minute. A scoop or ladle is then used for sub-sampling: one scoop of material is poured into the container of sub-sample 1; the original sample is stirred with the ladle again; then another scoop of material is poured

into container of sub-sample 2. This procedure is repeated until the original sample contents are completely sub-sampled.

6.2 Sub-Sampling by Freezing

This sub-sampling method is used for the segregating tailings samples in which the SFR is high, with solids content less than approximately 40%. The standard sub-sampling procedure for samples of this composition resulted in material segregation or non-homogeneities and variability within replicate sub-samples. Such variations could potentially lead to significant analytical errors. A freezing method for sub-sampling was developed by Shell CRC (KCB, 2010a), and included the following steps:

- Mix sample with a drill and impeller and homogenize as much as possible, making sure the sample does not stick to the side of the pail wall.
- Insert baffles all the way to the bottom of the pail (insert one piece at a time), creating 8 smaller sections.
- Close lid and immediately place sample into the freezer. Keep sample in the freezer for a minimum of 5 hours to ensure that the entire sample is frozen.
- Rinse the outside wall of the pail with warm water to loosen the sample.
- Turn the pail upside down on a metal pan; the frozen sample should drop out easily. If not, use a rubber hammer to tap on the side of the pail to help free the sample.
- Tap the side-wall of the baffles to break up the frozen sample, taking care not to lose any sample when breaking it apart. It should break into 8 blocks for sub-sampling. Place approximately equal volumes of sample into the sub-sample containers.

Prior to using this sub-sampling method on TDP samples, CRC conducted a separate analytical trial on synthetic samples they created. This QC process ensured that the freezing method was viable for this type of sample.

7 PREPARATION – DEAN-STARK

The Dean-Stark test is a common technique for determining the composition of soils containing organic components (mass / weight percentages of bitumen, minerals and water). It is also used for removal of bitumen from oil sands tailings samples.

This method applies to wet bituminous tailings, without special preparation. About 80 to 140 grams of wet tailings is placed in a cellulose extraction thimble. Solvents (Toluene and an emulsion breaker) are refluxed through the sample at a medium boil (115°C) until all water has been distilled and the solvent dripping is clear. Minimum reflux time is 8 hours. The thimble and bitumen free solids are placed in a drying oven at 90 ± 2 °C for a minimum of 4 hours.

If the dried material, which typically comes out in lumps, is subsequently used for PSD determination, it must be disaggregated by a rubber tipped pestle and mortar. Therefore, this thermo-chemical processing is

likely to affect the original material gradation, particularly of finer fractions.

8 PSD TESTING METHODS

Four testing methods were used to determine the PSD of the tailings samples.

The Laser Diffraction (LD) test has been used by Shell as the main tool for particle size determination as this method requires significantly shorter turn-around time compared to the sieve-hydrometer (SH) methods.

The Clean Hydrometer (CH) and Dispersed Dirty Hydrometer (DDH) tests were conducted to derive comparisons between these methods and the LD method.

The reason for running Non-Dispersed Dirty Hydrometer (NDDH) tests was to obtain the PSD of the fine tailings in their flocculated state. The results of NDDH tests are essential for assessment of the settling/dewatering behaviour of the tailings.

LD testing was performed on 225 samples, DDH on 211 and CH on 151 samples.

Detailed descriptions of all laboratory methods summarized in this section are included in the TDP Procedures Manual document (KCB, 2010a). Table 1 provides a step-by-step comparison of the different PSD determination methods used in the 2009 program.

In addition to the above test methods, for a limited number of clean and bituminous samples, the Scanning Electron Microscope (SEM) was used. This method is not included in this review.

8.1 Laser Diffraction

The objective of this testing was to determine the PSD of the solid phase of bitumen-cleaned tailings in a dispersed state. To prepare a sample for this test, the tailings sample first went through the Dean-Stark procedure in which the bitumen and water were removed and a clean (bitumen-free) sample was produced. This clean sample was dry, typically in hard lumps, and had to be disaggregated by a rubber-tipped pestle and mortar. De-ionized water, 1% triton-X (surfactant) and 5% Calgon solution (dispersant) were added to the dry sample before LD testing was conducted.

In this survey, all LD tests were performed by the Exova laboratory using the Mastersizer 2000, manufactured by Malvern Instruments.

The Calgon used by this lab was a consumer grade product which did not contain sodium hexameta-phosphate. It is suspected that Calgon does not provide the standard level of dispersion.

8.2 Clean Hydrometer

The objective of this test was to determine the PSD of the solid phase of clean tailings in a dispersed state. To prepare the sample used for this test, the tailings sample first went through the Dean-Stark procedure in which the bitumen and water were removed and a

bitumen-free sample was made. The sample was then divided into two portions; one containing particles larger than 2.0 mm (remaining on the No. 10 sieve), and the other containing particles passing the No. 10 sieve. Sieve analysis was conducted on the coarser fraction and hydrometer analysis was conducted on the finer fraction. Testing was completed according to the ASTM D422 standard. A 4% solution of sodium hexameta-phosphate was used as the dispersing agent. One concern about cleaning of the samples by the Dean-Stark method has been possible 'loss of fines'. Further discussion in this regard is provided in section 9.

All CH tests in this survey were conducted by the KCB laboratory.

8.3 Dispersed Dirty Hydrometer

The objective of this test was to determine the PSD of the solid phase of bituminous tailings in a dispersed state. The soil sample used in this test method was not oven dried, but was used wet, as received. The sample was mixed with a 4% solution of sodium hexameta-phosphate as dispersing agent, and after 12 hours sieve - hydrometer testing was conducted according to the ASTM D422 standard.

In this survey, all DDH tests were conducted by the Golder laboratory.

8.4 Non-Dispersed Dirty Hydrometer

The objective of this test was to determine the PSD of the solid phase of bituminous tailings in the non-dispersed (flocculated) state. The sample used in this test method was not oven dried. The testing procedure is similar to the ASTM D422 method, with the following exceptions: no dispersing agent is added to the wet sample and no high-speed mixing is applied, in order to prevent breakage of the natural (flocculated) particles. In this study, all NDDH testing was performed by the KCB laboratory.

The results of this method were not correlated with the others, as the NDDH test procedure was essentially incongruent with the other PSD tests that all used dispersion in sample preparation. It is worth noting that the fines contents and particularly clay contents determined by the NDDH method were always lower than the fines and clay contents obtained by other methods. This is a good indicator of the fine tailings conditions in an actual tailings pond: the fines there exist in a flocculated, not dispersed state.

9 RESULTS

Typical PSD curves for coarse and fine tailings samples are shown on Figures 1 and 2. The separation between PSD curves obtained by the various test methods increases as the amount of fines increases. In addition, the PSD curves of different tests tend to diverge toward the finer fractions. It is difficult to notice differences in

fines or clay contents in the PSD curves for coarse soils as they may be contained within the measurement error.

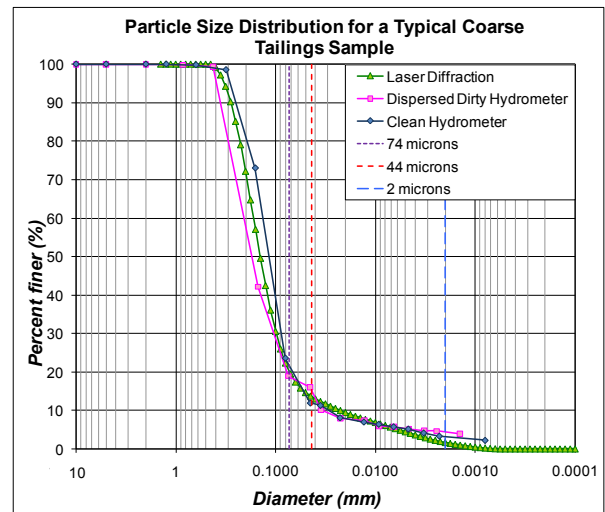


Figure 1. PSD for a typical coarse tailings sample

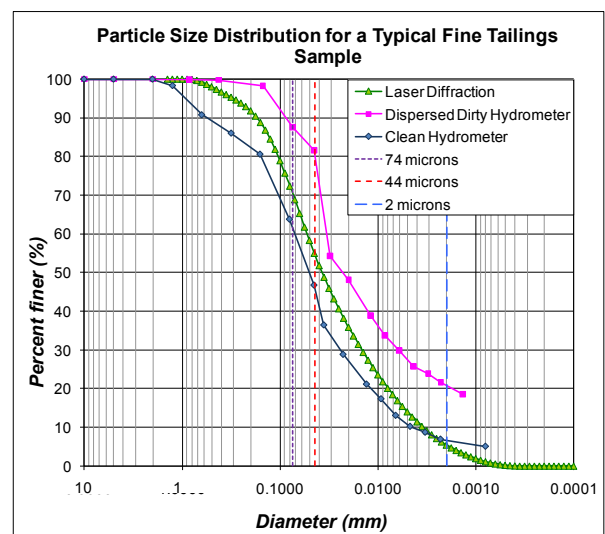


Figure 2. PSD for a typical fine tailings sample

9.1 Comparison of Fines Contents in 44- and 74-Micron Bases

Figure 3 presents comparison plots between FC_{44} and FC_{74} values for the three test methods (LD, DDH and CH). The largest dispersion of data, about $\pm 15\%$, is in the middle of the range, $FC_{44} = 50\%$, for all three test methods. The dispersion decreases toward the ends, entirely coarse and fine materials.

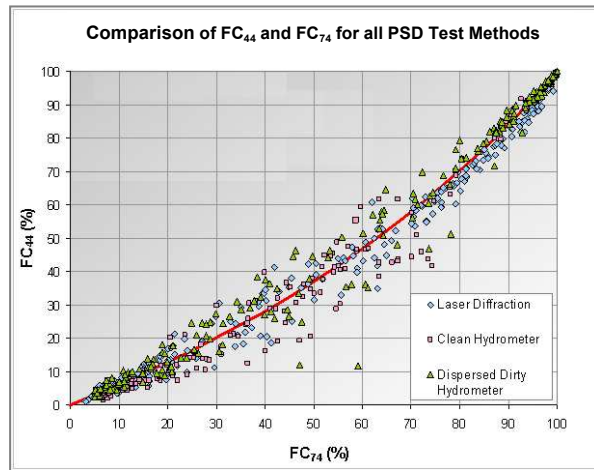


Figure 3. Comparison of FC_{44} and FC_{74} obtained by LD, DDH and CH test methods

It should be noted that the relationships are functions, not constants. Often, the relationship between FC_{44} and FC_{74} or, between the sand-to-fines ratios SFR_{44} and SFR_{74} , which is identical, is expressed as a constant. This should be valid for soils with a narrow range of fines contents such as ore or coarse sand tailings beaches. The difference between FC_{44} and FC_{74} defines a slope on the PSD curve for a particular fines content range. For the same soil, with similar gradations of samples taken, it is reasonable to expect to have a statistical comparison between closely spaced values, like the fractions passing 44- and 74-micron sieves.

9.2 Comparison of Fine and Clay Contents for Various Test Methods

Figure 4 presents a comparison plot for FC_{44} obtained by sieve-hydrometer (DDH and CH) and LD methods. Figure 5 presents the clay content comparison for the same test methods.

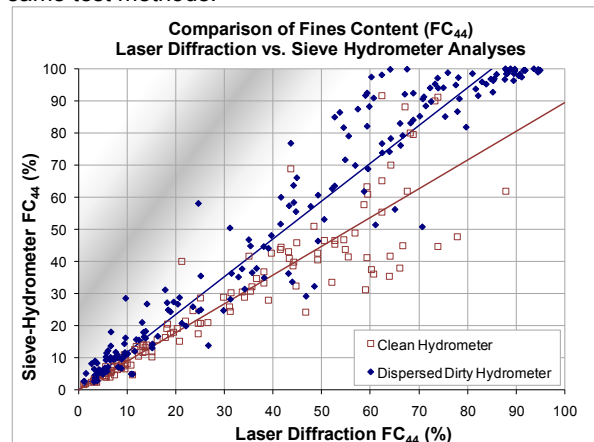


Figure 4. Comparison of fines contents in 44-micron basis obtained by LD, DDH and CH methods

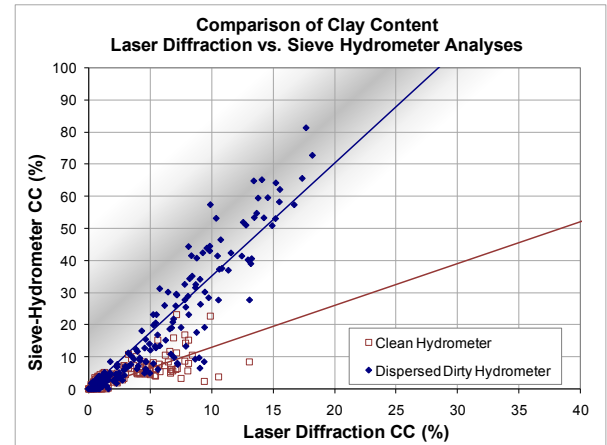


Figure 5. Comparison of clay contents in 44-micron basis obtained by LD, DDH and CH methods

10 EFFECTS OF DEAN-STARK METHOD AND BITUMEN

During an early stage of testing, it was observed that different PSD analysis methods (LD, DDH and CH) occasionally produced widely varying results for the same sample. The fines contents measured by the DDH method were, on average, higher than the fines contents measured by the LD and CH methods. As the latter two methods use bitumen-free samples, it was concluded that the PSD analyses that apply the Dean-Stark method underestimate fines contents compared to the DDH method. This was particularly true for fines-rich tailings, where the FC_{44} differences between the DDH and LD/CH were the highest. Two possible causes were speculated:

- High-temperature dehydration and chemical treatment (exposure to toluene) by the Dean-Stark method, used in the preparation of samples for LD and CH tests, adversely affect fine fractions (by creating hard lumps).
- Bitumen presence in oil sands tailings alters the electro-chemical state of the surfaces of fine particles (bitumen changes the geotechnical index properties of the fine fraction). This makes the particles 'stickier', allowing them to more easily create agglomerations that become hard lumps during the Dean-Stark removal of bitumen from the samples.

10.1 Dean-Stark Method

In order to assess the effect of the Dean-Stark process alone, a limited additional testing on synthetic bitumen-free samples was performed. Six artificial mixtures were composed of varying percentages of commercially available kaolin and sand, with FC_{44} from 10% to 100%. All three PSD test methods were applied to each

synthetic sample. It was found that the differences in FC_{44} values are within or slightly above the measurement errors. Therefore, some indication, but no conclusive evidence was found that the Dean-Stark method is solely responsible for the 'loss of fines' in LD and CH methods.

On the other hand, the clay contents measured in the LD analysis were consistently significantly lower than in the CH method, although both tests used bitumen-free samples. A possible explanation could be the degree of dispersion by Calgon, which is not subject to a rigorous level of production quality control.

10.2 Bitumen

It was speculated that dispersed bitumen in the DDH samples could be mistaken for fines in the hydrometer testing. The differences in measured FC_{44} and clay contents between DDH and LD / CH methods were inspected for this purpose. It was found that, in the large majority of tests, these differences were much larger than the bitumen content in the sample.

Another possibility was that light bitumen fractions increase the density of water in the hydrometer, make it more viscous and hinder particle sedimentation, resulting in more reported fines in hydrometer testing. This option could not be investigated within the time constraints of the project.

Therefore, bitumen may contribute, but is not the principal factor for DDH and LD / CH data discrepancy.

It was recommended to investigate the combined effect of bitumen and the Dean-Stark sample cleaning on the PSD analysis results by a more extensive and focused testing of synthetic bituminous samples. That work was out of the scope for this task.

11 CONCLUSIONS AND RECOMMENDATIONS

The fines contents measured by the DDH method, conducted on bituminous samples, are, on average, higher than the fines content values measured by the LD and CH methods, conducted on bitumen-free samples. The PSD analyses that apply the Dean-Stark method report lower fines contents compared to the DDH method. This is particularly true for fines-rich tailings. The clay contents measured by the DDH

method are, on average, significantly higher than the clay contents measured by the LD and CH methods. The Dean-Stark method magnifies the 'loss of fines' in the clay fraction.

The DDH may be a useful test for determining maximum clay content for use in design of plant components that are sensitive to clay content in the dispersed state. The NDDH may be a useful test for assessing settling and consolidation of thin fine tailings and mature fine tailings deposits. The 44 μ m fines content of ore typically ranges from 10% to 20% by weight of total mineral. LD and CH are considered reliable tests for minimum 44 μ m fines content and establishing correlations for converting LD fines to SH fines for ore to tailings mass balance exercises.

It is suspected that Calgon does not provide the standard level of dispersion. Additional focused testing of the dispersion level is recommended, with two series of identical samples, made with variable fines content, one using Calgon, the other Sodium Hexametaphosphate as dispersant.

It is recommended to investigate the combined effect of bitumen and the Dean-Stark cleaning procedure on the PSD analysis results, in controlled testing conditions with synthetic bituminous samples.

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Table 1. Comparison of PSD Methods Used in 2009 Pond Survey.

Test Method	Clean Hydrometer (CH)	Dispersed Dirty Hydrometer (DDH)	Non-Dispersed Dirty Hydrometer (NDDH)	Laser Diffraction (LD)
Laboratory	KCB	Golder	KCB	Exova
PSD definition	Mass	Mass	Mass	Volume
Sample state	Dry	Wet	Wet	Dry
Test temperature	High (115°C) during preparation	Room Temperature	Room Temperature	High (115°C) during preparation
Bitumen presence	Bitumen-free	Bituminous	Bituminous	Bitumen-free
Bitumen removal method	Dean-Stark	-	-	Dean-Stark
Sample size	~50 grams of dry soil	~50 grams of dry soil	~50 grams of dry soil	4 to 7 grams of dry soil
Preparation				
Disaggregation	Rubber-tipped pestle and mortar	No	No	Rubber-tipped pestle and mortar
Dispersion	Dispersed	Dispersed	Flocculated	Dispersed
Dispersing agent	125ml of the 4% solution of sodium hexametaphosphate	125ml of the 4% solution of sodium hexametaphosphate	No	Distilled water, 1% Triton-X (surfactant), and 5% solution of Calgon
Soaking	Minimum 12 hours	Minimum 12 hours	No	Yes
Mixing	High-speed mixing for 1 minute	High-speed mixing for 1 minute	No	Sample vial rotated in 3D for 30 seconds
Test procedure				
Container size	1 litre standpipe	1 litre standpipe	1 litre standpipe	Each sample is run twice and the instrument calculates an average. If poor agreement (the D ₅₀ must be within 6% of each other) the sample must be re-run. A sample will be re-run a maximum of 3 times and the average of the best two runs is taken as the final result.
Water	Distilled	Distilled	Process/pond water	
Shaking	Shake the standpipe upside down for 1 minute	Shake the standpipe upside down for 1 minute	Shake the standpipe upside down for 1 minute	
Post-test processing	Wet sample washed over sieve #325 (0.044 mm) and the coarser fraction is dried and sieved	Wet sample washed over sieve #325 (0.044 mm) and the coarser fraction is dried and sieved	Wet sample washed over sieve #325 (0.044 mm) and the coarser fraction is dried and sieved	