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PRINCIPLES OF SOIL PARTICLE SIZE ANALYSIS BY INDIRECT OPTICAL METHOD: ADVANTAGES AND DISADVANTAGES OF LASER DIFFRACTION ANALYSIS

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Abstract

Advances in computer technology have led to progressive development of optical methods. Optical methods can be divided into two groups: direct and indirect methods. The first group includes those methods that directly analyse photographic or electronic image record. The indirect methods use the relationship between particle size and properties of the scattered light. Laser diffraction uses scattering of electromagnetic waves on the particles to measure the particle size distribution. Angle of scattered light is inversely proportional to size of particle at which the incident light beam is scattered. This study presents the basic principles of laser diffraction, particle size analysis theories and design of a laser particle sizer ANALYSETTE 22 MicroTec plus (FRITSCH). Further, the advantages and disadvantages of this method are present in comparison with pipette method.

Key words: *particle size analysis, optical methods, laser diffraction, ANALYSETTE 22 MicroTec plus*

1 INTRODUCTION

Content of mechanical particles with mineral origin that have various shapes and sizes as well as different mineralogical and chemical composition determines the soil texture. Mineral soil particles of defined size are aggregated into groups with a specific name of grain size fraction. Every soil fraction consists of particles with similar dimensions and in addition, some of their basic physical and physico-chemical properties are more or less identical (Fazekašová & Torma 2007; Halaj & Bárek 2009). Quantitative representation of these fractions in the soil, expressed as a volume or mass percentage, is called the soil texture. Fine soil (particles less than 2 mm) is an essential component of soil. The grain size affects all the basic soil properties of the soil, e.g. porosity, air and water capacity, water and air permeability, adhesion, plasticity, soil sorption, and soil temperature (Bárek, V, Halaj, P & Báreková, A 2007; Čimo, J & Špánik, F 2009; Fazekašová et al. 2011; Ivanová et al. 2012). It is an important parameter in the understanding of sedimentary environments and depositional processes, as well as for pollutant transfer and studies of geochemical cycles (Loizeau et al. 1994)Knowledge on the size distribution of soil particles is used for land use proposals, in the crop cultivation as well as for determining soil tillage and fertilization (Fulajtár 2006; Čimo & Špánik 2010). Nowadays, there are present various methods of determining the percentage of particle size fractions that differ mainly by the type and amount of sample used for the analysis, demands on sample preparation, speed of analysis and processing of the results, the price of instrumentation and measurement range (minimum and maximum particle size) (Jesenák 2008). The classical techniques of particle counting, sieving, and settling are still in use though often using automated instruments. New ways of sensing old variables, such as discriminating and counting grains by image analysers, have also been developed. In addition there are some completely new principles in use – laser diffractometry and photon spectroscopy (Agrawal, McCave & Riley 1991). Sieve analysis is the simplest way of determining the percentage representation of individual fractions. It is also used for the pretreatment of samples for sedimentation

analysis. The most commonly used sedimentation methods include pipette method, hydrometer method according to Cassangrande and decantation method. Another group of methods to determine the distribution granularity of soil fractions are optical methods. These include traditional methods of optical microscopy, which became available shortly after the invention of the optical microscope (in 1590) as well as very modern and highly sophisticated techniques, using highly specialized instrumentation. Optical methods can be divided into two groups: direct and indirect methods. The first group includes those methods that directly analyse photographic or electronic image record. The indirect methods use the relationship between particle size and properties of the scattered light. In the latter case, the particle size is derived based on the theoretical model that characterizes this relationship. While the first group of methods can be used in almost all cases of particle size analysis, the second group of methods is used mainly for analysis of particulate matter (Jesenák 2008). Laser diffraction uses scattering of electromagnetic waves on the particles to measure the particle size distribution. Angle of scattered light is inversely proportional to size of particle at which the incident light beam is scattered. The partial deflection of the laser light results in a characteristic, ring-shaped intensity distribution behind the sample which is measured by a specially shaped detector. This image of the scattered light is called Fraunhofer diffraction pattern, in the middle there is located the zero maximum and then the course decreases sharply. Measured particle size is calculated based on the size of the gaps between the circles: large particles form rings with smaller spaces, small particles form rings with bigger space. Analysis of a large set of particles is based on the fact that particles of the same size are converging light beam to the same point.

An important methodological problem associated with determination of the particle size distribution by any method is the preparation of soil samples. Ryżak, Bieganowski & Walczak (2007) defined a number of common methods of soil sample preparation: mechanical (mixing various mixers, using an ultrasonic bath) and chemical (adding of appropriate detergent). Prior to particle size analysis by laser diffraction, various combinations of these methods are used for the preparation of soil samples. Eshel et al. (2004) dispersed overnight 0.1 to 0.5 g of soil in 10 ml of a 50 $g.l^{-1}$ hexametaphosphate solution. Before the measurements, the samples were subjected to a 1-min ultrasonification. Di Stefano, Ferro & Mirabile (2010) treated 10 g of soil sample with 30% hydrogen peroxide to assure complete organic matter removal. The samples were further dispersed in 100 ml of a sodium hexametaphosphate solution (having concentration equal to 50 $g.l^{-1}$) to remove aggregates. The treated samples were mixed overnight using an end – over – end shaking and subjected to ultrasonic treatment for less than 3-min duration. Vdović, Obhođaš & Pikelj (2010) dried the soil samples at 105 ° C, dispersed the samples in deionized water and treated with ultrasound for 5 minutes. Prior to analysis, they used various methods of disaggregation and sample dispersion: i) the original sample was dispersed in deionized water (10 $g.dm^{-3}$), ii) the sample was dispersed in a 0.1% solution of a dispersant (sodium hexametaphosphate), iii) the sample was treated with 15% solution of H_2O_2 and iv) sample was treated with H_2O_2 and then suspended in 0.1% dispersant solution. A method of soil sample preparation prior to analysis, the choice of method to determine the particle size distribution, as well as setting of measuring device during the analysis has a significant influence on the measurement results and thus the comparability of the results obtained by various methods of particle size distribution determination.

This study presents the basic principles of laser diffraction, particle size analysis theories and design of a laser particle sizer ANALYSETTE 22 MicroTec plus (FRITSCH GmbH, Idar-Oberstein). Further, the advantages and disadvantages of this method are discussed.

2 MATERIAL AND METHODS

2.1 *Design of laser particle sizer*

Laser particle sizer ANALYSETTE 22 MicroTec plus (FRITSCH GmbH, Idar-Oberstein) consists of dry dispersion unit, measuring unit and wet dispersion unit (Fig. 1). The selected technical parameters of measuring and dispersion units are shown in Table 1. Dry dispersion is especially suited for not too fine, free-flowing materials, which react in water or other liquids. This type of dispersion fits the best for the measurement of agglomerates of dry powder or for determining particle size distributions of free-flowing, coarse-grained materials. For soil samples, the wet dispersion is the ideal method for perfect dispersion. Therefore the samples are feed into a closed liquid circulation system. An integrated and freely programmable ultrasonic emitter ensures fast and extremely efficient degradation of the agglomerates – precisely adapted to each sample. Due to the integrated water connection, the unit can be automatically cleaned and refilled with new, clean liquid after each measurement.



Fig. 1 ANALYSETTE 22 MicroTec plus: dry dispersion unit, measuring unit and wet dispersion unit (left to right) (photo: Kondrlová, 2013)

Unit	Technical parameters
Measuring unit	Measuring range: 0.08 – 2000 µm (wet dispersion); 0.1 – 2000 µm (dry dispersion) Two semiconductor lasers: green (λ = 532 nm, 7 mW); IR (λ = 940 nm, 9 mW) Number of particle size classes: max. 108 Optical arrangement: Inverse Fourier design; movable measuring cell (FRITSCH patent) Fourier lenses: 260 mm and 560 mm focal length (green or infrared); 10 mm diameter of the laser beam in the Fourier lens Automatic laser beam alignment Sensor: 2 segments, 1x for vertical and 1x for horizontal direction of the laser light polarisation; 57 elements Typical measuring time: 5 – 10 s (measurement value recording of a single measurement); 2 min (entire measuring cycle)
Wet dispersion unit	Suspension volume 300 – 500 cm^3 Radial pump with adjustable speed Ultrasonic with adjustable output (max. 60 Watt)
Dry	Sample volume <1 – 100 cm^2

dispersion unit	High frequency feeder Annular gap Venturi nozzle Required compressed air supply: min. 5 bar, 125 l/min; oil-, water-, particle – free External exhaust system required

Table 1 ANALYSETTE 22 MicroTec plus – technical parameters (FRITSCH-b n.d.)

The measuring cell contains two sources of semiconductor lasers, inverse Fourier optical system, detector, amplifier, evaluation and signal control unit. Depending on the type of dispersion, an optically transparent measuring cell is inserted into upper part of measuring unit (Fig. 2). Inverse Fourier design is an alternative to the conventional design in the form of a convergent laser beam: by positioning the Fourier lens in front of the measuring cell, a convergent laser beam passes through the measuring cell.



Fig. 2 ANALYSETTE 22 MicroTec plus: measuring cell from wet dispersion unit with a flow cuvette (photo: Kondrlová, 2013)

The scattered light is focused directly onto the detector without additional optical elements (Fig. 3). A semi-conductor laser with green light carries out the measurement of small particles, while an infrared-semiconductor laser handles large particle size ranges. Both lasers can be optimally aligned extremely quickly, automatically and independently of each other through lateral motion. Your advantage: the ideal wavelength for every particle size and an ideal combination of large measuring range, outstanding resolution and small footprint. With redirection of the long wavelength red laser beam, the ANALYSETTE 22 MicroTec plus allows for maximum measurement precision even for large particles within a compact unit. To switch to the measurement of small particles in the short wavelength green laser, the detector and the laser source are simply moved as a unit – the measuring cell remains fixed in place (Fig. 4).

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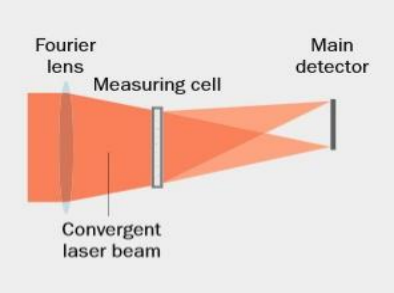


Fig. 3 Scheme of inverse Fourier optics design (FRITSCH-b n.d.)



Fig. 4 Scheme of measurement design according to the active laser (FRITSCH-a n.d.; modified)

2.2 *Control software*

The device is controlled by instructions from the MaS control software that is based on a relational database in which all user entries, parameters and results are securely stored and safe from manipulation. The software contains completely Predefined Standard Operating Procedures (SOPs) that regulate many typical measurement processes. Own SOPs can be completely freely defined, or the existing can be modified to perfectly suit measurement requirements: the dispersion process and duration, measuring frequency, time intervals and many other parameters can be easily selected and saved as separate SOPs. The report generator allows measurement reports to be organised exactly according to user needs. The user – friendly software interface enables clear organisation of the measuring data; fast comparison of different measurements; freely selectable user values issued in a table format; data export to Excel, PDF, etc., storage in SQL database (FRITSCH-b n.d.).

2.3 *Theories for analysis*

The actual result of a particle size measurement is only created through analysis with the supplied software. Depending on the particle properties and requirements, two common analysis theories are used for this: Fraunhofer theory for larger particles when their exact optical parameters are unknown and Mie theory for the smallest particles with known optical parameters. It is very easy to select both theories in the FRITSCH MaS control software. The Fraunhofer theory describes the portion of light deflection that occurs exclusively as a result of diffraction. If light encounters an obstacle or an opening, this results in diffraction and interference effects. If the incoming light is parallel (even wave fronts), this is referred to as Fraunhofer diffraction. Since for sufficiently large particles the light deflection is dominated by diffraction, Fraunhofer theory can be used for particle size distribution down to the lower micrometre range. One major advantage of Fraunhofer theory lies in the fact that no

knowledge of the optical properties of the examined material is required. For particles with diameters that are not significantly larger than the wavelength of the light used, the Mie theory is applied for the analysis of the measurements. This theory is the complete solution of the Maxwell equations for the scattering of electromagnetic waves by spherical particles. It can be used to analyse the characteristic intensity distributions for even very small particles, which, in contrast to Fraunhofer theory, are not restricted to scattering angles of less than 90° (forward scattering). In fact, scattering angles of greater than 90° also occur (backward scattering). In order to be able to use the intensity distribution for the calculation of the particle size, determined in this manner, the refraction index and absorption index of the sample must be known with the Mie theory in contrast to the Fraunhofer theory. The FRITSCH software includes a comprehensive database containing the refraction indexes of numerous different materials (FRITSCH-b n.d.). On the other hand, it is very difficult to estimate these values for heterogenic mixtures, e.g. soil. Since most of the current laser particle sizers works with a laser with a wavelength of 500 up to 700 microns, thus the use limit of methods ranges from 0.5 up to 0.7 microns (1 micron respectively) (Crolly n.d.). However, the decision of which method is used depends not only on particle size but also on the heterogeneity of the sample material and the particular application.

2.4 *Sample preparation prior to analysis*

To date, the standard method of soil sample preparation for laser diffraction has not yet been specified. Sample preparation methods that were used for our analysis were derived from the established practices used in standard sedimentation methods. In order to derive a rapid and simple method of soil sample preparation prior analysis, some steps (e.g. removal of carbonates and organic matter) were omitted or simplified. As an example, randomly selected soil sample (59/15) collected in the Nitra River catchment, West Slovakia was used for analysis. Prior to laser analysis sample was sieved and fraction < 2 mm was further divided into individual samples, that were treated according to proposed methods of soil sample preparation. The procedures included weighting, mixing, covering for 4 resp. 24 hours prior to analysis, changing the device settings – ultrasonic at energy level No. 1, 5 and 10 respectively.

List of methods used for soil sample preparation:

1. method – susp_24U10: 5g of fine soil mixed in 100 ml beaker with 45 ml of tap water and 5 ml of Graham's salt, dispersion for 24 hours, ultrasonic – energy level No. 10.

2. method – dest_24U10: 5g of fine soil was mixed with 5 ml of distilled water, dispersion for 24 hours, ultrasonic – energy level No. 10.

3. method – r_5g_24U10: 5 g of fine soil mixed with 3 ml of Graham's salt, dispersion for 24 hours, ultrasonic – energy level No. 10.

4. method – r_3g_24U10: 3 g of fine soil was mixed with 3 ml of Graham's salt, dispersion for 24 hours, ultrasonic – energy level No. 10.

5. method – r_3g_24U5: 3 g of fine soil was mixed with 3 ml of Graham's salt, dispersion for 24 hours, ultrasonic – energy level No. 5.

6. method – r_3g_24U1: 3 g of fine soil was mixed with 3 ml of Graham's salt, dispersion for 24 hours, ultrasonic – energy level No. 1.

7. method – r_3g_4U5: 3 g of fine soil was mixed with 3 ml of Graham's salt, dispersion for 4 hours, ultrasonic – energy level No. 5.

8. method – r_3g_4U1: 3 g of fine soil was mixed with 3 ml of Graham's salt, dispersion for 4 hours, ultrasonic – energy level No. 1.

3 RESULTS AND DISCUSSION

3.1 Particle size analysis by laser diffraction

Prior to the measurement, each sample was mixed well. Sample in the form of suspension was poured into the graduated cylinder and shaken several times. Appropriate SOP for wet dispersion was selected in the MaS control software. At first, the intensity of scattered light in dispersion liquid (tap water) without present sample particles was measured to obtain the background measurement. This measurement enables to assess any possible pollution of the measuring cell, which is then subtracted from the following measurement of the sample dispersed in the liquid. The required small quantity of sample has been dosed in an ultrasonic bath (volume set to 400 ml). The measurements were carried out over the whole measurement range of the instrument (0.08 – 2000 µm) using the Fraunhofer theory and automatic calculation model. In case of overloading the optimal dose (10 - 15% of beam obstruction), the sample was diluted to the optimal concentration using software tool. Pump unit (set to executive level 6) supplied smooth flow of dispersion liquid and dispersed sample in an ultrasonic bath as well as in the measurement cell. Each sample was dosed 3-times and each dose was analysed 3-times. After each sampling, the suspension was discharged from the dispersion unit into drain and the unit for wet dispersion was cleaned automatically to get ready for the next measurement.

3.2 Interpretation of the results in the MaS control software

After the measurement is done, the resulting values can be seen right on the screen in various forms. Graph window on the left side (default setting) presents cumulative distribution curve and fractional histogram for selected measurement (Fig. 5). Since there is possibility of multiplied selection, various number of measurement results can be graphically compared what can be very useful for repeated analyses. Result table on the right side (default setting) presents the cumulative percentage distribution of the particle size fractions (in µm) specified prior to analysis (Fig. 5). Results from selected measurements can be printed in report form and saved under various file types. To do more detailed analyses, it is possible to export the results of individual measurement in the Excel file (Fig. 6). All the measurements are stored in the database. One of the advantages of this database is the possibility to recalculate the analysis results under specific circumstances: changing calculation model, analysis theory (Fraunhofer to Mie and vice versa) or the particle size classification defined by different size fractions. In all these cases, repeating of the measurement is not needed.

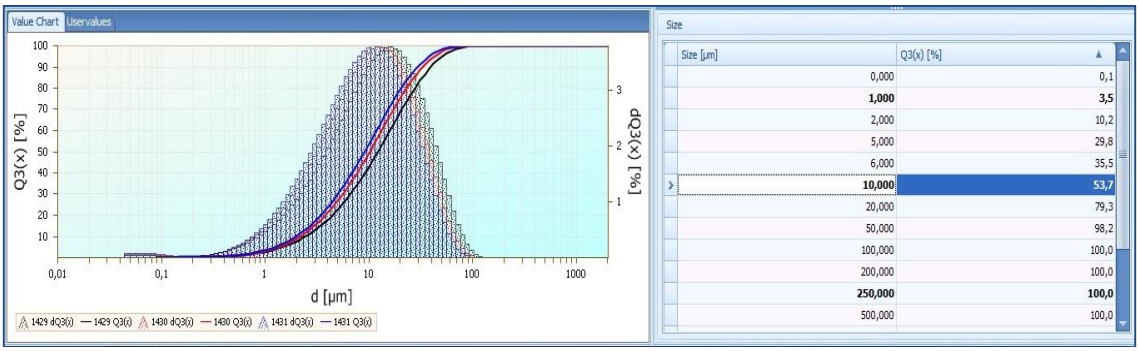


Fig. 5 Interpretation of the measured values in the MaS control software

Measurement results were averaged for each soil sample preparation method. The course of the distribution of the various particle size fractions is represented by the cumulative particle size curve (Fig. 6). Comparison of the results shows a significant effect of sample dispersion duration,

concentration of dispersion mixture as well as the ultrasonic intensity on the fragmentation of soil aggregates (Fig. 6). With increasing ultrasonic intensity, the distribution of finer particles was also arising and thus the highest proportion of fine particles was observed for methods 3 and 4 (sample in the form of watery soil paste, ultrasonic level No. 10). Methods 3 and 4 differed from each other only by the quantity of material used to prepare the samples. The course of their cumulative particle size curves is similar, so it can be concluded that for particle size analysis a very small amount of sample is needed (3 g). The dispersion duration did not influence the results at low ultrasonic intensity (method 6 vs. 8), but for higher ultrasonic intensities it should be considered (method 5 and 7). The comparison of the results shows that the ultrasonic intensity has got greater impact on the breaking of soil aggregates than the dispersant application at the indicated concentration. Chappell (1998) also considered the possibility that the exposure of the sample to too long ultrasonic treatment may also lead to breaking of quartz grains. His assumption was verified by conducted laboratory microscopic observations of sharp-edged grain fragments. In the case of method 2, where no dispersant solution was used, lower percentage distribution of fine particles was measured than at other methods with the same level of ultrasonic treatment (method 3 and 4), resp. the medium level (method 5 and 7). In case of method 1 – sample suspension, the effect of dispersant was lower, due to its lower concentration. At the same time, this method achieved the highest variation coefficients from 4 up to 8%, what means that this method of sample preparation was not as repeatable as the other methods. Despite the continuous stirring, to prevent particle deposition and representative sampling was very difficult. Due to sedimentation of particles in suspension, the results of analysed samples prepared by method 1 may be burdened with measurement errors. The same problem was described also by Arriaga, Lowery & Dewayne Mays (2006).

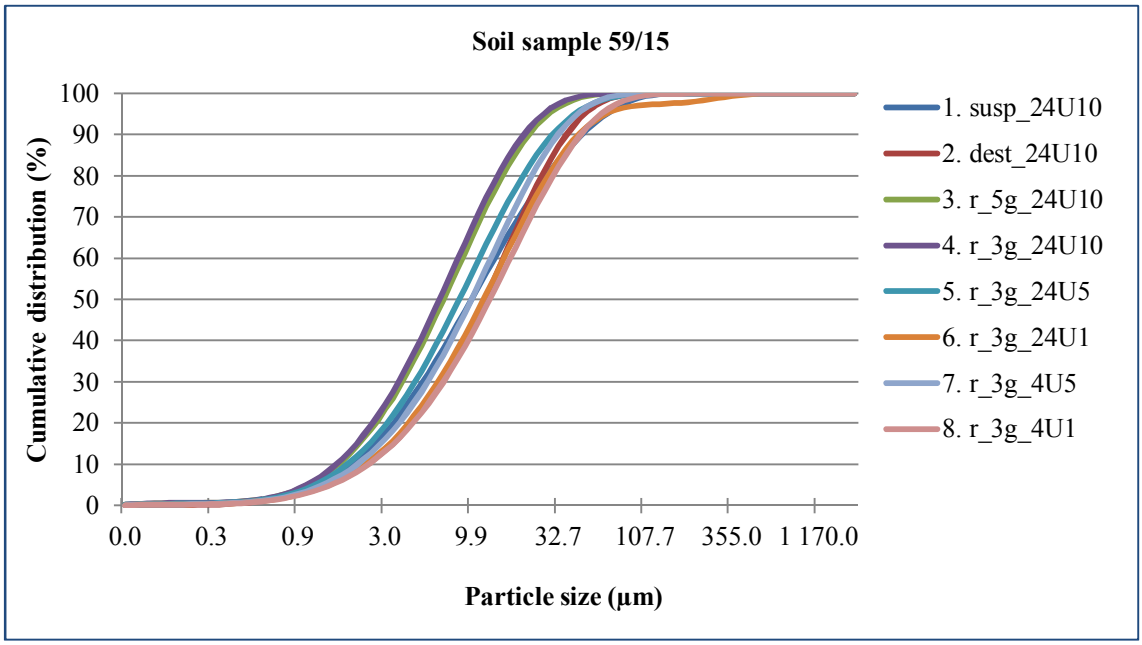


Fig. 6 Cumulative particle size curves for individual soil sample preparation methods

Several authors dealt with comparison of pipette method and laser diffraction (e.g. Eshel et al. 2004; Beusenlick et al. 1998; Vandecasteele & De Vos 2001; Arriaga, Lowery & Dewayne Mays 2006; Vdović, Obhođaš & Pikelj 2010). Since these methods are based on different physical principles, the

distribution of grain size fractions determined by the laser diffraction is not comparable with the sedimentation methods in the ratio 1:1 (Vandecasteele & De Vos 2001). While at pipette method the values on mass percentage are obtained, the latter method gives the results in volume percentages. This fact decreases trustworthiness of laser diffraction as a method of determining soil texture, despite of its many benefits. Beusenlick et al. (1998) quoted following advantages of this method: it is rapid and has good reproducibility, only single analysis is needed to obtain a wide range of particle sizes, small amount of sample material is needed, detailed information on the grain size distribution is obtained and results are given directly in a digital form (MS Excel spreadsheet) and can be easily manipulated.

4 CONCLUSIONS

Because of its advantages, laser diffraction is recommended method of soil texture determination. It allows more detailed analysis of the particle size distribution and simultaneous use of the results for the purpose of different national soil classification systems without necessity to repeat the measurements. Comparison of the results confirms the importance of sample preparation method and its effect on particle size analysis. It is also recommended to test the accuracy of the proposed methods on a larger set of samples in various settings and parameters of the device (ultrasonification level, measuring range).

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