# USE OF LASER ANALYSIS IN HYDROPEDOLOGY

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**Abstract**

*The main aim of the work was to analyse 176 samples taken from the Vah River catchment in Slovakia (from 15-20 cm and 40-45 cm deep) and 190 samples taken from the Nitra River catchment in Slovakia (from 15-20 cm and 40-45 cm deep) using laser analysers ANALYSETTE 22 MicroTec plus from Fritsch and MASTERSIZER 2000 from Malvern. 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 result was the percentage of the clay fractions (2nd fraction according to the methodology of the comprehensive soil survey in Slovakia) in soil is the determinant for soil type specification we recommend to use the derived relationships in soil science when the soil texture analysis is done according to laser diffractometry. The regressions with the three highest regression coefficients (R2) were then analyzed in more detail. The highest confidence level R2 was achieved on Analysette MicroTec plus with R2 = 0.53 for Vah catchment and R2 = 0.21 for Nitra catchment and on Mastersizer 2000 it was 0.69 (Váh) and 0.59 (Nitra).*

**Keywords:** *laser analysis, hydropedology, particle size analysis, soil*

## 1. INTRODUCTION

The soil is composed of grains of a different size, in terms of its practical and scientific assessment; the grain composition is an important feature because it affects the whole range of soil properties. The soil grain can be determined by laboratory methods (screen analysis, scrubbing, pipette, densitometry or optical methods). The results are in the form of tables, diagrams and grading curves. In the determination of soil particles by size, we have two basic grain categories, namely fine grain particles with a diameter of 2 mm and less, and skeleton - particles with a diameter above 2 mm [1].

Based on the percentage representation of characteristic grain categories (fractions) in the soil we determine soil species. In Slovakia we use the Novak classification, classification according to the Kopecky grading categories, the classification according to the methodology of complex soil survey (CSS), and abroad the most extensive classification of grain fractions according to Atterberg, classification USDA resp. FAO [2].

Technical progress allows the use of optical methods to determine the distribution of grain fractions of soil. They are divided into direct methods (photographic, electronic recording) and indirect methods (utilizing the relationship between particle sizes and diffuse radiation properties). The laser diffraction method is the most commonly used method of particle size analysis [3]. It has many advantages, such as short analysis time, high reproducibility, small sample size, wide range of measurement and range of fractions classification, but the laser diffraction method has a different physical principle, which results in soil particle size grain analysis results not identical to sedimentation methods. At present, many authors such as Wang, 2013 [5], Polakowski, 2014 [6], Kun, 2013 [7], Ryzak, 2011 [4] and others are working on laser diffraction analysis and compared the results with standard pipette method. Although several regression models have been made to compute the results of laser diffraction to values comparable to sedimentation methods, their results are not applicable to Slovak conditions. The differences are in the method of preparing the sample, in the used measuring equipment with different settings, as well as in the grading classifications used, which are not standard in our country. For this method, there is no methodology for the preparation of soil samples for analysis and another problem is

that the distribution of the grain fractions determined by the laser is not comparable with the conventional method in a ratio of 1: 1 [8].

The aim of the paper is to compare the results of laser diffraction using the ANALYSETTE 22 MicroTec plus and MASTERSIZER 2000 laser analyser and the standard pipette method. Use statistical methods to evaluate measured results, the classification into classification systems and elaboration of methodical measurement of soil grain-size by laser diffraction in conditions of Slovakia [12].

## 2. MATERIALS AND METHODS

There were used two laser analysers for soil analysis, the Analysette 22 MicroTec plus from Fritsch and Mastersizer 2000 from Malvern Instruments Ltd.

### *2.1 Laser analyser Analysette 22 MicroTec plus from Fritsch*

The laser analyser ANALYSETTE 22 MicroTec plus, from Fritsch, consists of a dry sampler, a wet sampler and measurement unit. The unit for wet samples with a 400 ml container was used. There is a dispersant fluid (tap water) in which a small amount of sample is dosed. Further, the apparatus includes *Analysette 22 MicroTec plus from Fritsch* a pump, a stirrer and an ultrasonic unit. The ultrasound has a frequency of 36 kHz and can be set to 0-10. The measuring range is from 0.08 microns - 2000 microns in one device with a resolution of up to 108 channels. In the measurement unit are two laser sources, the Fourier transform optical system, the inverse, the detector, the amplifier, the signal evaluation and control unit connected to the computer. The very wide measuring range of the ANALYSETTE 22 MicroTec plus allows the combination of two lasers with two different distances between the meter and the detector. Large particles are recorded by an infrared laser, for a small particle is used green laser [9]. The analyser is controlled by the MaScontrol instructions, where the resulting values are displayed directly on the screen in the form of cumulative curves, fractional histograms, and tables indicating the content of the individual soil fractions. Since all measurement results are stored in the MaScontrol database, it is possible to recalculate the results of the analysis when changing some of the analysis conditions, the representation of other grain fractions, another model of calculation, and there is no need to repeat the measurement [10] [12].

### *2.2 Laser analyser Mastersizer 2000 from Malvern Instruments Ltd*

This laser analyser consists of an optical module that contains a laser and a detection unit. The optical modem is connected to a computing unit or a personal computer that processes the data and displays the outputs of the measurement results. The Scirocco 2000 and Hydro 2000 MU dispersion units are attached to the optical unit. The wet dispersion unit "Hydro 2000 MU" was used for the analysis, which was used to determine soil texture. The soil sample is dispersed in the liquid (tap water), and circulates between the optical and dispersion units. The sample passes through the measuring cell, which is in the optical modem. The device is controlled by the Malvern SOP software (standard operating procedure). The resulting measurements can be displayed in different ways (graphs, histograms, tables). The measuring range is from 0.02 to 2000 microns. Mastersizer 2000 uses a red (large particle) and a blue laser (small particle) for measuring samples [11] [12].

### *2.3 Preparation of the soil sample for analysis*

Soil samples were dried in the laboratory and crushed. The air-dried soil was sifted through a sieve of 2 mm. To obtain a homogeneous soil samples was used quartering method. From the prepared 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. The dispersion time was 24 hours to interfere with the aggregates. Soil samples were exposed to ultrasonic bath for 5 minutes, prior to measurement. The analysis was performed using two analysers: ANALYSETTE 22 MicroTec plus (Fritsch GmbH) and Mastersizer 2000 (Malvern Instruments Ltd). The results obtained by laser diffractometry were compared with classical pipette method using the regression relationships [12].

## 3. RESULTS AND DISCUSSION

There were analysed 176 soil samples taken from the Vah River catchment in Slovakia and 190 soil samples, taken from the Nitra River catchment using analysers ANALYSETTE 22 MicroTec plus a Mastersizer 2000. Samples were taken from two soil depths (15-20 cm and 40-45 cm). We focused on the 2nd fraction (<0.01 mm) according to the methodology of complex soil survey (CSS) because we can determine the soil texture classes in Slovakia according to their percentage representation in the soil. Comparison of the distribution of grain-size fractions, as determined by laser diffraction (LD) and pipette method (PM) is shown in Figure 1and Figure 2. Then (PM and LD) regression dependence was derived between two methods using linear trend (LT), exponential trend (ET), power trend (PrT), and polynomial trend (PT) as well as the value of the reliability $R^2$ (Table 1, 2). Regressions with the highest reliability $R^2$ are marked in colour.

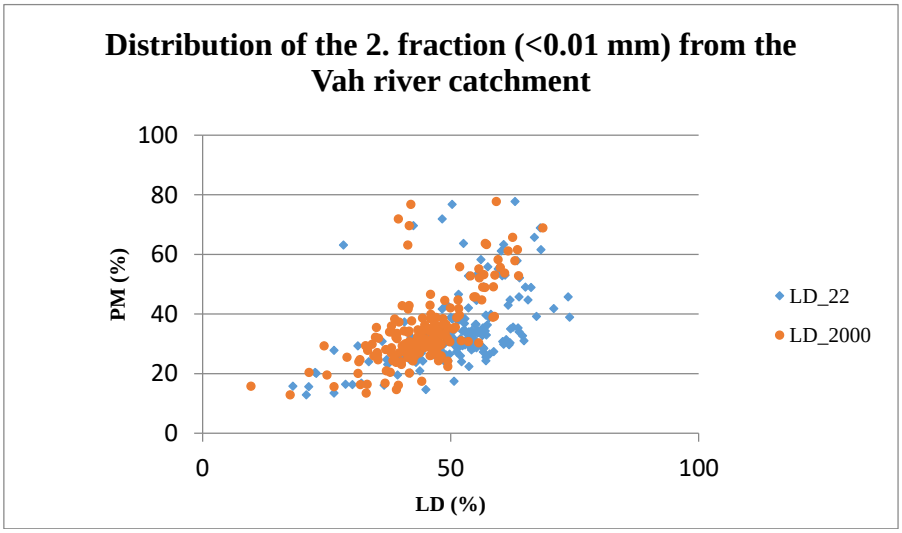



**Figure 1.** Comparison of the distribution of the 2nd fraction determined by laser diffraction (LD) and pipette method (PM) from the Vah river catchment.

**Table 1.** Regression dependence reliability and value for $R^2$ soil samples from the Vah river catchment

| | Analysette 22 | | Mastersizer 2000 | |
|---|---|---|---|---|
| **Trend** | **Classification** | **$R^2$** | **Classification** | $R^2$ |
| **Linear** | y = 0,6884x -1,2329 | 0,4041 | y = 0,9734x – 9,2049 | 0,5823 |
| **Expo.** | $y = 9{,}8659e^{0{,}0231x}$ | 0,5271 | $y = 8{,}6739e^{0{,}0295x}$ | 0,6187 |
| **Power** | $y = 0{,}7736x^{0{,}953}$ | 0,5208 | $y = 0{,}9792x^{0{,}9251}$ | 0,5090 |
| **Polyno.** | $y = 0{,}0014x^2 + 0{,}5573x + 1{,}5745$ | 0,4046 | $y = 0{,}0264x^2 – 1{,}1476x + 30{,}81$ | 0,6918 |

Figure 1 and 2 shows a comparison of the percentage distribution of grain-size fractions ((<0.01 mm), as determined by laser diffraction (LD), using analysers Analysette 22 MicroTec plus (LD_22) and Mastersizer 2000 (LD_2000), and pipette method (PM) from the Vah and Nitra river catchments.

In order to compare the relationship between PM and LD, regression dependencies were derived by linear trend (LT), exponential trend (ET), power trend (PT), polynomial trend (PT) and logarithmic trend (LT) , 2). Regressions with the highest reliability $R^2$ are marked in color.

For LD_22, the highest confidence level $R^2 = 0.53$ was recorded in the exponential trend for soil samples from the Vah River catchment. The highest value for samples from the Nitra catchment was $R^2 = 0.21$.

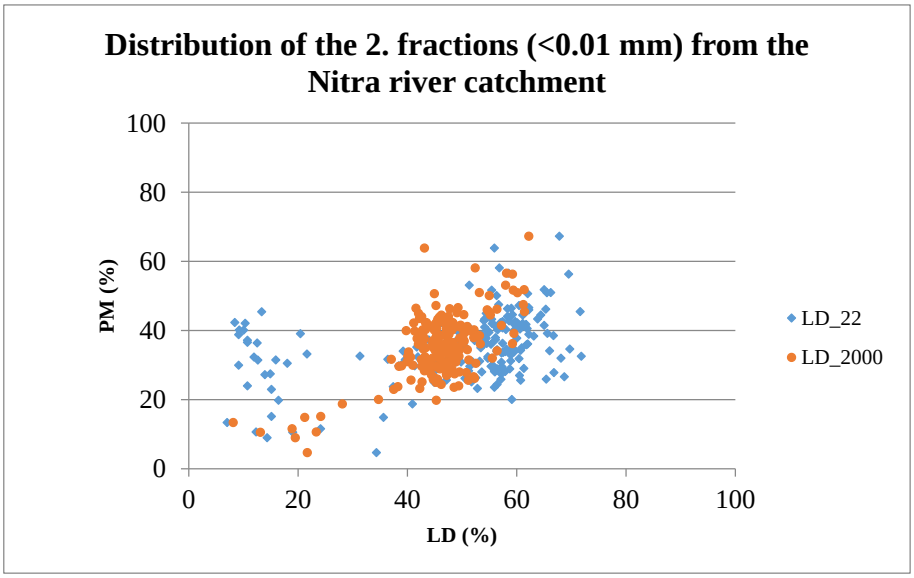



**Figure 2.** Comparison of the distribution of grain-size fractions, as determined by laser diffraction (LD) and pipette method (PM) for samples of the 2nd fraction from the Nitra river catchment

**Table 2.** Regression dependence reliability and value for $R^2$ soil samples from the Nitra river catchment

| | Analysette 22 | | Mastersizer 2000 | |
|---|---|---|---|---|
| **Trend** | **Classification** | **$R^2$** | **Classification** | $R^2$ |
| **Linear** | $y = 0{,}2158x + 25{,}205$ | 0,1912 | $y = 0{,}7555x + 1{,}087$ | 0,4870 |
| **Expon.** | $y = 22{,}983e^{0{,}0082x}$ | 0,2100 | $y = 8{,}6599e^{0{,}0299x}$ | 0,5847 |
| **Power** | $y = 14{,}726x^{0{,}2243}$ | 0,1810 | $y = 0{,}9795x^{0{,}9348}$ | 0,5782 |
| **Polyno.** | $y = 0{,}0044x^2 - 0{,}1099x + 29{,}2$ | 0,2045 | $y = -0{,}0018x^2 + 0{,}8935x - 1{,}3278$ | 0,4878 |

Subsequently, the average soil differences between PM and LD_22 and also between PM and LD_2000 were calculated for individual soil samples. Especially for samples from the Váh river catchment (Table 3) as well as samples from the Nitra river catchment (Table 4). The lowest average differences (LD-PM) were recorded for LD_22 PT = 1.89% (Váh) and ET = 0.39% (Nitra). For LD_2000 the lowest differences were recorded using LT = 1.14% (Váh) and MT= 0.28 (Nitra).

**Table 3.** Average values of calculated and measured data from the Vah river catchment

| Analysette 22 | | Mastersizer 2000 | |
|---|---|---|---|
| **Average [%]** | **< 0,01 mm** | **Average [%]** | **< 0,01 mm** |
| **ET – PM** | -2,82 | **LT – PM** | -1,14 |
| **PT – PM** | -1,89 | **ET – PM** | -2,02 |
| **MT - PM** | -2,97 | **PT –PM** | -1,21 |

**Table 4.** Average values of calculated and measured data from the Nitra river catchment

| Analysette 22 | | Mastersizer 2000 | |
|---|---|---|---|
| **Average [%]** | **< 0,01 mm** | **Average [%]** | **< 0,01 mm** |
| **LT – PM** | 0,69 | **ET – PM** | -0,29 |
| **ET – PM** | -0,39 | **PT – PM** | 0,53 |
| **PT - PM** | 0,35 | **MT –PM** | -0,28 |

## 4. CONCLUSIONS

There were analysed 176 soil samples taken from the Vah River catchment in Slovakia and 190 soil samples, taken from the Nitra River catchment using analysers ANALYSETTE 22 MicroTec plus a Mastersizer 2000. The results obtained by laser diffraction were compared with standard pipette method using the regression relationships. From these relations, 3 were chosen with the highest confidence values $R^2$. The result was the tightness of the derived relationships for the 2nd fraction (<0.01 mm), because according to their percentage representation in the soil, soil texture classes are determined in Slovakia (Novak classification). In view of the results obtained, for the samples of Vah catchment, we recommend for estimation of the representation of the clay fraction (<0.01 mm) exponential regression, where the highest value of reliability $R^2$ was 0.53 (the analyzers of Analysette 22) and a polynomial regression of 0.69 (for the Mastersizer 2000 laser analyzer). In case of the Nitra river catchement, we recommend exponential regression using the Analysette 22 laser analyzer, where the highest $R^2$ value was 0.21. For the Mastersizer 2000 laser analyzer, we recommend using exponential regression where the $R_2$ value was 0.59. The laser diffraction method is a new method for determining the distribution of soil particle size. It is advantageous in terms of short analysis time, the use of a small amount of sample, the use of results for the purposes of different national classification systems, a wide range and classification of fractions as compared to the conventional pipetting method. Nevertheless, it is still necessary to address this issue for the needs of hydropedological research and try to replace the standard pipetting method in the future by another, for example, laser diffraction method.

## ACKNOWLEDGMENTS

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