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Determination of Particle Size Distribution: Comparison of Standard Hydrometer Method and Laser Diffraction Analysis for Use in Forestry

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Abstract: Laser diffraction analysis is an alternative to standard sedimentation methods designed to determine particle size distribution. In this article, five samples from the forested part of the floodplain of the Svitava River in the Czech Republic were analyzed. Laser diffraction analysis and sedimentation hydrometer method were performed for each sample. The samples were divided according to soil classification into two groups depending on their classification–group A and group B. The results of laser diffraction analysis and hydrometer method were compared. Correlation relationships between both methods were established, and values were recalculated from laser diffraction analysis to the hydrometer method according to correlation equations. The article is a part of the methodology under preparation, which will include the most common soil types in the Czech Republic. This methodology focuses on the use of laser diffraction for the establishment of structures in forest environments using the regional specific standards for particle size distribution determination.

Keywords: establishment of buildings in the forest environment; geotechnical analysis; granulometry; optical methods; sedimentation methods; soil classification; soil texture

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1. Introduction

Soil texture is an elemental parameter for classification of soil. The texture indicates the content of the individual grain size fractions of soil. Particle size distribution (PSD) is one of the most important physical properties of soil. This is an important part of the analysis in the classification of soils for geotechnical purposes [1].

The distribution of soil particles of different sizes affects other soil physical and hydrophysical properties such as bulk density, soil water content, water holding capacity, permeability, and porosity [2–5]. PSD provides an initial understanding of the physical and mechanical behavior of soil, including for example workability, swelling and liquefaction potential, consolidation properties, and strength which are utilized in agriculture, forestry, and in many engineering geology applications [6]. Knowledge of these properties is also essential for the design of structures in the forest environment such as roads. Then, based on the exact classification according to the PSD, standardized characteristics important for structure design—such as strength, deformation, and permeability of soil—can be used.

The distribution of each particle size class can be determined by international standard procedure of PSD determination as using a sieving method or a combination of sieving and sedimentation techniques (for fine and mixed soils) [1]. The sedimentation process is mainly performed by hydrometer apparatus. Sedimentation methods apply Stokes' law to measure the suspension density for estimation of the sedimentation time of different particle sizes [7,8]. The hydrometric method is the only sedimentation method used for

classification according to ČSN (Czech Technical Standard) protocols, but it has several problems that reduce its accuracy. The analysis is time-consuming, at least 10–20 g of sample is required (a larger sample compared to laser diffraction analysis), and it is always necessary to take into account the personnel error that occurs during sample preparation or measurement [9].

As technology advances forward, new alternatives of PSD determination were developed. Most research and practical applications demand fast and unified methods, which offer reproducible and automated PSD measurements [10]. Certain attention is given also to indirect methods of PSD determination. For example, Yu et al. [11] presented a list of published works studying the potential of hyperspectral remote sensing for the estimation of soil texture. However, according to Mulder et al. [12], soil texture assessment from image data acquired by space-borne systems is still a complicated issue, mainly due to atmospheric distortions and the low spatial and spectral resolution of sensors. Laser diffraction analysis (LDA) is a laboratory optical method that is based on scattering of the light by suspensions, and it has been implemented since the late 1970's [13]. LDA has certain advantages in comparison to standard internationally accepted sedimentation methods, the (much) shorter analysis time being the most remarkable one. According to DiStefano, Ferro, and Mirabile [14], hydrometer methods usually combined with sieving do not give reliable results for particles smaller than 1 μm because of the effect of Brownian motion on the rate of the sedimentation. Lamb, 2013 in Gorączko and Topoliński [15], puts this threshold to 0.2 μm of ideal soil particles with spherical shape. The producers of the most current laser analyzers claim a wide measuring range, e.g., from 3.5 mm up to 1 nm for the Mastersizer 3000 laser analyzer [16] with the same measurement accuracy. LDA requires a much smaller amount of sample in comparison to the sedimentation method. However, some authors might see this as a source of uncertainty in the results because of the representativeness of the soil sample [17]. The analysis on the device is fully automated (except manual addition of sample) and directed by the standard operational procedure selected by the user. The use of this method in geotechnical engineering is not yet standardized and does not correspond to protocols. For the possible wider use of LDA in geotechnical engineering, the following should also be taken into account: Because the hydrometric method is used as a standardized method, equipment for this method is common in geotechnical laboratories and is relatively affordable. This is in contrast to laser diffraction equipment, which is in a different price range.

Since the sedimentation and optical methods are based on different principles, the PSD analysis does not result in the same representation of specific grain size fraction. The published literature provides an explanatory description of the differences and relationships between the results of PSD comparing the pipette method with LDA [5,18–22], the hydrometer method (ARM), and LDA [7,14,23–25]. However, most of these studies classified the samples using certain international soil textural classifications, such as the U.S. Department of Agriculture (USDA) [26] textural triangle or the Food and Agriculture Organization of the United Nations (FAO) [27] textural triangle. As Table S1 shows, the classifications can differ significantly when considering the particle sizes of soil separate classes with the same or similar name. While in soil science, knowledge of PSD provides sufficient information for soil type determination according to its texture, other applications, e.g., civil engineering requires additional tests such as evaluation of the Atterberg limits linked to the content of silt and clay within the soil. In the USA, a methodology by the American Association of State Highway and Transportation Officials (AASHTO) [26] and the Unified Soil Classification System (USCS) [28] is used, the latter being preferred for building construction. European countries adopt ČSN EN ISO 14688-1 [1] (Table S1), based on USCS. Differences in the classifications used can make it difficult to compare the performance and reliability of LDA for use in soil mechanic and civil engineering. Moreover, compared to soil science, geotechnical applications are more demanding regarding the number of evaluated soil separate classes often requiring measurements of numerous soil samples up to considerable depths. This is needed because the subsoil of the foundation significantly

affects the stability of any built structure. The structure support and the carrying structure of the object act as one static unit, and therefore, it is necessary to consider the subsoil of buildings as an integral part of every building structure and to know its mechanical physical properties. The foundation of every building structure and the subsoil interact with the construction. The soil is therefore the medium to which the forces from the foundation are transferred. This leads to a change in the tension state in the building foundation, subsequent deformations, or exceeding of the foundation bearing capacity. Up-to-date studies comparing the LDA and sedimentation methods for geotechnical applications are rarer [7] when compared to its applications in soil science.

Therefore, the article aimed to introduce the methodology of LDA application in the soil mechanics for forest roads and structures based on the carrying capacity of the soils. The aim of the article was to compare the two methods regarding the amount of sample needed for the analysis, as it was hypothesized that LDA can provide repeatable results with suitable accuracy besides smaller size of the soil sample. It was also hypothesized that it is possible to establish a dependency between the laser diffraction analysis and the hydrometer method for faster and easier classification for soil mechanics purposes.

2. Materials and Methods

2.1. Soil Sampling

All samples were taken in the Czech Republic, South Moravian Region. The sampling sites are located at an altitude of 200–300 m above sea level and belong to the climate zone T3 (warm, slightly humid) [29]. The samples of mineral soils were taken at five localities–Zbonek (1), Chrudichromy (2), Skalice (3), Rajec (4), and Herspice (5).

A 70 cm deep test pit was excavated during the collection. About 3 kg of soil sample was taken from the bottom of the pit. The samples were transported to the laboratory in plastic bags.

2.2. Soil Sample Preparation

To classify the samples according to the standard [1], it was necessary to perform the following tests: sieve analysis, hydrometer test, and consistency limits (plasticity and liquidity). Limits were determined using Atterberg tests. The liquid limit consists of determining the soil water content at which the soil changes from a plastic state to a liquid state. The plasticity limit stands for the soil water content at which the soil changes from a plastic consistency to a solid one. Tests for soil classification were performed according to ČSN EN ISO 14688-1 [1]. The same amount of soil sample was used for measurements by both methods and the same preparation was performed. Since the content of organic matter was negligible, the organic fraction of the soil was not removed. The sample was air-dried in the laboratory, sieved through a 2 mm sieve, weighed, put in a 150 mL beaker, and mixed with 20 mL of 0.05 M sodium hexametaphosphate. Distilled water was added to reach four-fifths of the total volume of the beaker and then stirred intensively for twenty minutes to achieve a perfect separation of the soil aggregates. All samples for the hydrometer were prepared and measured at the same time. Samples for LDA were prepared and measured sequentially, within a time limit of 24 h.

2.3. Hydrometer Method

Grain size measurement using the hydrometer (densitometric, areometric) method is based on the principle of sedimentation. The sample was rinsed through a 0.063 mm sieve with distilled water. The sample over the sieve was dried for 12 h at 105 °C and was sieved through a system of sieves (mesh sizes 2, 1, 0.5, 0.25, 0.125, and 0.063 mm). The undersieve fraction was poured into a measuring cylinder (volume 1 L), which was filled with distilled water. Then, the temperature and density of the suspension were measured at precise time intervals (2′, 5′, 15′, 30′, 60′, 120′, 240′, and 24 h) (Table S2). The density was measured with a Casagrande's densitometer [1]. The PSD was determined for 14 fictitious sieves (grain size fractions ranging from 1.55–2000 μm).

2.4. Laser Diffraction Analysis

The principle of laser diffraction consists of irradiating the sample particles with a laser beam, which is bent by the measured particles—this process is called diffraction. The light refraction angle is inversely proportional to the particle size—small particles scatter light at a large angle but with high intensity, and vice versa. The light amount that is determined in different directions is used to calculate the particle size, and the calculation depends on the sample refractive index and the medium (water, air) in which it is dispersed [30].

The LDA measurement was performed on a Mastersizer 3000 from Malvern Instruments Ltd. (Malvern, UK). Laser analyzer is composed of an optical unit, a dispersion unit, and a measurement cell. The wet dispersion unit was used. The measurement range was 0.01–3500 µm [16]. The particle refractive index was set to 1.457, and the Mie scattering model was used. Mie's theory considers all particles to be spherical; this assumption is comparable with ARM. The dispersant refractive index was 1.33, the particle absorption index was 0.01 and distilled water was used as a medium [23]. The device was operated by the selected predefined measurement sequence (standard operating procedure). For a valid measurement, the laser beam obscuration did not exceed 20%.

The sample was gradually pipetted from the beaker and measured in portions in the analyzer. The values of 14 individual fractions according to the fictitious sieves from the hydrometer were used for the measurement. To show the different demand of used methods for amount of soil sample needed, the same sample mass as for ARM was taken and did as many measurements of sub-samples (one subsample equals to one LDA measurement) until it has been used as much soil as for one ARM measurement. Therefore, 26–43 LDA measurements per sample were performed (Table 1). The weight of subsample per one LDA measurement was calculated as a ratio between the total amount of sample (same for both methods) and the total number of LDA measurements. While the weight of soil sample used for ARM ranged from 15.03–17.70 g, only 0.37–0.57 g of sample was needed for single-LDA measurement.

Table 1. Total number of LDA measurements and average sample weight per single LDA measurement.

Locality	Total Weight of Samples (g)	Total Number of Measurements	Single Sample Weight (g)
Zbonek (1)	15.84	30	0.5280
Skalice (3)	17.70	31	0.5710
Rajec (4)	15.03	26	0.5781
Chrudichromy (2)	15.49	39	0.3972
Herspice (5)	15.84	43	0.3683

2.5. Statistical Analysis

The results of all measurements were exported to MS Excel (v. 16.0) and processed. An average of all LDA measurements of individual samples was made, which was then compared with the value from the hydrometer separately for each grain size fraction (14 fractions). Correlation analysis was performed to find the relationship between LDA and ARM. In the final, the approximation was done by recalculation of LDA to ARM results according to derived correlation equations.

2.6. Verification of the Developed Correlation betweeen LDA and ARM

Three new control soil samples (V1–V3) were used for verification of the estimated relationships between LDA and ARM. These soil samples were provided by an external laboratory which also done the standard ARM test and shared the results using the same grain size fraction classifications as used in this study. Samples originated from localities in southern Moravia-V1 from Znojmo, V2 from Nove Mlyny and V3 from Brno-Herspice. PSD of control samples was estimated as an average of five measurements. This was a trial number of measurements, and it may change during the preparation of the overall

methodology. Relevant correlation equations developed for the sample of the same soil type (determined according to EN-ČSN classification) were used for recalculation.

3. Results

3.1. Soil Classification

The cumulative grain size curves of all samples are shown in Figure 1. Comparing the shape of the curves obtained by the ARM method (Figure 1a), the samples can be divided into two groups according to similar PSD. The first group (group A) comprised of samples from Herspice and Chrudichromy, while the second group (group B) consisted of samples from Zbonek, Rajec, and Skalice. Similar division between samples was also observed also for LDA but with overall smaller variation in PSD between individual samples (Figure 1b).

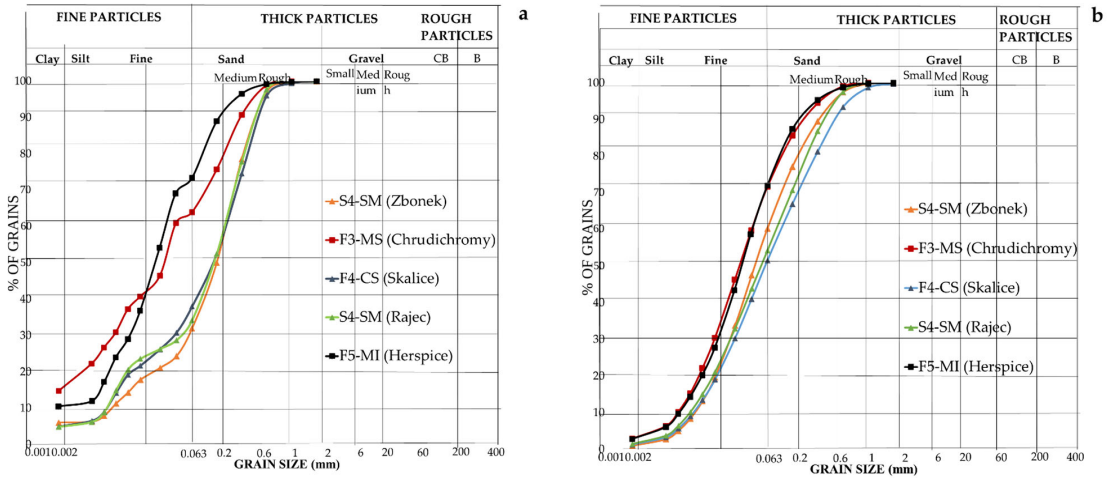


Figure 1. The grain size cumulative curve for all samples by ARM (**a**) and LDA (**b**).

For the classification of the soils used for the experiments, European regulations have been applied. Geotechnical analyses were carried out, such as soil water content tests (according to the protocol ČSN ISO/TS 17892-1 [31]), sieving tests (according to the protocol ČSN ISO/TS 17892-4 [32]), hydrometer tests (according to the protocol and consistency (plastic-liquid limit)), and Atterberg limit tests (according to the protocol ČSN ISO/TS 17892-12 [33]) at the Mendel University in Brno in the Laboratory of Department of Landscape Management, Faculty of Forestry and Wood Technology.

Table 2 presents the soil types when the samples were classified using various soil type classification systems. Using the geotechnical classification of soils according to the ČSN EN ISO 14688-1 [1] and ČSN EN ISO 14688-2 [34] and Unified Soil Classification System (USCS), the soil was classified as S4-SM and saSi for Zbonek (1), F3-MS and siSa for Chrudichromy (2), F4-CS and cISa for Skalice (3), S4-SM and saSi for Rajec (4), and as F5-MI and cISi for Herspice (5). According to the USDA soil science classification [26], all samples are types of loam. When taking into account the percentage representation of grain size fraction <0.01 mm (according to Complex Soil Survey in the former Czechoslovakia) [35]), all samples belong to the group of light soils. Furthermore, Samples 1, 3, and 4 can be classified as sandy soils (Group A), and Samples 2 and 5 can be classified as loamy sand soils (group B) [35].

Table 2. Soil type classification of the samples according to ARM.

Locality	Classification				Group for Article Purposes
	USDA	EN-CSN	USCS	CSS	
Zbonek (1)	Sandy loam	S4-SM	saSi	Sandy soil	Group A
Skalice (3)	Sandy loam	F4-CS	clSa	Sandy soil	Group A
Rajec (4)	Silt loam	S4-SM	saSi	Sandy soil	Group A
Chrudichromy (2)	Loam	F3-MS	siSa	Loamy sand	Group B
Herspice (5)	Silt loam	F5-MI	clSi	Loamy sand	Group B

3.2. Results of Laser Diffraction Analysis

For each soil sample measurement, the cumulative representation of 14 size fractions was calculated (<1.55 µm; <3.91 µm; <5.46 µm; <7.62 µm; <10.6 µm; <15 µm; <25.9 µm; <40.8 µm; <63 µm; <125 µm; <250 µm; <500 µm; <1000 µm; and <2000 µm) using the Mastersizer 3000 software. The general trend of the PSD cumulative curves for samples from five sites closely resembled what was observed at ARM, as shown in Figure 1.

Figure 2 presents the results of the repeated measurements for the selected cumulative size fractions by LDA. In general, LDA underestimated the distribution of fine particles when compared to ARM (Figure 2a,b). At cumulative size fraction <25.6 µm and above, the PSD determined by LDA was comparable with ARM for the same sample (Figure 2c–f).

When doing the repeated measurements of the same sample, the results of PSD by LDA slightly fluctuated what was expected. But from plots in Figure 2 it is clearly visible that the extent of changes in PSD of selected cumulative grain size fractions was increasing with repeated LDA measurements of the same sample. The first LDA measurements of subsamples were taken when the sample was taken from a full container. As the amount of sample in container decreased, it was harder to thoroughly mix the remaining sample before taking the subsample for LDA and was assumed that this might lead to higher variability in PSD observed after 14th measurement. Because of this, increasing variability was used only in observations for the first 14 repeated measurements per sample for further evaluation of the results.

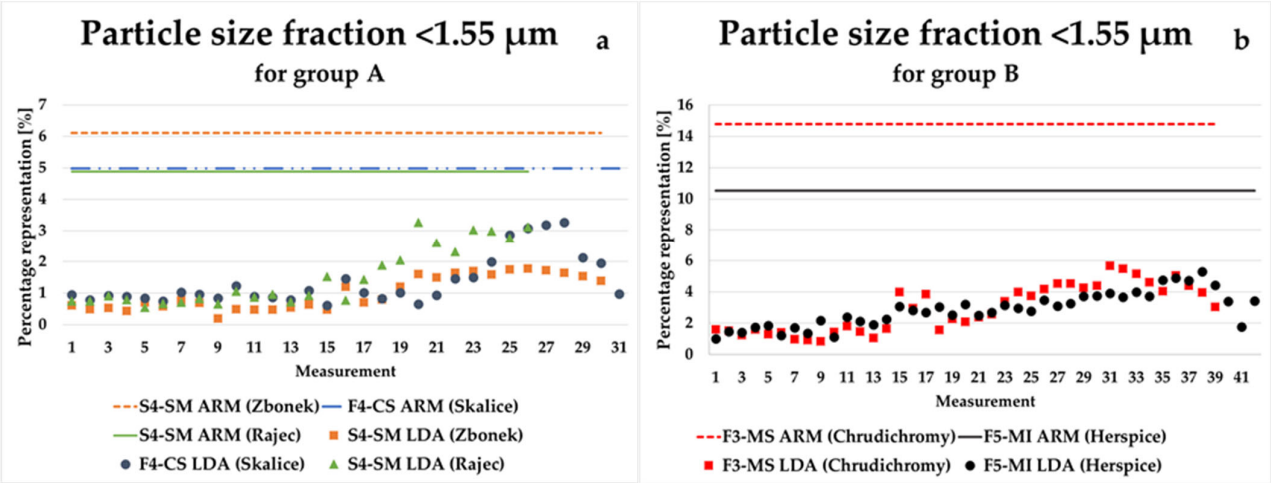


Figure 2. *Cont.*

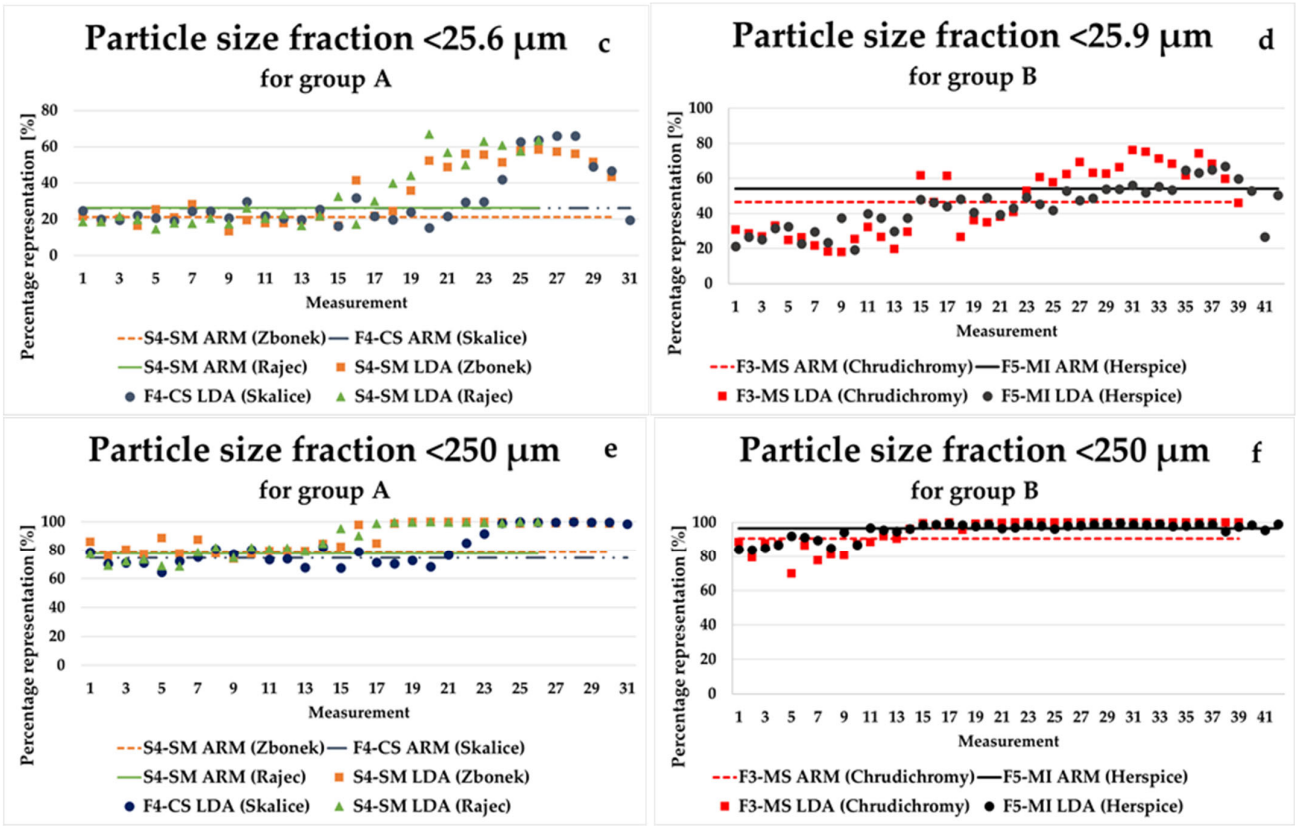


Figure 2. Percentage representation of cumulative particle size fractions <1.55 µm, <25.9 µm, and <250 µm in Group A soil samples (**a**,**c**,**e**) and Group B soil samples (**b**,**d**,**f**) as determined by the laser diffraction analysis (LDA) method and hydrometer method (ARM).

3.3. Correlations and Trends

The correlation between LDA and ARM and the variance of LDA values against ARM for individual fractions is shown in Figure 3. A similar trend was observed for all samples of Group A soils. The strength of the correlation ranged between 0.98 and 0.99. From the size fraction of 15 µm and higher, the LDA resulted in overestimation of the ARM value.

The correlation between LDA and ARM for group B soils is shown in Figure 4. These samples showed a similar trend. The calculated strength of the correlation ranged between 0.95 and 0.97, thus it was slightly lower than for group A soils. A larger percentage of LDA values was found below the relevant ARM values. Thus, LDA most underestimated the percentage representation of cumulative size fractions in comparison to ARM values.

The cumulative grain size curves for group A soils are shown in Figure 5. Along with the original LDA and ARM curves. Additionally, the recalculated values are given approximated to ARM from LDA using the correlation equations shown in Figure 3. The recalculation made the results of LDA to fit closer to the original ARM values. Around the 0.063 mm fraction, the new curve values were above the original ARM. The conversion worked best for fine particles.

Figure 6 shows the cumulative grain size curves for Group B soils. In comparison to Group A soils, the recalculated approximated curve according to correlation equation in Figure 4 resulted in better approximation of coarser particles representation. The fine particles are displayed rather inaccurately, which is due to the large difference in the original measurements. However, it should be noted that the correlation equations were not developed for large number of samples and thus the results currently present have only illustrative meaning.

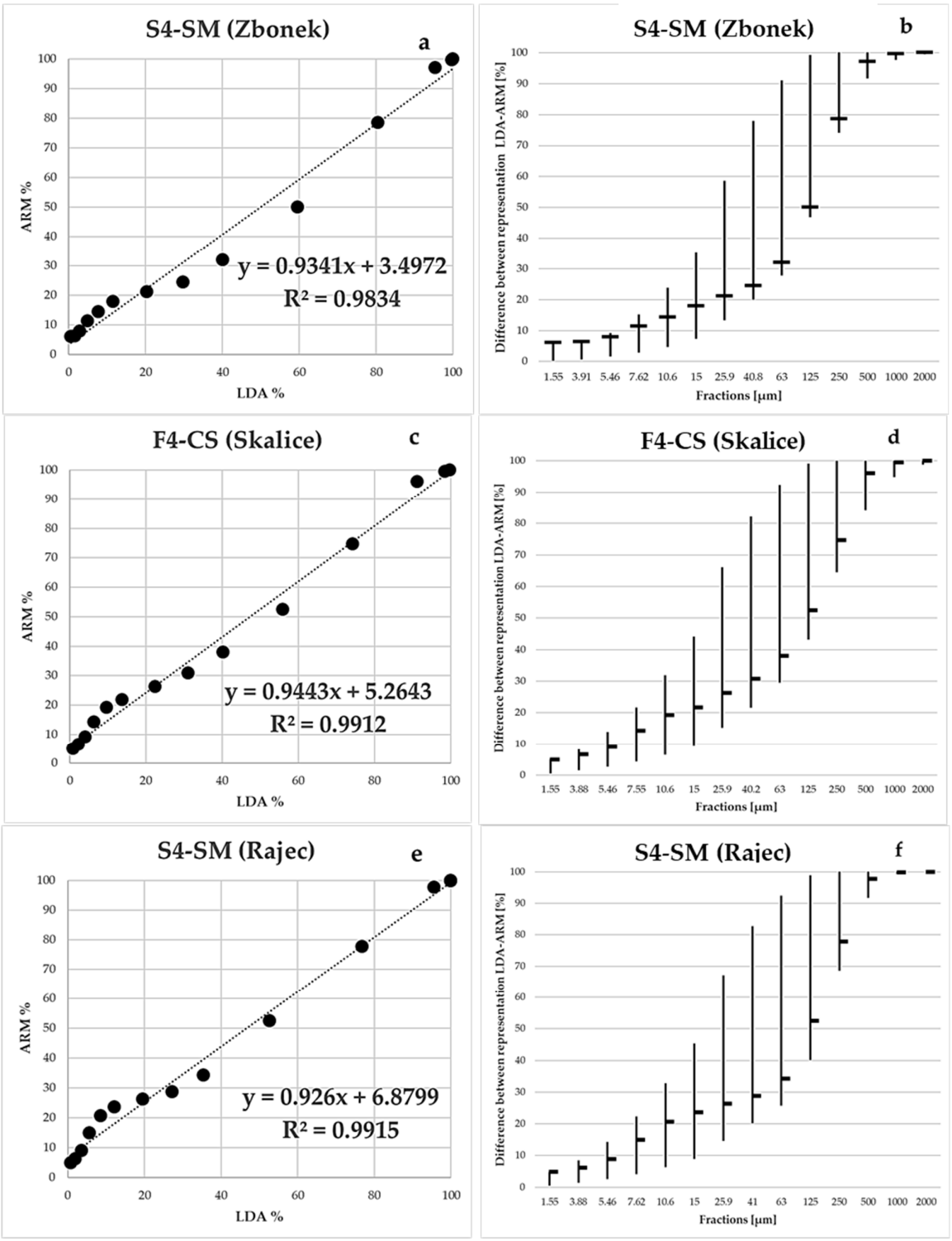


Figure 3. Group A soils: correlation between ARM and LDA for S4-SM (Zbonek) (**a**), F4-CS (Skalice) (**c**), and S4-SM (Rajec) (**e**) samples. The variance of LDA values against ARM for individual soil samples (**b**,**d**,**f**). The horizontal lines in plots represent the distribution of specific grain size fraction according to ARM, and the vertical lines represent the minimum and maximum values observed by LDA.

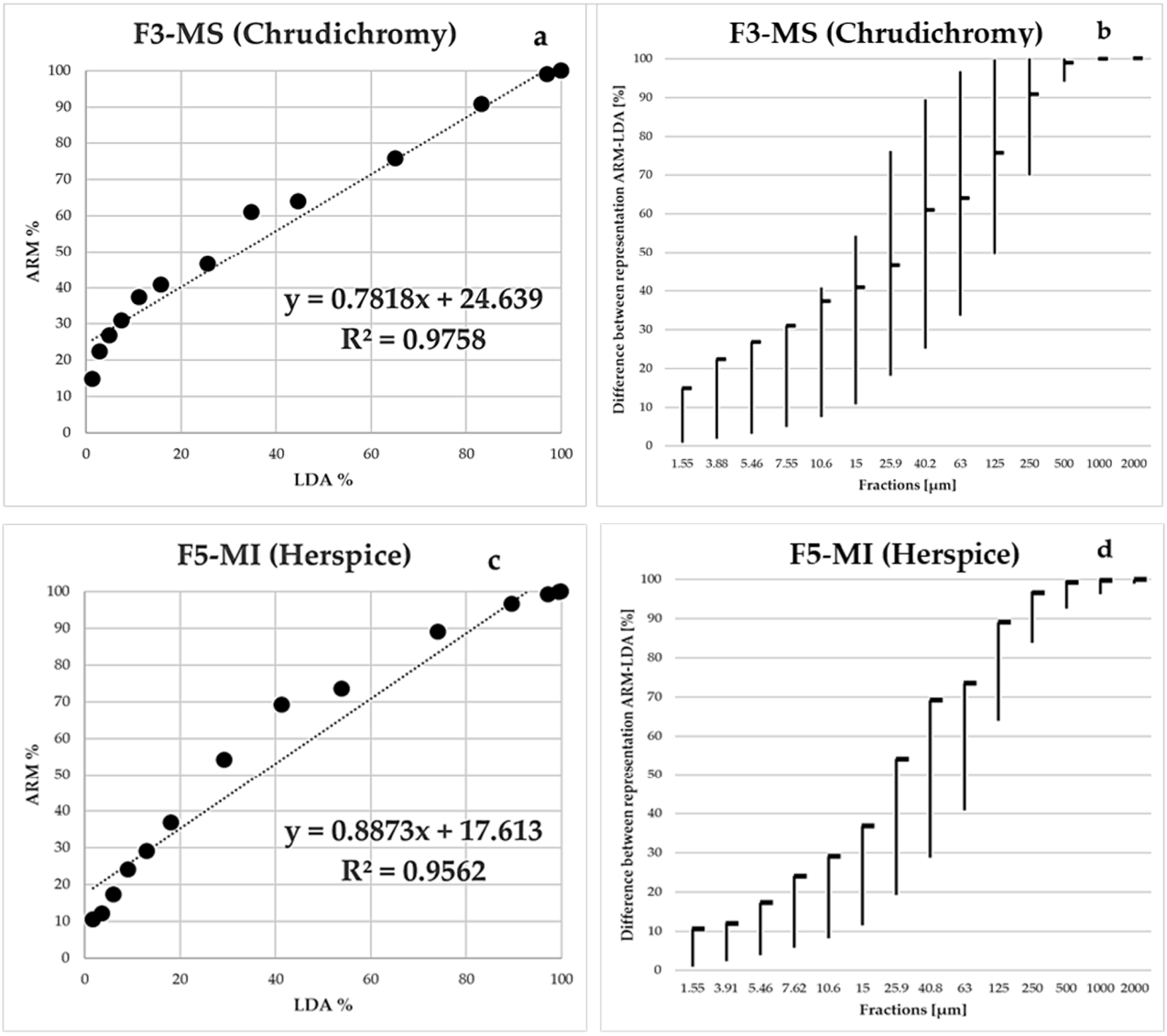


Figure 4. Group B soils: correlation between ARM and LDA for F3-MS (Chrudichromy) (**a**) and F5-MI (Herspice) (**c**). The variance of LDA values against ARM for individual soil samples (**b**,**d**). The horizontal lines in plots represent the distribution of specific grain size fraction according to ARM and the vertical lines represent the minimum and maximum values observed by LDA.

For evaluation of the relationships between ARM and LDA, only the first 14 LDA measurements were taken into consideration. However, in praxis, even 14 LDA measurements are not necessary. Similar regression coefficients between ARM and LDA were obtained when averages of PSD from only 3 measurements according to LDA were taken into consideration. In the Supplementary Material (Figure S1), the results show the average of the first measurements when the difference in the distribution of the cumulative size fraction up to 10.6 µm was less than 1.5% (Table S3). The threshold value of max 1.5% difference in the content of fraction up to 10.6 µm was chosen because similar variability is tolerated also for the standard sedimentation methods (below 1%–3% difference according to ISO 11277:2009(E) [36]). Similarly, high correlations (R^2 above 0.93) between LDA and ARM were observed when the samples were evaluated according to the two soil type groups. Once again, a similar strong relationship was observed when analyzing the average results from 14 or 3 LDA measurements (Figure S2).

Using the ČSN EN ISO 14688-1 [1]), the soil samples V1 and V2 were classified as F4-CS (same soil type as the Skalice sample), and V3 was F3-MS (same as the Chrudichromy

sample). Thus, a sample from both groups was available for comparison. Figure 7 shows that the recalculated values are not quite ideal.

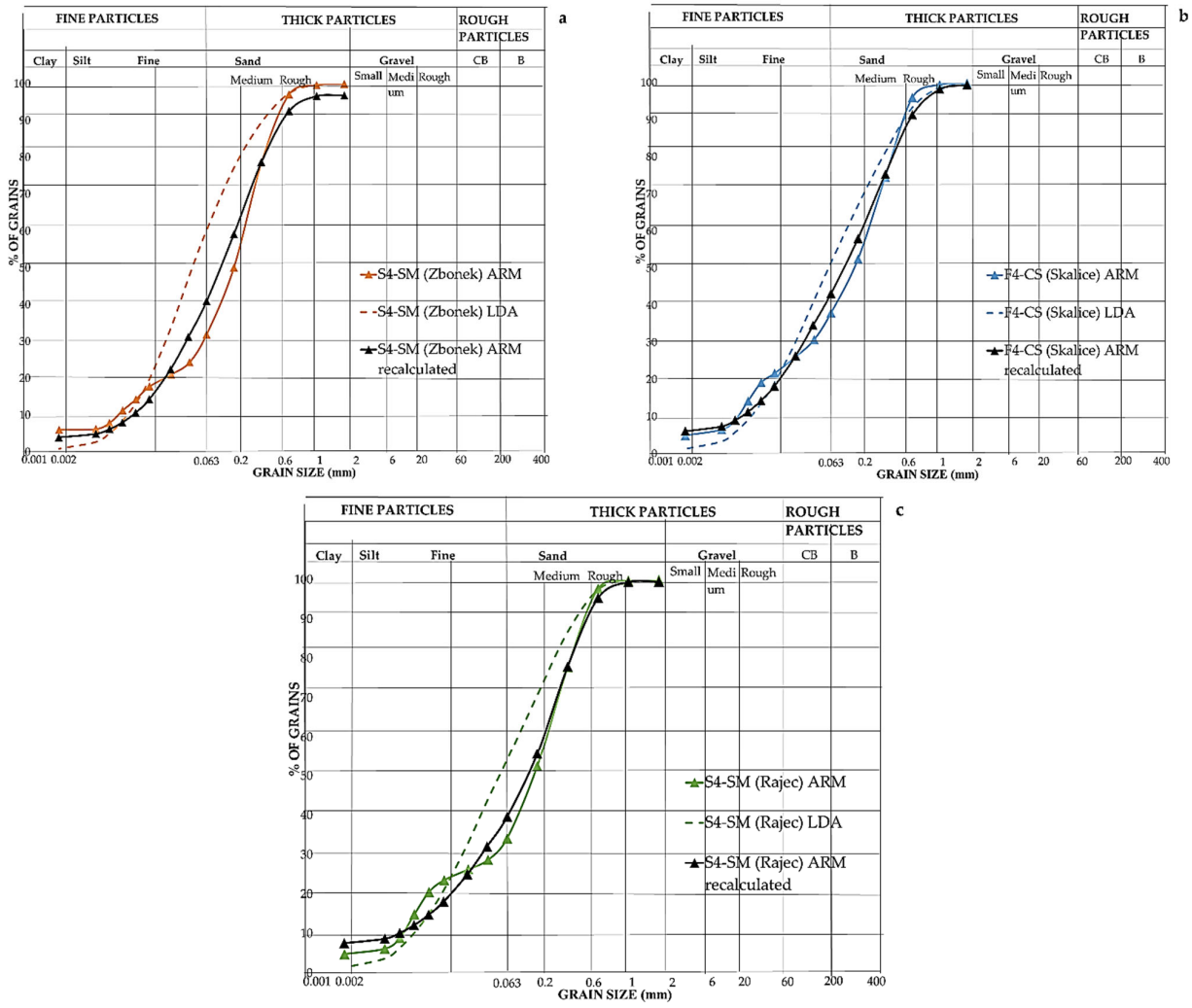


Figure 5. Grain size cumulative curve for ARM, LDA and recalculated ARM (according to correlation equations in Figure 3) for S4-SM (Zbonek) (**a**), F4-CS (Skalice) (**b**), and S4-SM (Rajec) (**c**) localities.

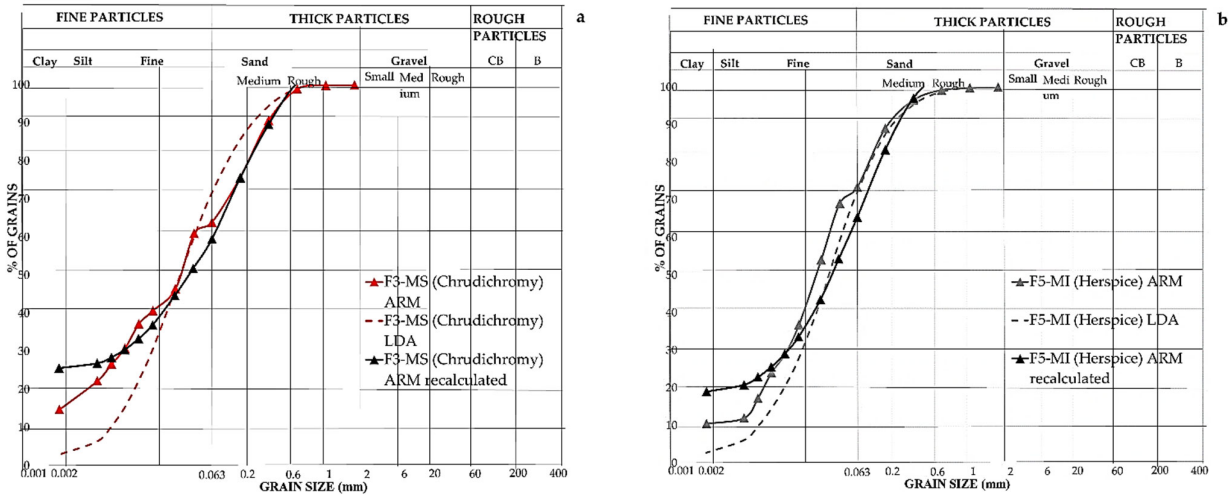


Figure 6. Grain size cumulative curve for ARM, LDA and recalculated ARM (according to correlation equations in Figure 4) for F3-MS (Chrudichromy) (**a**), and F5-MI (Herspice) (**b**) localities.

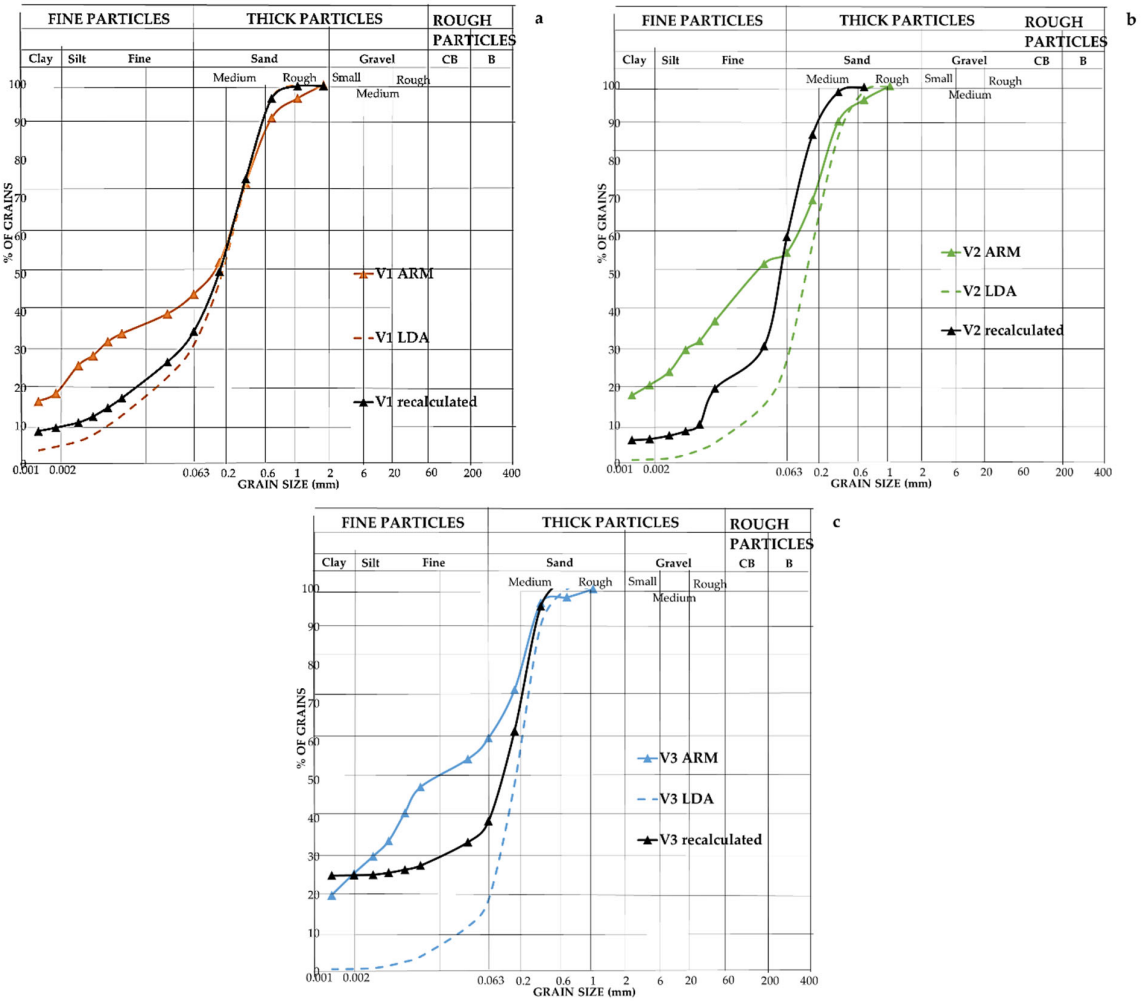


Figure 7. Grain size cumulative curve for ARM, LDA, and recalculated ARM (according to specific correlation equations in Figures 3 and 4) for verification samples V1 (**a**), V2 (**b**), and V3 (**c**).

The V1 sample came out the best, the V3 sample had the worst result. Thus, it is clear that further measurements need to be made to see for which soils the results are most optimal.

4. Discussion

This study was aimed to compare the results of PSD by ARM and LDA of soil samples prepared for analysis by the same procedure and at the same amount. The repeated measurements by LDA showed that the first measurements of the same sample resulted in similar PSD of the selected fractions and that for the specific amount of soil sample used in this study (around 15–18 g), no more than 14 repeated measurements should be taken. Otherwise, error due to not sufficient representativeness of the sample could be introduced in the regression analysis. Subsequent comparison of the relationships between LDA and ARM showed that the average of 3 repeated measurements with small variation are sufficient for the PSD determination (Figure S1 and Table S3). This conclusion is in agreement with other studies, as the authors usually used three repetitions of the sample [19,37]. In their study, Bittelli et al. [22] used four replicates per soil sample. In order to find LDA measurements fulfilling the given threshold value (the difference for the representation of size fraction < 10.6 µm to be below 1.5%), a maximum of six measurements (from the beginning of the analysis) had to be taken (Table S3).

Regarding the results for control samples (Figure 7), the difference between original ARM and recalculated ARM is due to the different origins of the used methods. Two

different methods using different physical principles were used (sedimentation for ARM and LDA for the recalculated values). The curve shape showing the recalculated LDA values to ARM values is smoother since the correlation equation with a linear trend was used for the calculation. Lower representation of the content of fine particles by LDA was confirmed for all samples in this study. This observation is in agreement with other previously published studies [5,7,37,38]. According to Di Stefano et al. [38], particle shape deviations from sphericity affect both LDA and ARM. In the case of LDA, an irregularly shaped soil particle reflects a cross-sectional area greater than that of a sphere with the same volume. Non-spherical particles in the ARM have longer settling times than spheres with the same volume, which results in overestimation of the clay fraction. Thus, the effect of shape works in opposite direction in LDA and sedimentation method.

Based on the results in this study, it is clear that for the specific amount of soil sample used (around 15–18 g), not more than 14 repeated measurements should be taken. Otherwise, error due to not sufficient representativeness of the sample could be introduced in the regression analysis.

Al-Hashemi et al. [7] compared LDA to the hydrometer method (ARM). They measured seven samples of different grain sizes (silt, clay, and colloids). The procedure of the hydrometer test was different than in Czechia—50 g of the sample was mixed with 125 mL of sodium hexametaphosphate (according to ČSN, the sample was mixed with 20 mL). The Microtrac S3500 LDS instrument was used. This analyzer can detect particles in the range of 0.02 µm to 2.8 mm (it is less accurate than a Mastersizer 3000, which measures in the range of 0.01–3.000 µm). They used dry and wet analyses of samples. The samples for the dry analysis were dispersed using two different pressures. For the wet analysis, Al-Hashemi et al. [7] used the same dispersing agents as for hydrometer analysis ($(NaPO_3)_6$), with ultrasound and distilled water. The sample feeding was performed using 2 mL disposable pipette droppers. The sample concentration was 1.4g/L. Al-Hashemi et al. [7] concluded that the differences between the two methods, from a geotechnical perspective, are qualitatively insignificant. Silt content results were slightly overestimated and highly correlated with each other (7.01%). For clay and colloids, the LDA and hydrometer results highly correlated with each other (5.49% and 6.81% for colloids and clay, respectively). The authors believe that it will be possible to replace ARM with LDA [7].

Lopez et al. [24] made comparisons for clay content in fine-grained soils. They measured 19 fine-grained samples from different localities in Finland with different grain sizes. In addition to the LDA and ARM, organic content analysis and specific gravity test were also performed. For the hydrometer test, they used 50 g of the sample with distilled water and 2.23 g of sodium hexametaphosphate. Measurements were performed on two analyzers—LS 13 320 by Beckman Coulter systems (a size range of 0.02 to 2000 µm) and Mastersizer 3000. For Mastersizer, the samples were mixed with 500 mL of deionized water with an addition of 0.5 g of sodium hexametaphosphate. Then, 150 mL of solution was used for the tests—one after stirring the solution for 1 h, another after 6 h, and the last test after stirring overnight. When using Mastersizer analyzer, Mie theory was used. In case of LS 13 320, the authors applied Mie and also Fraunhofer theory, which gave more accurate results for smaller particles, such as clay. Data were processed separately for each device. The result was a poor correlation between these two methods. The highest adjusted coefficient of correlation was 0.161 for Mastersizer. Poor correlation was between LDA results from LS 13 320 and Mastersizer on the same samples (0.402). Results from two different laboratories showed a good correlation for the hydrometer method (0.980). In conclusion, the authors mention in particular the need for the pre-treatment of the sample by ultrasonic techniques for the disaggregation of the particles because the simple use of a deflocculant as a pre-treatment seemed to have no effect for clay in LDA. They do not recommend LDA for geotechnics [24].

Yang et al. [19] compared LDA with scanning electron microscopy (SEM) and the pipetting method. They measured over 200 samples, and SEM randomly selected 100 samples. The samples were prepared with hydrogen peroxide and hydrogen chloride and

sodium hexametaphosphate was used for better dispersion. The Mastersizer 2000 laser analyzer was used for the analysis of samples dispersed in distilled water. Worse results were observed for silt (LDA underestimated clay fraction by a mean magnitude of 18.9%; silt was overestimated by 25.3%). The research results confirmed the suitability of the use of LDA instead of the pipetting method but pointed out that each soil is different, and further research needs to be continued in the future [19].

Ryżak and Bieganowski [25] compared laser diffraction with the hydrometer method. They worked with dry samples of sand, and they used the Mastersizer 2000. The dispersant was distilled water, and they measured 23 samples. Only three fine-grain-size fractions were compared: clay and silt up to 0.05 mm and sand up to 2 mm. Better correlations were observed for sand fraction, and worse correlations were observed for finer fractions, but they were still statistically significant [25].

Callesen et al. [21] published study on comparison of laser diffraction with sieve/sedimentation method. These authors used three sets of forest soil samples from three different European countries. The pipetting or hydrometric method and various dispersing agents were used for sample analysis. The procedure of the hydrometric method corresponded to the standard in force at the research country. Three laser diffraction instruments—Mastersizer 3000, Sympatec HELOS and Coulter LS230 were used in the comparison experiment. They also compared LDA between the 3 instruments/laboratories. In this comparison, the hydrometer was chosen as the sedimentation method. Using all three laser analyzers resulted in significantly lower clay content relative to the hydrometer method approach; only the sandy loam sample was the exception. Compared to the sedimentation method, LDA estimated lower clay fraction content and higher silt fraction content. It should be mentioned that a detailed comparison with results in this study is problematic because Callesen et al. [21] followed different standards for using the sedimentation method.

The results presented in this work do not compare very well to other articles dealing with the same topic. Some of the authors used different methods. In studies in which ARM was compared to LDA, different measurement procedures were used. Each country has set up its hydrometric measurement standards differently; the Czech standards are very specific. The differences in results can also be caused by the instruments used, which have different ranges and accuracies of measurement. In addition, the instrument settings also matter. In the final, the differences and problem of comparison with other studies is related to analyzed soil. In the case of the article measurements, the differences were not very significant, as all samples were alluvial soils (which were deliberately chosen, as this is part of the methodology under development).

5. Conclusions

The study presents a comparative analysis of determining particle size distribution (PSD) for five samples of typical soils coming from Czech Republic through the standard hydrometer method (ARM) and laser diffraction analysis (LDA). Correlation relationships between both methods were built and values were recalculated from LDA to ARM according to correlation equations for samples of two groups (Group A and Group B) that were determined according to the initial PSD results by ARM and classification of the soil types.

The methods differed in the requirements for the amount of sample needed for analysis. From the amount of soil sample required for one ARM analysis, 26–43 LDA measurements were performed. It was found that maximum of 14 LDA repeated measurements should be done out of 13–15 g soil sample to avoid excessive variability in the representation of the specific size fractions. Further it can be concluded that:

- LDA generally underestimated the content of fine grain size fractions. The difference was more obvious for samples in group B.
- Using the ARM and LDA measurements, strong regression relationships were estimated. Using higher (n = 14) or lower (n = 3) amount of LDA repeated measurements,

resulted in similar strong correlations with ARM. After recalculation, LDA usually overestimated the fine fraction for the group B samples when compared to ARM.

- The representation of grain size fractions of 0.001, 0.002, and 0.063 mm determined by LDA differed from the ARM measurement by an average of 8% and 15% for size fractions < 0.001 mm and <0.002 mm, respectively.

The developed correlation coefficients varied from 0.95 to 0.99 and showed a high dependence between the percentage values determined by LDA and the ARM conversion. When comparing only the own hydrometer test with LDA, after the LDA result conversion, the difference in comparison to ARM was up to 5%. This study is a part of the methodology under preparation which will include the most common soil types in the Czech Republic and which utilizes a grain size classification system that differs from standard classification systems in different regions. It can provide scientists and users with useful information on the reproducibility of the LDA results and show its potential to replace the hydrometer part in the standard sieve/sedimentation procedure of PSD analysis (for example soil mechanics purposes) in order to benefit from fast LDA analysis. On the other hand, this article provides a guide on how to recalculate the LDA results and make an approximation closer to ARM method, which is based on different physical principles.

However, it should be noted that the procedure of correlation relationships calculation in this manuscript was performed only for small sample dataset of sandy and loamy sand soils (according to Complex Soil Survey soil type classification). Therefore, the relationships determined for soil of Groups A and B in this study should be considered to have such limitations. To determine common correlation equations for a certain soil type or geographical region, a dataset as large as possible is strongly recommended to cover the variation in the soil samples related to the soil heterogeneity. For this reason, three control samples were measured but did not show clearly interpretable results. Currently, the hydrometric method is more applicable in the field of soil mechanics. It is determined by the standards and based on extensive research. In order to create an LDA methodology applicable to measurements in the Czech Republic, it is necessary to expand the sample dataset. Then, the users could benefit from the faster analysis time in LDA and the wide range of determined grain size fractions.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/f15020327/s1, Figure S1: The linear correlation between ARM and LDA for average of three LDA measurements.; Figure S2: The linear correlation between ARM and LDA for average of 14 LDA measurements and Group A (**a**) and Group B (**b**) and the average of 3 LDA measurements for Group A (**c**) and Group B (**d**); Table S1: Comparison of various sizes and names of soil separates according to various classification systems; Table S2: Temperature and reading time for determining the specific grain size fractions by the ARM method; Table S3: The percentage representation of particle size fractions for three selected measurements according to the LDA method.

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