IOP Conf. Series: Materials Science and Engineering **245** (2017) 072025 doi:10.1088/1757-899X/245/7/072025

Soil Particle Size Analysis by Laser Diffractometry: Result Comparison with Pipette Method

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Abstract. Soil texture as the basic soil physical property provides a basic information on the soil grain size distribution as well as grain size fraction representation. Currently, there are several methods of particle dimension measurement available that are based on different physical principles. Pipette method based on the different sedimentation velocity of particles with different diameter is considered to be one of the standard methods of individual grain size fraction distribution determination. Following the technical advancement, optical methods such as laser diffraction can be also used nowadays for grain size distribution determination in the soil. According to the literature review of domestic as well as international sources related to this topic, it is obvious that the results obtained by laser diffractometry do not correspond with the results obtained by pipette method. The main aim of this paper was to analyse 132 samples of medium fine soil, taken from the Nitra River catchment in Slovakia, from depths of 15-20 cm and 40-45 cm, respectively, using laser analysers: ANALYSETTE 22 MicroTec plus (Fritsch GmbH) and Mastersizer 2000 (Malvern Instruments Ltd). The results obtained by laser diffractometry were compared with pipette method and the regression relationships using linear, exponential, power and polynomial trend were derived. Regressions with the three highest regression coefficients (R2) were further investigated. The fit with the highest tightness was observed for the polynomial regression. In view of the results obtained, we recommend using the estimate of the representation of the clay fraction (<0.01 mm) polynomial regression, to achieve a highest confidence value R2 at the depths of 15-20 cm 0.72 (Analysette 22 MicroTec plus) and 0.95 (Mastersizer 2000), from a depth of 40-45 cm 0.90 (Analysette 22 MicroTec plus) and 0.96 (Mastersizer 2000). Since the percentage representation of clayey particles (2nd fraction according to the methodology of Complex Soil Survey done in Slovakia) in soil is the determinant for soil type specification, we recommend using the derived relationships in soil science when the soil texture analysis is done according to laser diffractometry. The advantages of laser diffraction method comprise the short analysis time, usage of small sample amount, application for the various grain size fraction and soil type classification systems, and a wide range of determined fractions. Therefore, it is necessary to focus on this issue further to address the needs of soil science research and attempt to replace the standard pipette method with more progressive laser diffraction method.

1. Introduction

In terms of practical and scientific evaluation, particle size distribution is an important feature of the soil, since it affects the whole range of soil properties. Therefore, it is important to include soil particle

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size distribution determination in each pedological or hydropedological survey. Individual soil particles are divided into bounded intervals by grain size, known as grain size fractions.

According to the percentage representation of one or more grain-size fractions present in the soil, individual soil types are identified. In Slovakia, soil type classification system according to Novak is standardly used. Regarding the grain-size fraction classification, system according to Kopecky and a comprehensive soil survey (CSS) are predominately used on the national level while USDA resp. FAO classifications are widely used abroad. The percentage distribution of grain size fractions can be determined by different methods based on different physical principles e.g. sedimentation (pipette, hydrometer), mechanical (sieve analysis) and optical methods (laser diffraction method, X-ray).

Laser diffraction has many advantages (short analysis time, high repeatability, low required sample volume, a wide measurement range and a wide range of sorted fractions). A different physical principle, on which the laser diffraction is based, causes a situation that the results of grain size analysis are not identical with the results according to sedimentation method, and thus the results determined by laser diffraction cannot be compared with the conventional method (pipet, hydrometer etc.) in a ratio of 1:1. Therefore, this fact often discourages professionals from accepting laser diffraction as a method of soil texture determination. Although the development of multiple regression models that allow conversion of the results of laser diffraction to values comparable to sedimentation method, the results are not applicable for Slovak conditions; firstly due to differences in methodological procedures for preparing the soil samples, usage of different measuring devices with different settings and secondly due to different grain size classifications used as a standard in the national level. The main aim of this paper was to compare the results of laser diffraction, using a laser particle analyser ANALYSETTE 22 MicroTec plus (Fritsch GmbH) and Mastersizer 2000 (Malvern Instruments Ltd) with the results obtained by pipette method. Evaluation of the measurement results was done using statistical methods. The paper presents the results under the current grain size classification system used in Slovakia, highlights the advantages and disadvantages of laser diffraction analysis and describes the preliminary development of methodological standards of particle size determination bylaser diffraction in Slovakia.

2. Materials and methods

2.1 Laser diffraction method

Size distribution of soil particles (presence of particles of a certain size with respect to all particles contained in the mass) is one of the most important soil properties, since it affects the properties of the soil, such as the distribution of the pores, water retention, thermal and sorption properties, and indirectly affects the nitrifying of soil [1].

Representation of the particle as a whole can be calculated according to the number, weight, volume and surface. Currently, there are several methods of particle dimension measurement available that are based on different physical principles. In the process of selection of an appropriate method several aspects should be taken into account. The selected method should allow the most objective measurement of the particle size distribution, which means that the selected method and apparatus should have a measuring range suitable for the particle size of measured sample. If the particle size range of the soil sample is wider than the measuring range of the device, firstly sieve analysis is carried out, or at least attempts should be made to separate the particles of a certain size at the site. In the obtained mixtures, the specific particle size fractions can be assigned to appropriate measurement methods (e.g. by sieve analysis to coarse particles and sedimentation analysis to fine particles or any of the optical methods) [2].

Laser diffraction method is an indirect optical method that is used to indirectly measure the particle size distribution according to dispersion of electromagnetic waves on the particles. For the calculation of particle size distribution, two different theories are used: Fraunhofer and the Mie theory. The Fraunhofer theory is acceptable only for determination of some particle sizes, since it describes the portion of light deflection that occurs exclusively as a result of diffraction. Very few particles have a disc shape; most of them are transparent and therefore Mie theory is generally more acceptable as the theory that accurately predicts the behavior of light scattering of all materials in all conditions (Figure 1). This theory was developed to predict the light scattering pattern by the spherical particles and deals with the way in which light passes through or is absorbed by these particles. This theory is more accurate, provided that user already has some specific information on the particle material properties, such as refractive and absorption indices [3].

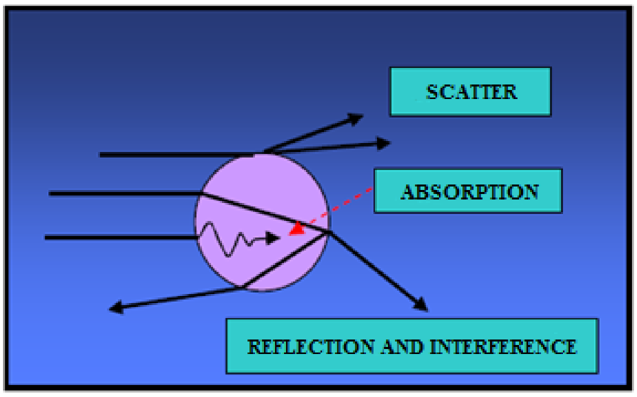

Figure 1. Laser beam diffraction according to the Mie theory, [4]

The Mie theory is used to calculate, the distribution of particles with a diameter smaller than the wavelength range of the light source if also refractive index and absorption coefficient are known or can be estimated. Larger particles with unknown optical parameters are calculated using the Fraunhofer theory, [10].

Laser diffraction is performed using various analyzers that have an individual measurement range and the results on grain size fractions distribution can be evaluated according to different classification grain size systems. The uniform composition and the process for the preparation of the soil sample is also not specifically defined, so many authors used differing processes, such as:

- W. Wang et al., 2013 [5] performed measurements on a Beckman Coulter LS-13 with 5 mW laser diode (wavelength: 750 nm). The average range was from 0.38 to 2000 microns. The soil was sieved through a 2 mm sieve, and 5 g of the sample was put in 100 ml cone, followed by sodium polyphosphate was used (the soils of pH> 7.5), sodium oxalate (soil with pH 6.7 to 7.5) and hydroxide bicarbonate (pH <5.5). Dispersion was carried out for 24 hours, and calculation was made according to Fraunhofer and Mie theory. The results of Mie theory was used but the laser diffraction method underestimated clay fraction by 35% and overestimated the dust fractions by 27%, which led to the conclusion that the values measured by laser diffraction method are not appropriate for determining the particle size distribution of the soil directly.

- Polakowski, 2014 [6] used the method of laser diffraction apparatus Mastersizer 2000 Hydro G dispersion unit (Malvern, UK), also optical microscope morphology G3 with software. Analysis performed on samples of sandy soil (0.05 to 0.01 mm) using potassium hexametaphosphate and sodium carbonate. Particle size distribution was measured three times in triplicate. The measuring time was set to 60 seconds for each measurement (30 seconds for the red light and 30 seconds for the blue light). The author concluded that the sandy soil containing the smaller particles in the range of 0.05 to 0.1 millimeters should be analyzed preferably by laser diffraction method, than sieving method.

- Kun et al., 2013 [7] used the Analysette 22 MicroTec plus (Fritsch) device. The analysis included 25 collected samples which were dried at 105 °C and passed through a 2 mm sieve. Samples were added to 0.5 g of sodium pyrophosphate and 400 ml of distilled water. The samples were then shuffled and added to a mixing apparatus for 6 hours. The homogenized samples were placed into ultrasound (f = 36 kHz, P = 60 W) for 3 minutes. The results on grain size distribution were obtained for the following grain size fractions: < 2 microns, 2-5 microns, 5-10 microns, 10-20 microns, 20-50 microns and > 50 microns. Pipette method overestimated the presence of sandy fractions in comparison with the results of laser diffraction method.

Methods for the soil sample preparation prior to laser diffraction analysis differ from author to author not only in the weight of the soil specimens, but also in organic matter and carbonate removal, the kind of used dispersing agent, respectively dispersion time, and application of ultrasound to break potentially present soil aggregates during measurement. Currently, many authors as Wang, 2013 [5], Polakowski, 2014 [6], Jena, 2013, [13] Kun, 2013 [7], chestnut, 2011 [1], Barasa, 2014 [14] and others, engaged in laser diffraction analysis and they compared the results with the standard pipette method. These authors were working with a different type of devices, as well as used various methods of soil sample preparation. To evaluate the results, they used a classification of soils based on the USDA triangular diagram, where the dimensions of grain size fractions differ from classification systems used in Slovakia (Kopecky classification, CSS classification). Although the laser diffraction method has many advantages, there is still no single methodology for the soil sample preparation prior to analysis. Another problem is that the distribution of the size fractions determined by laser diffraction is not comparable to conventional (sedimentation) methods in a ratio of 1:1 [8].

2.2 Preparation of soil sample

The study area of interest was the Nitra River catchment, where 111 sites with various soil types were identified. Soil sampling was performed and in total 222 samples were taken of agricultural soil from a depth of 15-20 cm and 40-45 cm. All locations were marked using GPS and subsequently identified in the GIS environment. For all samples pF curve and subsequent grain size analyzes were performed using pipette method [9]. The particle size distribution was evaluated as percentage representation 5 grain-size fractions (according to the CSS methodology), Table 1, [17].

Table 1. Comprehensive soil survey (CSS) methodology

2 - 0.25 mm	medium sand
0.25 - 0.05 mm	fine sand
0.05 - 0.01 mm	coarse dust
0.01 - 0.001 mm	medium and fine dust
< 0.001 mm	clay

From this soil sample database, 132 samples of medium fine soil were selected and used for the purposed of laser diffraction analysis. Before measurement, soil samples were taken in a laboratory, air-dried and the larger lumps of the soil gently crushed, in order to obtain fine soil. The air-dried soil was sifted through a sieve of 2 mm. To obtain a homogeneous soil sample of specific amount quartering method was used (Figure 2). From the prepared fine soil, 10 grams were weighted, and added to 10 ml of 0.05 M - sodium metaphosphate (Graham's salt $(NaPO_3)n$), to obtain a thick soil suspension. Dispersing time was 24 hours. Before the measurement, the samples were exposed to ultrasound for 5 minutes. The prepared sample was ready to be analyzed in the laser analyzer.

Figure 2. Procedure of quartering method

The analysis was performed using analyzers: ANALYSETTE 22 MicroTec plus (Fritsch GmbH) and Mastersizer 2000 (Malvern Instruments Ltd). The results obtained by laser diffractometry were compared with pipette method using the regression relationships.

2.3 Laser analyzers Analysette 22 MicroTec plus

Optimally dispersed sample is a prerequisite for the reliable determination of particle size distributions. It is necessary to set the correct concentration of the particles of the material sample. In principle, the dispersion process can be carried out in a stream of air (dry dispersion) or the liquid (wet dispersion). Dry dispersion is suitable especially for small, fine, well-flowing materials that react with water or other fluids. The required amount of dispersed sample for dry dispersion is significantly greater than for wet dispersion, which facilitates the preparation of a representative sample. Many materials must be measured by wet dispersion. They include sticky materials such as clay or materials that have a tendency to agglomerate when dry. Even very fine powders with a particle size less than 10 microns can agglomerate when being dispersed dry and often spread imperfectly. Therefore, wet dispersion is considered as a much more powerful and flexible. Using modular design of the device ANALYSETTE 22 MicroTec plus as well as measuring cells in the form of cartridges, it is possible to make a change in the measurement from wet measurement to dry measurement in a very short time [10]. Laser particle analyzer ANALYSETTE 22 MicroTec plus is composed of a unit for measuring the dry samples, the unit for measuring the wet samples and the measuring unit (Figure 3). In this case, the wet unit is comprised of 400 ml wet container with the dispersing liquid (tap water), in which a small amount of the sample is fed, a pump, mixer and the ultrasound unit.

Figure 3. Laser analyzer ANALYSETTE 22 MicroTec plus (left to right): unit for dry samples, measurement unit and the unit for wet samples

The ultrasound has a frequency of 36 kHz, and can be set from 0 to 10. Measurement range of the analyses is from 0.08 microns - 2000 microns in a single unit with a resolution of up to 108 channels. In the measurement unit, there are two sources of semiconductor lasers, inverse Fourier transformation optical system, detector, amplifier, and an evaluation and control unit that carries signals to the connected computer. The analyzer allows the combination of two lasers with two different distances from the measuring cell (Figure 4) and the detector [10].

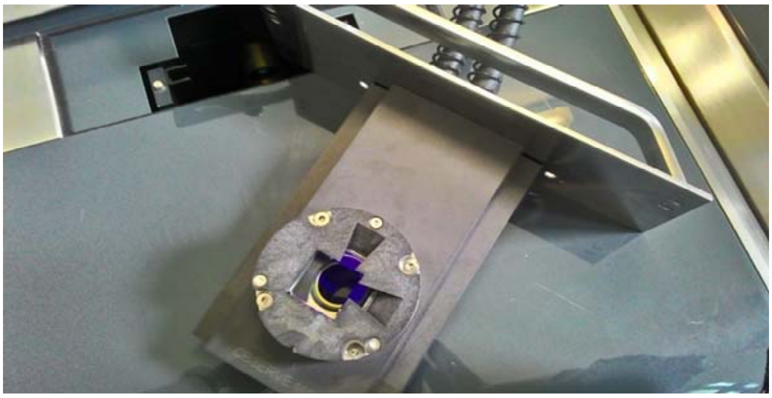

Figure 4. Measuring cell

Large particles are recorded by an infrared laser; and green laser is used for the small particles. The measurement by laser diffraction comprises an irradiation of particles spread in soil suspension with a laser beam. The partial deflection of the laser light intensity causes a distribution of rings, which is measured by a specially shaped detector. Particle size distribution is calculated from the spacing of these rings [10]. The device is controlled by instructions from MaScontrol software where the resulting values display directly on the screen in the form of cumulative curves, fractional histograms and tables showing the content of each soil fraction. Because all measurement results obtained with LD are stored in the MaScontrol application, it is possible to recalculate the analysis results while changing some specific conditions of analysis, such as different range of grain-size fractions, different calculation model without necessity to repeat the measurement [11].

At the start of the analysis, intensity of the scattered light in the dispersion liquid is measured in absence of particles. This step will detect any contamination of the measuring cell, which is then subtracted from the following measurement of the sample analyzed. A small amount of soil sample was charged into an ultrasound bath. Measurements were carried out over the full measurement range of the device; the results were estimated according to Fraunhofer theory and automatic calculation model. The pump device ensured smooth flow of suspension and dispersed the sample in the ultrasonic bath and in the measurement cell. Each sample was analyzed three times and the suspension was discharged from the dispersing unit into the outlet, dispersing unit was cleaned and prepared for the next measurement [12].

2.4 Laser analyzer Mastersizer 2000

The laser analyzer Mastersizer 2000 from Malvern Instruments Ltd. Company consists of an optical module that includes laser and detection unit. The optical module is connected to the computer unit, respectively personal computer, which processes the data and displays the output of the measurement results. Optical drives are connected to dispersing unit (Figure 5), whose main role is to dispense the prepared sample examined material in the optical module.

Figure 5. Laser analyzer Mastersizer 2000 (left to right): dry samples dispersion unit (Scirocco 2000), optical bench and the wet samples dispersion unit (Hydro 2000 MU)

Dry dispersion unit - "Scirocco 2000" is suitable for all kinds of unconsolidated materials (sand). The dispersion medium is air that flows through the dispersion unit, and carries the powdered material studied in the optical module. Wet dispersion unit - "Hydro 2000 MU", is best suited to determine soil texture. Dispersion medium is a liquid, where the sampled examined material is dispersed and circulated between the optical and dispersion unit [3].

When analyzing soil samples, their preparation prior analysis is important. The soil sample is dispersed to the required concentration in the dispersion system, and is conveyed to the optical module by means of a dispersion unit. In the optical module, the sample is fed through the measuring cell (Figure 6) that allows to be analyzed by the laser beam detector assembly and the detection unit captures a sample of the scattered light. Each detector collects light scattered from a certain angle [16].

The device is controlled by software Malvern SOP (standard operating procedure). When the SOP is running, it automatically performs the defined measurement processes, analyzes data, and indicates user to perform tasks such as adding soil samples. SOP has full control over dispersion unit and also performs all adjustments and settings such as pump speed and power of ultrasound. The resulting measurements can be displayed in various ways (graphs, histograms, tables). Measurement range is 0.02 to 2000 microns. The device uses the sources of red and blue light to perform the measurement. For a sampled material, refractive index is significantly different in red and blue light. This is affected by material's absorption ratio in these two wavelengths [3].

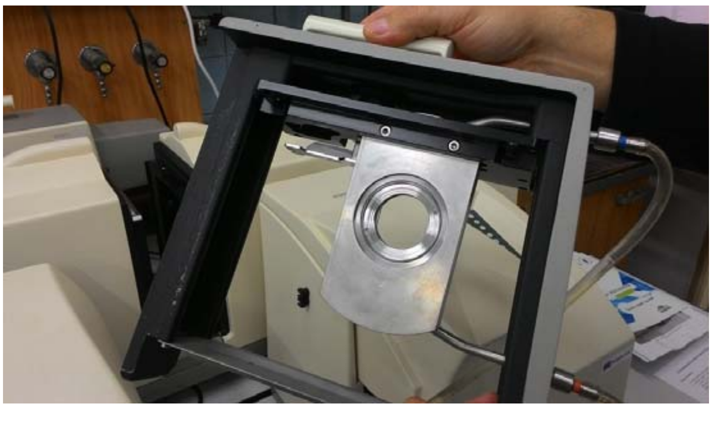

Figure 6. Measuring cell analyzers Mastersizer 2000

At the start of the analysis, intensity of the scattered light in the dispersion liquid is measured in the absence of particles. Pump speed, intensity and duration of ultrasound were manually adjusted. A small amount of soil sample was fed into a unit for measuring wet samples. The measurement was performed across the entire measurement range of the device according to the Fraunhofer theory. Each sample was analyzed three times, the suspension was manually drained; cleaned and dispersing unit was readied for the next measurement.

3. Results and Discussion

The analysis of 132 soil samples of medium fine soil, taken from the Nitra River catchment in Slovakia (depth of 15-20 cm and 40-45 cm) was performed using analyzers ANALYSETTE 22 MicroTec plus a Mastersizer 2000. Comparison of the distribution of grain-size fractions, as determined by laser diffraction (LD) and pipette method (PM) is shown in Figure 7 and 8. Then (PM and LD) regression dependence was derived between two methods using linear (LT), exponential (ET), power law (PLT), polynomial trend (PT) as well as the value of the reliability R^2 (Table 2, 3). Regressions with the highest reliability R^2 are marked in color.

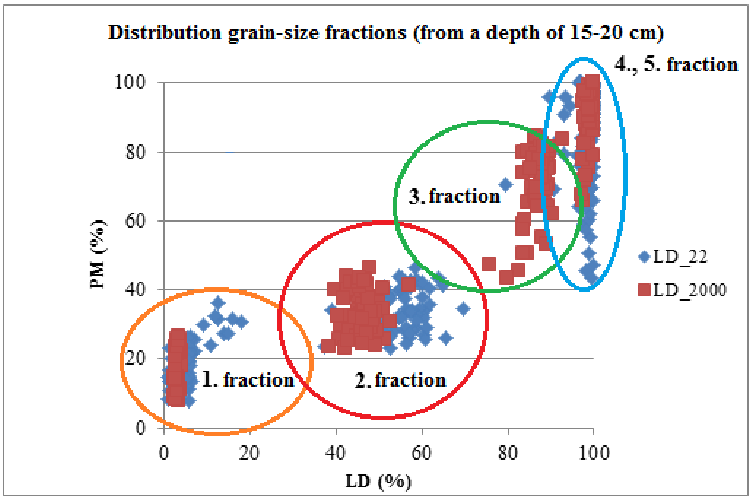

Figure 7. Comparison of the distribution of grain-size fractions, as determined by laser diffraction (LD) and pipette method (PM) for samples taken from a depth of 15-20 cm

Figure 7 shows a comparison of the percentage distribution of grain-size fractions, as determined by laser diffraction (LD), using analyzers Analysette 22 MicroTec plus (LD_22) and Mastersizer 2000 (LD_2000), and pipette method (PM) for samples from a depth of 15-20 cm. The display is in a ratio of 1:1 and the LD and the PM results were divided into five fractions, within the CSS methodology. The values obtained at LD_22 and LD_2000, compared with the PM, underestimated the representation of the first fraction of clay (<0,001 mm) and overestimated the third - coarse dust fraction (0.05 to 0.01 mm) and the fourth - fine sand fraction (0.25-0. 05 mm).

Figure 8 shows a comparison of the percentage distribution of grain-size fractions, as determined by laser diffraction (LD), using analyzers Analysette 22 MicroTec plus (LD_22) and Mastersizer 2000 (LD_2000), and pipette method (PM) for samples from a depth of 40-45 cm. The display is in a ratio of 1:1 and the LD and the PM results were divided into five fractions, within the CSS methodology. The values obtained at LD_22 and LD_2000, compared with PM, underestimated the first fraction of clay (<0,001 mm) and overestimated the third - coarse dust fraction (0.05 to 0.01 mm) and the fourth - fine sand fraction (0.25 - 0.05 mm).

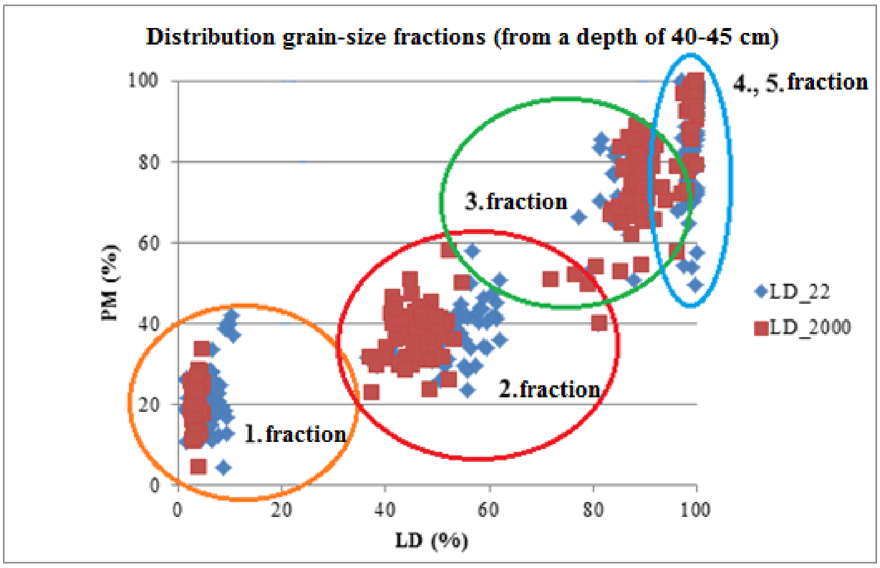

Figure 8. Comparison of the distribution of grain-size fractions, as determined by laser diffraction (LD) and pipette method (PM) for samples taken from a depth of 40-45 cm

Table 2. Regression dependence reliability and value for R^2 soil samples from a depth of 15-20 cm.

	Trend	Classification	R^2
Analysette 22	**Linear**	y=0.7014x+16.263	0.6936
	Exponential	$y=18.021e^{0.0157x}$	0.7385
	Power law	$y=8.2734x^{0.4872}$	0.7262
	Polynomial	$y=0.0068x^2-0.0392x+24.946$	0.7230
Mastersizer 2000	**Trend**	**Classification**	R^2
	Linear	y=0.8315x+6.9553	0.8910
	Exponential	$y=14.827e^{0.0184x}$	0.9287
	Power law	$y=8.5232x^{0.4806}$	0.8065
	Polynomial	$y = 0.0088x^2-0.1103x+17.841$	0.9489

Table 3. Regression dependence reliability and value for R^2 soil samples from a depth of 40-45 cm.

	Trend	Classification	R^2
Analysette 22	**Linear**	y=0.7211x+17.471	0.7479
	Exponential	$y=20.289e^{0.015x}$	0.7645
	Power law	$y=8.8141x^{0.4823}$	0.7226
	Polynomial	$y=0.006x^2+0.0655x+25.986$	0.7724
Mastersizer 2000	**Trend**	**Classification**	R^2
	Linear	y = 0.8111x+10.199	0.9049
	Exponential	$y = 17.431e^{0.0169x}$	0.9260
	Power law	$y = 9.5257x^{0.4639}$	0.8122
	Polynomial	$y = 0.008x^2-0.0531x+20.505$	0.9550

Of these relations were three selected with the highest reliability R^2. From a depth of 15-20 cm, these relations were exponential (ET), the power law (PLT) and polynomial trend (PT) for the device Analysette 22 MicroTec plus, and linear (LT), exponential (ET) and polynomial trend (PT) for Mastersizer 2000. From a depth of 40-45 cm it was linear (LT), exponential (ET) and polynomial trend (PT) for the Analysette 22 MicroTec plus and linear (LT), exponential (ET) and polynomial trend (PT) for the Mastersizer 2000. In the international studies the authors (Buurman, 2001[17]; Beuselinck, 1998 [18]; Blott, 2006 [19]; Miller, 2011 [20] etc.) used in LD different instruments with another measurement range, otherwise preparation of soil samples for analysis and results are evaluated for fractions of a different classification system (USDA). On this basis, it is not possible to compare our derived dependence between LD and PM for each grain size fraction and with foreign literature.

The selected relationships (Tables 2 and 3) were chosen for verification, when the derived relationships were used to calculate the estimated values according to LT, ET, PLT and PT trends. These estimated values were then compared with the results obtained by pipette method (PM).

For better illustration (of these differences were averaged) were average values for the fractions given in Table 4 (for samples from a depth of 15-20 cm) and Table 5 (for samples from a depth of 40-45 cm).

Table 4. Average values of calculated and measured data from a depth of 15-20 cm.

	Average	**< 0.001 mm**	**< 0.01 mm**	**<0.05 mm**	**< 0.25 mm**	**< 2.00 mm**
Analysette 22	**ET–PM [%]**	2.50	6.98	4.38	-13.90	-13.93
	PT–PM [%]	8.02	8.08	6.76	-11.55	-11.51
	PLT–PM [%]	0.31	20.95	0.55	-19.24	-22.17
Mastersizer 2000	**Average**	**< 0.001 mm**	**< 0.01 mm**	**<0.05 mm**	**< 0.25 mm**	**< 2.00 mm**
	ET–PM [%]	-1.23	0.73	2.48	0.20	-6.65
	PT–PM [%]	0.68	-2.41	3.81	1.71	-5.20
	LT–PM [%]	-7.43	11.32	8.23	-2.21	-9.90

Table 5. Average values of calculated and measured data from a depth of 40-45 cm.

	Average	**< 0.001 mm**	**< 0.01 mm**	**< 0.05 mm**	**< 0.25 mm**	**< 2.00 mm**
Analysette 22	**PT–PM [%]**	6.79	6.57	3.48	-8.06	-7.73
	ET–PM [%]	2.35	4.92	1.52	-9.71	-9.36
	LT–PM [%]	1.87	14.39	4.37	-10.05	-10.58
Mastersizer 2000	**Average**	**< 0.001 mm**	**< 0.01 mm**	**< 0.05 mm**	**< 0.25 mm**	**< 2.00 mm**
	PT–PM [%]	0.62	-2.32	3.85	1.21	-4.82
	ET–PM [%]	-1.28	0.57	3.23	0.45	-5.55
	LT–PM [%]	-6.69	10.10	7.54	-2.23	-8.71

To validate the results, the difference was determined from calculated (according to LD) and measured values (according to PM) for samples from a depth of 15-20 cm:

- in the case of the device Analysette 22 MicroTec plus the smallest difference was estimated for the fraction <0.001 mm, in the range from 0.31 to 8.02%, and the fraction <0.05 mm with values from 0.55 to 6.76%. High overestimation, respectively underestimation of the particle representation was determined at a fraction <0.25 mm (from 11.55 to 19.24%) and <2.00 mm (from 11.51 to 22.17%),
- in the case of the device Mastersizer 2000, the smallest difference was estimated for the fraction <0.01 mm, in the range from 0.68 to 7.43%, and the fraction <0.25 mm with values from 0.20 to 2.21%. High overestimation, respectively underestimation, of the particle representation was determined at a fraction <2.00 mm (5.20 to 9.90%).

For samples from a depth of 40-45 cm:

- the smallest difference regarding Analysette 22 MicroTec plus was estimated for fraction <0.001 mm (1.87 to 6.79%), and the fraction <0.05 mm, ranging from 1.52 to 4.37%. High overestimation, respectively underestimation, of the particle representation was determined at a fraction <0.25 mm (from 8.06 to 10.05%) and <2.00 mm (from 7.73 to 10.58%),
- the smallest difference regarding Mastersizer 2000 was estimated for fraction <0.01 mm (0.62 to 6.69%) and the fraction <0.25 mm, ranging from 0.45 to 2.23%. High overestimation, respectively underestimation, of the particle representation was determined at a fraction <2.00 mm (4.82 to 8.71%).

The results achieved by laser diffraction were compared with standard pipette method. In published works of authors, for example Buurman, 2001[17]; Beuselinck, 1998 [18]; Blott, 2006 [19]; Miller, 2011 [20] etc. used for laser diffraction different devices, with another measuring range and the results are evaluated for the classification of different fractions (USDA). On this basis, we cannot compare us derived dependence between LD and PM with foreign literature.

4. Conclusion

Using analyzers ANALYSETTE 22 MicroTec plus (Fritsch GmbH) and Mastersizer 2000 (Malvern Instruments Ltd.), analysis was performed on 132 samples of medium fine soil, taken from the Nitra River catchment in Slovakia, from depths of 15-20 cm and 40-45 cm. The results obtained by laser diffractometry were compared with pipette method and the regression relationships using linear, exponential, power and polynomial trend were derived. The average difference between the calculated and the measured percentage distributions for fraction of clay particles (< 0.01 mm) was 8% (Analysette 22 MicroTec plus) and 2% (Mastersizer 2000) for soil sample depth of 15-20 cm, respectively 7% (Analysette 22 MicroTec plus) and 2% (Mastersizer 2000) for soil sample depth of 40-45 cm). Since the percentage representation of clay particles (2nd fraction according to the methodology of Complex Soil Survey done in Slovakia) in soil is the determinant for soil type specification, we recommend using derived relationships in soil science when the soil texture analysis is done according to laser diffractometry.

In view of the results obtained, we recommend using the estimate of the representation of the clay fraction (<0.01 mm) polynomial regression, to achieve a highest confidence value R^2 at the depths of 15-20 cm 0.72 (Analysette 22 MicroTec plus) and 0.95 (Mastersizer 2000), from a depth of 40-45 cm 0.90 (Analysette 22 MicroTec plus) and 0.96 (Mastersizer 2000). The calculations suggest that either one will focus on the conversion of all fractions by CSS or for each fraction separately will be determined by separate conversion trends. Therefore it is necessary to focus on this issue further to address the needs of soil science research and replacing the standard pipette method with more progressive laser diffraction method should be attempted in the future.

Acknowledgment(s)
This study was supported by the Slovak Research and Development Agency under the contract APVV-0512-12 and the grand projects KEGA 019SPU-4/2017 and KEGA 026SPU-4/2017.

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