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# Comparison between hydrometer and laser diffraction methods in the determination of clay content in fine-grained soils

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### Comparison between hydrometer and laser diffraction methods in the determination of clay content in fine-grained soils

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Abstract. Clay content is the main characterizing parameter when classifying fine-grained soils. Hydrometer method (HD) is commonly used in geotechnics to obtain the grain size distribution properties of fine-grained soils. However, this test is laborious and time consuming as compared to alternative methods such as laser diffraction (LD). In this study, a comparison between the results for clay content of 19 samples from different locations in Finland using HD and LD methods was carried out aiming at obtaining a mathematical expression that allows to transform LD measurements into HD results. The results were statistically compared showing significant differences between the results of these two techniques and underestimation of the clay content by the LD method was observed. Based on the results, the reliability of the laser method in the determination of clay content is discussed.

#### 1. Introduction

Particle size distribution of soils is an important characteristic affecting soil properties such as compressibility, frost susceptibility and water transport and retention. Particle size distribution of finegrained materials in geotechnical engineering has been traditionally determined by hydrometer method (HM) or pipetting. The main objective of the hydrometer analysis is to obtain the clay content (percentage finer than 0.002 mm), which controls the classification of fine-grained materials (soil name).

As an alternative to the methods previously mentioned, laser diffraction method (LDM) might be used for particle size analysis of fine-grained soil samples. The method offers fast and reliable results for a great variety of materials and users have the possibility of using both wet and dry dispersions. Compared to hydrometer method, the results are obtained significantly faster, in a wider range of size and with less human intervention. However, if the result are to be used for geotechnical design, its calibration against hydrometer method is required, according to standard EN 1997-2:2007 [1].

Various research studies have been conducted to compare results from both methods using soils of different textures. Results of the correlation between both techniques are varied, especially when it comes to measure the clay fraction. Statistically significant differences have been found between laser diffraction and traditional methods such as hydrometer and pipette method, but in some cases, a correlation was possible to establish. Ryżak et al [2] presented a regression equation obtained from the comparison of particles size distribution of 22 soils measured by HM and LDM, with a coefficient of correlation ( $\mathbb{R}^2$ ) of 0.88.

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Di Stefano et al. [3] also presented a linear regression equation to transform the results from LDM into HM results, but the samples were divided into textural groups (with similar soil classification). The authors stated that for a more reliable correlation between particle-sizes with the investigated techniques, the regression equation should be obtained for a specific textural group. This statement is in agreement with the observation made in a more recent study by Ryżak & Bieganowski [4], who found that for a better correlation between HM and LDM, the samples should belong to the same soil type. The  $R^2$  obtained in this study was 0.75, for fraction <0.002 mm. On the other hand, Orzechowski et al. [5] obtained a  $R^2$  between LDM and HM as low as 0.263 for glaciolimnic sediments belonging to a same location. While the clay fraction was the highest detected by hydrometer method, laser diffraction method results showed a predominance of the silt fraction, leading to considerably different soil classification. Likewise, the amount of clay fraction (< 0.002 mm) analyzed by pipette and hydrometer method was 3–4 times higher in comparison to laser method.

Centeri et al. [6], also found a significant difference between the two methods when measuring the clay fraction. In most of the cases, the clay content was underestimated but in other cases an overestimation was obtained. However, there were also cases where no difference was presented between the clay content determined by HM and LDM. The authors state that this was due to a disaggregation technique performed before the laser analysis and therefore, they recommend the introduction of a sample preparation protocol such as the use of certain techniques for disaggregation when laser method is to be used in particle size distribution measurements.

Both hydrometer and LDM were also used in a sediment research made for planning the deepening of Oulu sea route by dredging [7]. Altogether more than 40 samples were taken and 19 of them were studied with both HM and LDM. Based on HM the soils were mainly silts and sands and the average clay content was less than 4%; highest value 24%. Based on grain size curves made with HM results, 74% of the samples did not have any clay particles. According to LDM the samples had much higher clay contents; average >20% and highest value 94% and only 1 sample had clay content lower than 2%. The correlation between HM and LDM was very low ( $R^2 < 0.01$ ).

Despite the differences of the correlations obtained by various authors, clay content is usually (but not always) underestimated by laser diffraction method and a higher correlation is obtained when analyzing coarse fractions, which is widely reported [3], [4], [5], [6], [8]. The aim of this study is to compare the results of clay content obtained by two different methods: hydrometer and laser diffraction method, aiming at obtaining a regression equation if a statistically significant correlation is observed between the two techniques. The main motivation to carry out this comparison is to apply laser method in the clay content determination required by the Baltic Marine Environment Protection Commission (HELCOM) for the dredged material recovered from ports and harbors that will be later deposited at the Baltic sea. When dredging at sea areas clay content is important because detrimental elements have been observed to concentrate in fines and clay fraction. Especially metals adhere to clay particles and organic material. HELCOM guidelines for management of dredged material at sea report [9] states: "It is recommended that normalized values of contaminants should be used to enable a more reliable comparison of contaminant concentrations in dredged material with those in sediments at deposit or reference sites, as well as with action levels." According to Ympäristöministeriö guidelines [10] normalization is recommended to be done based on clay content and organic content and limit values/action levels for depositing dredged material back to sea are given based on these normalized contaminant concentrations.

According to the HELCOM guidelines for management of dredged material at sea [9], grain size analysis either by sieving or laser methods should be performed on the material to be deposited at the sea, in order to determine percentage of clay fraction. However, following standard EN 1997-2:2007 [1], laser method must be calibrated using hydrometer as the reference method. This will allow to use the laser method in combination with hydrometer, saving time and costs. According to regulations and guidelines for dredging of sediments from Finnish Ministry of Environment [10], the grain size distribution of fines should be determined with sedigraph, pipetting or hydrometer.

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#### *1.1. Hydrometer method*

Hydrometer test, also known as aerometer method or sedimentation test is a grain size analysis used in geotechnical engineering to obtain the particle size distribution (PSD) of fined-grained soils passing 0.063 mm sieve (fine fraction). For soil constituents retained by 0.063 mm sieve (coarse fraction), the test is paired with a sieving analysis. The main instrument used when performing the test is a hydrometer, a device designed to measure the density or specific gravity of liquids, that is, the ratio of the density of the liquid to the density of the water.

The general procedure in Finland consists in mixing oven-dried soil (using wet samples is also possible) in distilled water previously mixed with a dispersing agent and inserting a hydrometer to obtain different readings of the specific density of the solution at different times. When soil is mixed and the test starts at t=0, all particles are in suspension, but after less than one minute, only silt and clay (very fine particles, <0.0063 mm) are in suspension while the heavy particles will settle at the bottom. Eventually, only clay particles will be in suspension in the same degree of concentration as at the start of the test. In other words, particles will settle at different rates that are dependent upon their diameter.

The mathematical equation for the estimation of the velocities at which, small spherical particles settle in a fluid medium constitutes Stoke's Law. This equation relates settling velocity with diameter of the particles (assuming spherical particles). With every specific gravity measured at measuring depth, the concentration of particles finer than a specified size is determined using Stoke's Law and consequently, the percentage of these particles within the solution.

#### *1.2. Laser diffraction method*

Laser diffraction is a technique for measuring PSD consisting in sending an incident laser beam through the dispersed particles producing a scattering pattern when the light is diffracted that varies according to the size of the particles, the wavelength of the laser and the optical properties of the medium and particles. The scattering pattern is formed by light intensity as a function of the scattering angle. The angle of diffraction is inversely proportional to the particle diameter and the intensity of the diffracted light at certain angle is a measure of the number of particles with a specific diameter [11]. Each constituent particle in the sample creates a scatter pattern from the diffracted light and the pattern measured by the laser device is the sum of all the patterns produced.

Measurements of the pattern of light scattered by the particles in the sample to obtain the PSD are carried out using the Mie theory of light scattering and the Fraunhofer diffraction model [12]. The Mie theory requires that the optical properties – the refractive index – of the sample being measured to be known, because it takes into account the effect of the refraction of the light in the estimation of the scatter pattern, in addition to diffraction [11]. In smaller particles, the effect of the refraction on the intensity of the pattern becomes important. Thus, Mie theory can more accurately estimate PSD with particles as small as 0.045  $\mu$ m [13].

Fraunhofer model does not require the refractive index of the material to calculate PSD. For larger particles, the refractive index values have a significantly smaller contribution to the scatter pattern than small particles because for in the case of the first ones, diffraction takes place in the forward direction, being the same as the direction of the laser beam [13]. Therefore, to use Fraunhofer diffraction model accurately, the particles must be considerably larger than the wavelength of the light [12], at least 10 times the laser wavelength [11], [13], such that, refraction can be neglected. Using Fraunhofer optical model to estimate PSD in fine-grained samples might lead to an underestimation of the clay fraction, because refraction is not accounted [12] and [14] in [15].

#### 2. Materials and methods

#### 2.1. Characterization and pre-treatment of the samples

Test material consisted in 19 fine-grained soil samples from different locations in Finland and with varying PSD. Each sample was subjected to HM in Aalto University's geotechnical laboratory and in an external laboratory. For LDM two laboratories were used: Aalto University's laser diffraction

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analyzer and an external laser diffraction laboratory. Thus, two sets of results for each method (HD and LDM) were obtained for each sample. All samples were dried, grinded and homogenized to perform organic content analysis, specific gravity test, hydrometer test and laser diffraction method. The analysis for organic content was carried out before the PSD methods, based on the recommendations of standards ISO 17892-4:2016 [16] and ASTM D422-63 [17], which describe a standard procedure for the pretreatment of samples whose organic content is high enough to affect the results from hydrometer test. Some authors have reported an underestimation of clay content proportional to the organic matter content and recommended to remove the organic matter before sedimentation tests when estimation of soil particles <2  $\mu$ m is intended [18], [19], [20].

#### 2.2. Particle size distribution analysis

Hydrometer method was performed according to standard ISO 17892-4:2016 [16]. The soil solution consisted in 50 g of soil sample, 1000 ml of distilled water and 2.23 g of sodium hexametaphosphate acting as a dispersing agent. Sieve analyses were carried out at the end of each test with the soil settled at the bottom of the jar to determine the particles retained by #0,063 mm sieve. The results were combined with the results from hydrometer test to draw the grain size distribution curves.

In Aalto the grain size distribution of the test material by laser diffraction method was estimated using a Single-Wavelength Laser Diffraction Particle Size Analyzer model LS 13 320 by Beckman coulter systems. The equipment measures the scattering pattern produced by particles in the sample when a laser beam of 750 nm wavelength is diffracted. The particle size analysis covers a size range of 0.02 to 2000  $\mu$ m. Measurements can be made for particles suspended in a liquid or in dry powder form. Results shown in this study were obtained for samples in dry powder form, as this was the technique used also by the external laboratory performing the other set of tests, which allows evaluating the reproducibility of the laser diffraction method.

Two different optical models are offered by the instrument's measurement module to carry out the particle size analysis, Fraunhofer diffraction model and Mie theory. The latter provides more reliable sizes for finer particles such as clay ( $<2 \mu m$ ) than the Fraunhofer model, considering that the wavelength of the laser is 0.75  $\mu m$ . To ensure accuracy of the results obtained by Fraunhofer diffraction model, particles should be  $\geq$ 7.5  $\mu m$  in diameter (10 times the laser wavelength), according to the restriction in size particle for applicability of this model reported in the literature.

However, calculations based on the Mie theory are only possible if the refractive index RI of the particles is known, and the accuracy of the sizes depends on choosing the correct RI values. In this study, calculations were carried out using Fraunhofer diffraction model due to uncertainty in the values of RI for the samples. Nevertheless, the study by Ryżak & Bieganowski [4], where both Fraunhofer diffraction model and Mie theory were used, concluded that the choice of the optical model to calculate the scatter pattern does not have an important effect on the coefficient of determination (R<sup>2</sup>) obtained when results from HM and LDM are compared.

Additional LD tests for 5 of the 19 samples were carried out at a second external laboratory that we will refer to as external2. Tests were performed using the Mastersizer 3000 laser diffraction particle size analyzer and liquid dispersion method, consisting in diluting the soil samples previously dry and grinded, in de-ionized water. In this case, 50 g of soil was diluted in 500 ml of deionized water with an addition of 0.5 g of sodium hexametaphosphate to act as a deflocculant. For each sample, three tests were performed using 150 ml of the 500 ml solution: one after stirring the solution for 1 hour, another one after 6 hours and the last test after stirring overnight. For each test five measurements were taken, for a total of 15 measurements.

The Mastersizer 3000 analyzer uses Mie theory to compute particle sizes. A refractive index of 1.55 was assumed for the samples, a value that falls within the range of RI for clay minerals [21]. Having PSD measurements at different times of stirring the soil-water-deflocculant solution allows to assess the effect of a pre-treatment of the samples to avoid segregation and the use of the Mie theory will confirm if the choice of this theory over Fraunhofer theory gives more accurate results for smaller particles such as clay.

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#### 3. Results and discussion

#### 3.1. Hydrometer method

The amount of clay fraction obtained with hydrometer tests performed by Aalto University ranged from 10 to 87%. On the other hand, for the results obtained by the external laboratory on the same test, the range was between 7 to 84%. The average clay content determined in Aalto was 44,8% and in external laboratory 43,1%. Table 1 shows the results of clay contents determined with the two hydrometer tests as average of the two laboratories for all the samples with the corresponding soil name according to Finnish GEO-classification. For 2 samples there were small deviations in soil name between hydrometer test performed in Aalto and the one performed in the external laboratory for all the data set. Figure 1 shows the relationship between hydrometer test results performed by Aalto and the external laboratory. Figure 1 shows a good correlation between the results ( $R^2=0.98$ ) for clay content by using hydrometer method in two different laboratories. This shows the good reproducibility capacity of this method. Considering the high similarity in the hydrometer method results, the clay content values from Aalto's hydrometer and the external laboratory were averaged and statistically compared to data from laser method performed in Aalto and by the external laboratory.

**Table 1.** Results for clay content and corresponding soil name by test performed and laboratory.

 Results at external2 laboratory are classified according to the stirring time of the soil solution with deflocculant. LD=Laser diffraction. LOI= Loss on Ignition.

			Hydro	ometer	LD. Aalto		LD. External1 <sup>a</sup>		LD. External2.		
Sample	Water	LOI	averag	ged							
	(%)	(%)	Clay	Soil	Clay	Soil	Clay	Soil	1	6	24
			(%)	name	(%)	name	(%)	name	hour	hours	hours
HUT 1	55	2.5	50	liSa	27	saSi	25	saSi	-	-	-
HUT 2	48	1.5	27	laSa	14	saSi	14	saSi	-	-	-
HUT 3	36	1.0	16	saSi	16	saSi	11	saSi	-	-	-
HUT 4	38	1.2	18	saSi	12	saSi	11	saSi	-	-	-
Lahti 1	45	1.9	26	saSi	12	saSi	8	-	-	10	9
Lahti 2	45	2.1	35	laSa	17	saSi	12	saSi	13	12	13
Lahti 3	53	2.4	55	liSa	23	saSi	21	saSi	25	25	21
Stara 1	76	4.5	81	liSa	16	saSi	2	-	-	-	-
Stara 2	78	4.6	86	liSa	9	siHk	19	saSi	-	-	-
Stara 3	53	3.5	53	liSa	12	saSi	13	saSi	-	-	-
Stara 4	59	3.5	56	liSa	24	saSi	20	saSi	-	-	-
Stara 5	68	3.9	67	liSa	26	saSi	23	saSi	-	-	-
Stara 6	31	1.9	25	saSi	12	saSi	10	saSi	-	-	-
Ram. 1	102	3.8	70	liSa	23	saSi	22	saSi	44	43	44
Ram. 2	77	4.9	41	laSa	17	saSi	13	saSi	-	-	-
Ram. 3	96	3.9	43	liSa	12	saSi	16	saSi	-	-	-
Ram. 4	121	8.6	30	saSi	25	saSi	6	-	-	-	-
Ram. 5	36	3.1	47	laSa	30	saSi	27	saSi	26	27	28
Ram. 6	28	1.2	9	Si	8	Si	<2	-	-	-	-

<sup>a</sup> It was not possible to determine soil name for samples whose clay content was less than 10% in the results of laser diffraction method performed in the external l laboratory as the whole particle size distribution was not provided.

#### 3.2. Laser diffraction method

Aalto University and external1 laser diffraction laboratory performed laser diffraction method for all the 19 samples. Results of clay fraction from Aalto are in the range of 7 to 30%, whereas external1 obtained clay fractions over the range of 2 to 27%. In figure 2, the results obtained from LDM performed by both laboratories are presented and compared. A low correlation ( $R^2=0.40$ ) was obtained between the results of laser diffraction from Aalto and the external1 laboratory, despite being performed in the same conditions and with homogenised samples.



**Figure 1.** Comparison of the results obtained with hydrometer tests for all soil samples.

Figure 2. Comparison of the results obtained with laser diffraction methods for all soil samples.

#### 3.3. Comparison of laser diffraction method to hydrometer method in clay content

In order to find a correlation between hydrometer test and laser diffraction method, a regression and correlation analyses were carried out to estimate the relationship between the results for clay content from both techniques. As it is intended to reduce the use of hydrometer test in the future, the prediction models obtained have as dependent variable the clay content obtained by laser diffractometer. Analyses were performed for 2 groups of results:

- Group 1: averaged hydrometer results and laser results by Aalto.
- Group 2: averaged hydrometer results and laser results by the external laboratory.

A summary of the correlation analyses is presented in Table 2. A comparison for clay content of all soil samples according to the analysis group is presented in figure 3 and figure 4. The coefficient of determination ( $R^2$ ) returned by the correlation analysis, was used as parameter for comparing the results obtained with the two methods (HM and LDM).

In agreement with the results obtained by various previous studies comparing the hydrometer and laser method, our results show a consistent underestimation in the clay content percentage by the laser diffraction method. The amount of clay content ( $<2 \mu m$ ) measured by hydrometer method are between 1.0 to 40,6 higher in comparison to laser diffraction method. Thus, significant differences were obtained in soil classification between the hydrometer and laser diffraction methods. In fact, laser results led to different soil classifications in 13 of 19 samples, being this especially critical for the soils samples classified as fat clays (liSa, Stara 1 and Stara 2) by HDM. Sample Stara 2 (liSa) for instance, was classified as silty sand (siHk) according to the results of particle size distribution obtained by Aalto's laser method. All other samples were classified as clayey silts (saSi) or silts (Si) with LDM. Figure 5 shows the grain size distributions obtained for the sample Stara 2 with hydrometer and laser diffraction method, both performed in Aalto University.

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	5	5	
Analysis group	Adjusted R <sup>2</sup>	P-value	
Group 1	0.0402	0.2029	
Group 2	0.1610	0.0500	

 Table 2.
 Summary of correlation analyses.



Figure 3. Scatter plot for set of results of group 1 Figure 4. Scatter plot for set of results of group 2



Figure 5. Grain size distributions obtained for the sample Stara 2 with hydrometer and laser diffraction method, both performed in Aalto University.

It is observed that there is a significant difference between the clay fraction estimated with the two methods, resulting in two entirely different soil size distribution curves despite belonging to the same sample. Likewise, despite the wide range of results provided by hydrometer test, the laser shows a low variability of the results. Figure 6 shows the distribution of the data between both investigated methods (HM and LDM). The Figure allows to compare the width of the range of the results in clay content determined by HM and LDM. A wide range of clay content in laser results is expected as the data set is

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composed by different soil samples from different locations, which was proved by the hydrometer results, whose range is significantly wider than the range of clay fraction results with laser, with clay content up to 87%.

The results for 5 of the 19 samples obtained in external2 laboratory by laser diffraction method were similar to the results obtained by Aalto University and the external1 laboratory, except for the sample Ram. 1 (Table 1). Therefore, neither the pre-treatment of the samples with deflocculant nor the use of liquid dispersion method had much effect in the estimation of the clay fraction by laser diffraction. It is worth noting that the use of the Mie theory for computation of the particle's sizes did not contribute either to a higher accuracy, despite the recommendations found in the literature. However, this is agreement with observations by Ryżak & Bieganowski [4], who found that the selection of the Mie theory in laser diffraction method did not improve the coefficient of correlation obtained in the comparison between HM and LDM for clay fractions calculated according to Fraunhofer theory.



**Figure 6.** Interquartile ranges of soil fractions by the studied methods (Hyd=hydrometer method, LD=Laser diffraction). The band corresponds to the median and the red dot to the mean of the data set.

The statistical analyses showed that the results from hydrometer method and laser diffraction method are statistically different. Poor correlations between these methods for the computation of clay fraction have been observed also before [5].

#### 4. CONCLUSIONS

No reliable regression model was obtained for laser diffraction and hydrometer results due to the poor correlation between the investigated methods. The highest adjusted coefficient of correlation was 0.161 for the averaged hydrometer results and the laser diffraction results by the external l laboratory. Likewise, the poor correlation between the laser results from Aalto and the external l laser diffraction laboratory on the same samples ( $R^2=0.402$ ) is an indication of a lower reliability of this method for clay content estimation compared to the hydrometer method, whose results from two different laboratories showed a good correlation ( $R^2=0.980$ ). Furthermore, it was observed that the range in which results from laser diffraction fall is significantly narrower than the range for hydrometer results and the samples with

the highest clay content according to hydrometer test did not necessarily had the highest clay content estimated by laser diffraction method.

Laser diffraction method for the estimation of clay content might be improved following recommendations by previous studies, such as the pre-treatment of the sample by ultrasonic techniques for the disaggregation of the particles. The simple use of a deflocculant as a pre-treatment seems to have no effect in the computation of clay content by laser diffraction. Liquid dispersion method and the selection of the Mie theory for PSD computations did not improve the correlation either, according to external2 laboratory results. Comparing results for soils from the same location and depth an improved correlation with these two methods could possibly be found in the future. Based on this study laser diffraction method cannot be recommended to be used for soil characterization in geotechnical design or dredging at the moment.

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