

PREPARATION METHODOLOGY OF SOIL SAMPLES FOR LASER DIFFRACTION

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Abstract

This paper introduces the methodology of preparation of samples medium heavy soils (20 – 45% wt.) for grain size analysis using laser diffraction. We focused on three different methods of preparation of samples that have been removed from the basin of the river Nitra. These methods are different in the amount used in the soil, by applying chemicals, distilled water and ultrasound energy level on the soil sample. Laser diffraction method provides a rapid solution to determine the soil particle size distribution especially in the analysis of large particles of soil samples. The advantage of laser diffraction is a short analysis of the samples, but this method does not introduce methodology of preparation of soil samples for analysis. The paper aimed to comparison of the results using a different method of preparation of samples soils with pipette method. The percentage distribution of grain size fractions were performed on the device analyzer ANALYSETTE 22 Micro Tec plus dispersing wet. Results of the particle size distribution, which resulting from the tree different methods of preparation of samples were compared with the standard pipette method.

Key words: grain size analysis, laser diffraction analyzers, Analysette 22, pipette method

1. INTRODUCTION

Particle size distribution is one of the most important soil characteristics, which influences many soil properties (Ryzak, Bieganski, 2011). The need for classification of soil particles according to their sizes granularity divided into intervals that are called grain size category. At home and abroad there are several classification systems based on differently defined thresholds granularity intervals. The basic division of fine soil particles and the skeleton (particles less than 2 mm) is the same in most systems (Skalová, 2016). The clay (0-2 μm), silt (2-50 μm) and sand (50-2000 μm), are three texture fraction which are commonly used to characterize the soil particle distribution of fine soil (< 2 mm) by classifying it into a soil texture class according to a soil texture triangle (Vandecasteele, B., De Vos, B., 2001). According to the percentage content of individual grain size fractions determine the soil types. For this purpose, it assembled several national and international classifications. To express grain soils with us most used Novak classification. The based on of content by coarse clay (fraction < 0.01 mm) determining the seven kinds of soil. The good thing about such a classification is a good clarity for users in practice. It allows fairly accurately classify soil types already in the field (Soil portal, 2016). The simplest determination of individual grain size fractions is using sieve method which commonly used to separate the sand fraction from the clay and silt fractions by drying and weighing the material remaining on the sieve (Xianlong Yang, 2015). The soil particle size distribution was mostly derived from the classical pipette method. The classical procedure of the pipette method for determining the soil particle size distribution includes sedimentation pipette method for clay, silt fractions and wet sieving for sand fractions. The pipette method gives precise and reproducible results; however, it is the longer process determining particle size distribution (Wei-Peng Wang, Jian-Li Liu, 2013). Distribution of grain-size fractions of soil can be determined also by optical methods. New methods of particle size analysis include laser diffraction method, where progress has been influenced by the development of computer technology (Jesenák, 2008). Currently, many authors as Wang, W., 2013; Polakowski, C., 2014; Jena, R., 2013; Kun A. 2013; Ryzak, M. 2011; Barasa, E. 2014, and others concerned with laser diffraction analysis and the results are compared with the standard pipette method.

The objective of this study was to compare the results of the particle size distribution, resulting from the three different methods of sample preparation, with a standard pipette method.

2. MATERIAL AND METHODS

2.1. Principles of laser diffraction methods

This optical method used for measuring the particle size distribution, scattering of electromagnetic waves on the particles. For the calculation of the particle size distribution are used, two different theories - the Fraunhofer theory and the Mie. The smaller the particle diameter in the wavelength range of the light source should be calculated using Mie theory, also particles of a lower refractive index and a low absorption coefficient. At low optical performance Mie theory should be used for the coarse particles of diameter over wavelength. Larger particles with unknown optical parameters are calculated according to Fraunhofer theory (Fritsch, 2016). Laser diffraction is performed using various devices (Analysette 22 MicroTec plus, MalvernMasterSiz E Coulter LS100, LS230 Coulter and others) that have a different range of measurement results for grain size fractions were evaluated according to different classification systems – Novak (Table 1), Kopecky (Table 2) and USDA (Fig. 1).

Table 1. Novak soil classification (Antal, Fidler, 1989)

Contains particles I. cat. (% wt.)	Identification of the type of soil by Novak	Name of the soil in terms of workability	Model of particle size distribution in the system BPEJ	
			5 - local	7 - local
0 – 10	Sand	Light soils	1	1
10 – 20	Loamy sand			
20 – 30	Sandy loam	Medium heavy – lighter soils	2	5
30 – 45	Loam	Medium heavy – heavier soils		2
45 – 60	Clayey loam	Heavy soils	3	3
60 – 75	Clayey soil			
> 75	Clay	Very heavy soils	4	4

Table 2. Classification of grain size fractions by Kopecky (Fulajtár, 2006)

The original version		A modified version		
Fraction name	Particle diameter (mm)	Fraction name		Particle diameter (mm)
Physical clay	<0,002	Colloidal clay		<0,0001
Fine silt	0,002-0,01	Physical clay		<0,001
Coarse silt	0,01-0,05	Fine silt		0,001-0,01
Powdery sand	0,05-0,1	Clay particles	I. fraction	<0,01
Sand	0,1-2,0	Silt	II. fraction	0,01-0,05
Skeletal code	>2,0	Powdery sand	III. fraction	0,05-0,1
		Sand	IV. fraction	0,1-2,0
		Skeletal code		>2,0
		Hail sand		2-5
		Chesil		5-10
		Coarse gravel		10-50
		Stone		>50

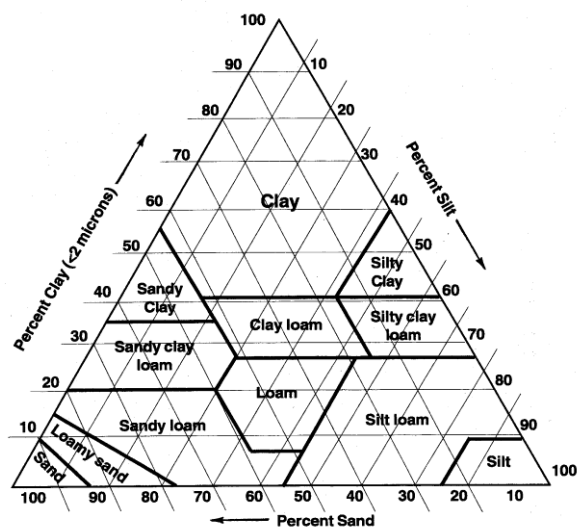


Fig. 1. USDA triangular diagram for the classification of types of soil (Soils, 2016)



Fig. 2. ANALYSETTE 22 MicroTec plus: measuring unit and wet dispersion unit, ultrasound
(Source: Šinkovičová, 2016)

Samples were analyzed by laser particle analyzer ANALYSETTE 22 MicroTec plus (by Fritsch GmbH, Idar-Oberstein) dispersing wet. The apparatus comprises a unit for measuring the dry samples, the unit for measuring the wet sample and the measurement unit (Fig. 2). In this case, the unit used to measure the wet, comprising a container having 300-500 ml dispersion liquid (tap water), to which is fed a small amount of the sample, also the pump, agitator, and ultrasonic unit. The unit of measurement, there are two sources of semiconductor lasers, inverse Fourier transformation optical system, detector, amplifier, an evaluation and control unit signals connected to the computer. The device is controlled by instruction from MaSControl that in addition to the control function ensures the evaluation functions and implements large-scale calculations of measured data stored in the database. Part of the wet dispersion unit is a measuring cell with an optically transparent flow cell, which is inserted into the measuring unit. Through the measuring cell passes a convergent beam which is diffused on the particles flowing through the cell and circulating together with the dispersion liquid. The concepts of convergent beam optics allow one to measure the particle size over a wide range from 0.08 to 2000 μ (Kondrlova et al., 2013).

2.2. Principles of the pipette method

Determination of grain composition of the soil pipetting method according to Novak, the soil aggregate disrupted Graham's salt, and then the sample is poured into the sedimentation cylinder, quenched with distilled water tempered to a particular volume. Pipetting the specific time and depth, the cylinder taken from a sample of the exact amount of evaporated water is collected from the sample, the sample can be dried, consider a computation, the percentage of the various fractions (Fiala, 1999).

2.3. Samples and sample preparation

The standard method of soil sample preparation for laser diffraction hasn't yet been specified. Soil samples have been removed from the basin of the river Nitra. Ten samples of medium fine soil were selected and used for laser diffraction analysis. Methods for preparing samples are derived from the processes used in standard sedimentation method. To create a homogeneous sample of soil was used quartering method (Fig. 3). Measured results and preparation method soil samples were averaged and compared with the results of standard pipette method. Authors such as Wang, W. et al., 2013; Kun et al., 2013; Ryzak and Bieganski 2011; Kondrlová, 2011, dealt with sample preparation procedure. To determine the method of sample preparation for mechanical analysis, we sampled were treated in the following ways:

1. method	The soil has been screened through a 2 mm sieve, quartering method are collected 10 g medium fine soil was mixed in 50 ml beaker with 10 ml Graham's salt ((NaPO ₃) _n) and covered 1 hour, ultrasound – 5 minutes.
2. method	The soil has been screened through a 2 mm sieve, quartering method are collected 10 g medium fine soil was mixed in 50 ml beaker with 10 ml Graham's salt ((NaPO ₃) _n) and covered 48 hours, ultrasound – 5 minutes.
3. method	The soil has been screened through a 2 mm sieve, quartering method are collected 10 g medium fine soil was mixed in 50 ml beaker with 10 ml Graham's salt ((NaPO ₃) _n) and covered 24 hours, ultrasound – 5 minutes.



Fig. 3. Quartering method (photo: Šinkovičová, 2016)

3. RESULTS AND DISCUSSION

3.1. Comparison of the results of laser diffraction (LD) and the pipette method (PM)

Each sample of soil was prepared by three different methods, which were then compared with the pipette method. Because all the measurement results obtained using LD are stored in database (program MaScontrol), it was possible to determine the grain size distribution for different size

fraction without need of another measurement. The measured results were averaged for each method and soil samples. Measured results by LD were averaged and compared with the results of PM by derivation of regression relationships with the linear, exponential and power law trend. The obtained values at LD, in comparison with PM, underestimated the distribution of clay fraction (<0.001 mm) and overestimated the distribution of silt fraction (0.001-0.05 mm) for all methods of soil sample preparation. Table 3 shows the calculated regression of (a number of trends) and R^2 values of reliability for each sample preparation methods for analysis LD. Regression of the highest R^2 for each trend are marked in color. In the above trends with the PM thus it correlates most of Method 3 is the preferred method of sample preparation for grain size analysis of medium heavy soils. This method correlated with PM in exponential trend the best ($R^2 = 0,922$ – method 3).

Table 3. Regression dependence and reliability R^2 value for each sample preparation to analysis by laser diffraction

Method of preparation	Linear trend	R^2	Exponential trend	R^2	Power law trend	R^2
Method 1	$y = 0,8253x + 6,0244$	0,866	$y = 15,551e^{0,0177x}$	0,915	$y = 4,7252x^{0,6127}$	0,834
Method 2	$y = 0,8377x + 45155$	0,859	$y = 15,016e^{0,018x}$	0,912	$y = 3,9508x^{0,6515}$	0,829
Method 3	$y = 0,8328x + 5,856$	0,879	$y = 15,568e^{0,0177x}$	0,922	$y = 4,6863x^{0,6156}$	0,836

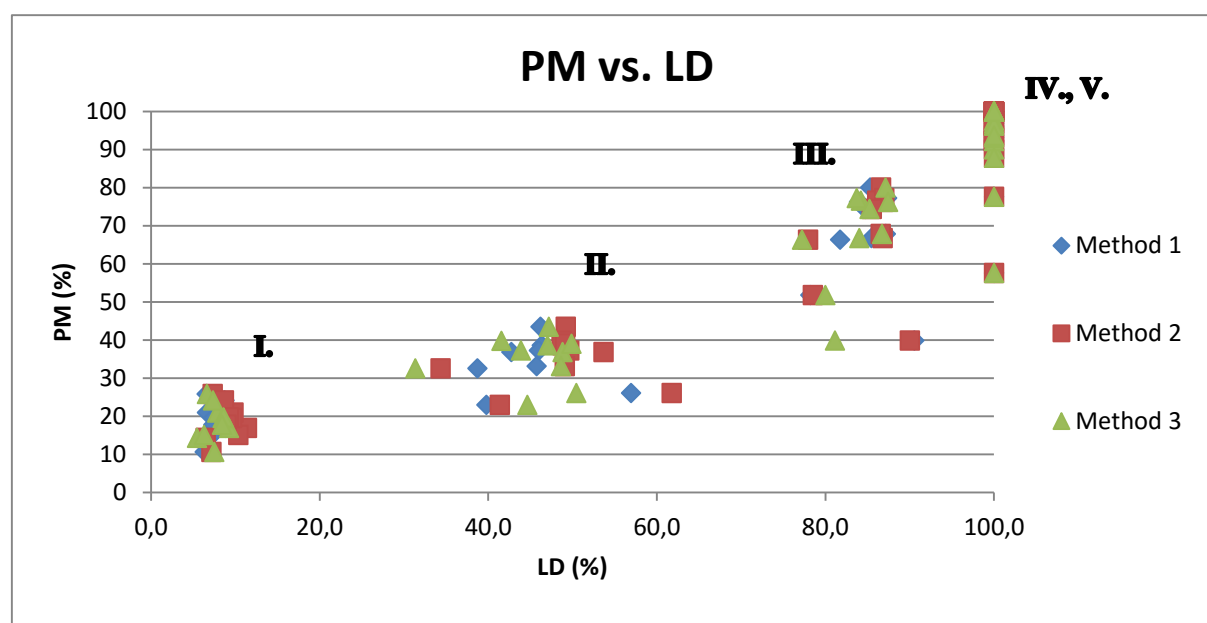


Fig. 4. Comparison of cumulative value of distribution granularity fractions as determined by laser diffraction and pipette method

4. CONCLUSION

The laser diffraction can use of the results for the purpose of different national soil classification systems without necessity to repeat the measurements and allows more detailed analysis of the particle size distribution. The results of the particle size distribution were compared to a standard pipette method; they were derived with the regression of linear, exponential and power law trend. The most preferred method of sample preparation for grain size analysis moderate soil appears to Method 3. At laser diffraction is possible to achieve relevant results and using a much smaller amount of sample than is possible with standard pipette method. It is also needed to test the accuracy of the methods on a large set of samples in various settings and parameters of the device (ultrasound energy level, measuring range).

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