

**Determination of sedimentation speed of soil micro-particles  
from laser diffraction measurements**

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In hydropedological research and in various scientific experiments, the determination of the settling rate of soil microparticles is a frequent task. Many laboratory procedures for measuring sedimentation rate are based on the Stokes equation. In recent years, methods based on the principle of laser diffraction can be used to measure grain-size distribution and deposition rate of microparticles. The output of the measurements by a laser diffraction method is statistical distribution of soil texture in the measured sample by particle size expressed in % of volume. Measurements were performed in a wet way by MALVERN Instruments device called Mastersizer 2000. The proposed method is based on measuring the time required for soil particles of certain diameter to pass certain distance. The size of soil microparticles present in space and time is defined by probability. Probability is defined in the form of a grain size distribution function. The advantage of the proposed method is its robustness and elimination of human factor errors. This paper presents the results of theoretical approaches and experimental measurements of the settling rate of soil microparticles. Soil samples were taken in the East Slovakian Lowland. Measurements are performed for the selected sizes of soil microparticles for a probability of occurrence of 90%, i.e. for  $d(90)$ . The results are compared with the results calculated by the Stokes equation.

KEY WORDS: sedimentation rate, soil microparticles, dispersion system

**Introduction**

Soil microparticles are of particular interest in hydropedological research. They are usually composed of clay particles with a high proportion of clay minerals. Clay particles are defined as particles  $\leq 2 \mu\text{m}$ . Soils with a high proportion of clay particles ( $> 45\%$ ) are called clay soils. Clay minerals can bind water in their structure. When they are saturated with water, their volume increases, and when they are dried, they shrink. During these processes, the retention properties and hydrodynamics of the unsaturated zone of the soil profile change significantly. Knowledge of these properties is essential for the investigation and numerical simulation of the water regime of heavy soils (Gomboš, 2012; Hašková, 2007; Igaz et al., 2011; Skalová et al., 2015; Šoltész et al., 2021; Velísková, 2010; Gomboš et al., 2019; Številová et al., 2017; Gomboš et al., 2018a; Šoltész et al., 2019; Tall et al., 2019; Dulovičová et al., 2021; Zvala et al., 2021; Taubner et al., 2009). So far, many laboratory procedures for measuring settling rate have been based on sedimentation methods, the theoretical basis of which is the Stokes equation. This is valid for the laminar flow area, for the spherical shape of the particles. The disadvantage of these methods is the fact that as the sedimenting particles are reduced,

the dispersion system changes to a colloidal state. The results are influenced by diffusion and thus the validity of Stokes' law is limited to soil microparticle sizes  $< 2 \mu\text{m}$ . The settling process in the dispersion system continues until the dynamic sedimentation equilibrium of the system occurs. At sedimentation equilibrium, the sedimentation rate of the dispersed particles equals the rate of their diffusion in the opposite direction. This is especially the case for colloidal dispersions. Diffusion is not measurable in coarse dispersions. Conversely, no measurable sedimentation occurs in analytical dispersions. In recent years, a method based on the principle of laser diffraction has been used for sedimentation analysis (Aydin et al., 2012; Gomboš et al., 2018b; Igaz et al., 2020; Mihalache et al., 2010; Šinkovičová et al., 2017; Yang et al., 2015; Zhu et al., 2016).

This contribution summarizes the basic theoretical background applied to address this issue. The aim of the contribution is to present the methodological procedure and results of experimental measurements of the settling rate of soil microparticles. The velocity calculation is based on the use of sedimentation analysis obtained by the laser diffraction method. Soil samples were taken in the East Slovakian Lowlands. Velocity measurements by the proposed method are performed for

soil microparticle sizes with a probability of occurrence of 90%, i. for d(90), d(50), d(10). The results are compared with the results calculated using the Stokes equation.

## Material and methods

### Theoretical basis

Sedimentation rate of particles in a disperse system depends on the forces acting on particles. In gravitational field, a settling particle in fluid is under the influence of gravity  $F_g$  and the opposing forces, buoyancy  $F_b$  and drag force  $F_D$ . At the beginning of sedimentation, settling particles increase their speed. At certain speed, drag force increases to such an extent that the forces come into equilibrium. The sedimentation rate is constant if the forces are balanced:

$$F_D = F_g - F_b \quad (1)$$

The equation (1) can be expressed in the form:

$$f \cdot u = V \cdot \rho \cdot g - V \cdot \rho_0 \cdot g \quad (2)$$

where

$f$  is the friction coefficient, which depends on the shape and size of particles,

$u$  is the sedimentation rate,

$V$  is the volume of a settling particle,

$\rho$  is the volumetric mass density of a settling particle,

$\rho_0$  is the volumetric mass density of a dispersion medium,

$g$  is the gravitational acceleration on the surface.

For spherical particles with the radius  $r$ , dispersed in a dispersion medium with the viscosity  $\eta_0$ , Stokes derived an equation for the friction coefficient  $f$  based on the Navier-Stokes equations:

$$f = 6 \cdot \pi \cdot \eta_0 \cdot r \quad (3)$$

The combination of equations (2) and (3) gives Stokes' equation (4) describing the sedimentation rate of spherical particles:

$$u = \frac{2}{9} \frac{(\rho - \rho_0) \cdot r^2 \cdot g}{\eta_0} \quad (4)$$

The equations are valid for spherical particles which are far bigger than the molecules of the surrounding environment, assuming that their surface is smooth without any electric charge and they move at low velocities within laminar flow at low Reynolds number ( $Re < 0,5$ ). When calculating the sedimentation rate of soil particles, the deviations from the real conditions are caused by the fact that the conditions of spherical particles and the size of particles are not met. If the particles are not spherical, the equation for Stokes' drag must be modified as follows:

$$F_{D_m} = f_e \cdot u = 3 \cdot \pi \cdot \eta_0 \cdot d_e \cdot u \cdot K \quad (5)$$

where

$d_e$  is the diameter of a sphere with the same volume as a non-spherical particle, i.e.

$$d_e = \left( \frac{6}{\pi} \times \text{volume} \right)^{\frac{1}{3}} \quad (6)$$

$K$  is the correction factor which depends on the shape and size of a particle.

In the experiment, one of the oldest methods of particle size analysis was used – sedimentation analysis. Dispersed phase (i.e. soil particles) in the polydisperse system was statistically divided in the groups of particles of similar size called fractions. In the sedimentation analysis, the distribution of particles was expressed by means of frequency and cumulative distribution functions. Frequency distribution functions  $F(r)$  of particle weight express the frequency of the particles of certain size [% of weight] in a dispersed phase:

$$F(r) = \frac{dm_r}{m \cdot dr} \quad (7)$$

where

$m_r$  is the weight of the particles with the size,

$r$ ,  $m$  is the weight of the whole dispersed phase.

Soil dispersed phase is composed of many fractions. Frequency distribution function  $F(r)$  is a continuous distribution curve, assuming that:

$$\int_0^\infty F(r) dr = 1 \quad (8)$$

Cumulative distribution functions  $I(r)$  of particle size express the weight proportion of the fractions containing the particles with the size equal or smaller than the defined value  $r$ :

$$I(r) = \int_0^r F(r) dr \quad (9)$$

In the measurements based on laser diffraction analysis, the frequency of occurrence of particles of certain size in dispersed phase is expressed in % of volume. Numerically, it is the same value as expressed by % of weight.

Outputs from the measurements by laser diffraction method in the form of frequency of occurrence of particles of a certain size allow the calculation of the particle velocity. During the experiment, suspension (colloid) samples for laser diffraction are taken from the dispersion system at two height levels  $H_1$  and  $H_2$  at selected time intervals. A dependency is analysed for each height:

Outputs from measurements by the laser diffraction method in the form of a number of particles of a certain size allow the calculation of the speed of movement of a microparticle of a certain size. In the actual work procedure, suspension (colloid) samples for laser diffraction are taken from the dispersion system from two

height levels  $H_1$  and  $H_2$  (Fig. 1) at selected time intervals. A dependency is sought for each altitude level:

$$d(P)^{H_1} = f(t)^{H_1} \quad (10)$$

$$d(P)^{H_2} = f(t)^{H_2} \quad (11)$$

where

$d(P)^{H_1}$  and  $d(P)^{H_2}$  are functions of time.

These are particle sizes with a diameter  $d$  and a probability of occurrence  $P$  that pass through the plane of  $H_1$  and  $H_2$  levels at different times  $t$ . From equations (10) and (11) it is clear that in order to calculate velocity, it is necessary to find such times  $t^{H_1}, t^{H_2}$  at which the left sides of said equations are the same, i. the investigated particle has the same size and probability of occurrence at different heights  $H_1$  and  $H_2$  spaced  $\Delta H$  apart. It is logical that such a situation occurs at different times from the beginning of sedimentation. Then if

$$d(P)^{H_1} = d(P)^{H_2} \rightarrow \Delta t = f(t)^{H_2} - f(t)^{H_1} \rightarrow \text{speed } v = \frac{H_1 - H_2}{\Delta t} = \frac{\Delta H}{\Delta t} \quad (12)$$

Some form of analytical expression is preferred for expressing the right-hand sides of equations (10) and (11). In the case of measurements, it is possible to use some interpolation method (linear, cubic), resp. extrapolation. Sometimes it is advantageous to divide the dependences (10) and (11) into intersection intervals and then express each dependence in the form of several equations. Measured speed with speed calculated according to Stokes equation (4).

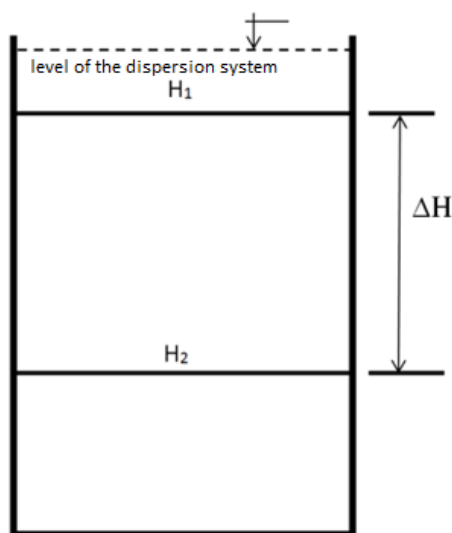


Fig. 1. Experiment scheme.

### Description of the experiment

The investigated sites where soil was collected for the sedimentation experiment are located in three localities on the East Slovakian Lowlands (N 48° 39' 53.41", E 22° 02' 53.22"; N 48° 28' 06.41", E 21° 58'

53.32"; N 48° 39' 53.41", E 22° 02' 53.22"). It is an area of tectonic character with a complex structure of decline faults. It consists of Quaternary and especially powerful Neogene sediments. The area is characterized by wetlands, which conditioned the genesis of the local heavy soils. Sampling in the mentioned localities was realized in two soil layers. The basic characteristics of the sampling points are given in Table 1. The particle sizes in  $\mu\text{m}$  are given for the individual layers at the beginning of the measurements and at the end of the measurements at the upper sampling level ( $H_1$ ) above the bottom of the settling vessel.

Soil samples were used to form a dispersed fraction. Subsequently, a dispersion system was created, in which the settling of soil microparticles was investigated. In the experiment, solid dispersed fraction in liquid (distilled water) dispersion system formed a heterogeneous dispersion phase. At the beginning of settling, the dispersed phase formed a coarse suspension  $d > 10^{-6}$  m. During settling, a colloidal lyosolic dispersion was formed where the dimensions of particles in disperse phase are defined as  $10^{-9} \text{ m} < d < 10^{-6} \text{ m}$ . Depending on the particle shape, a laminar dispersion system with platelet-shaped clay anisometric particles is envisaged. In terms of particle size distribution, it is a polydisperse system consisting of particles of different sizes.

The procedure for the preparation of the dispersion system was as follows: The soil sample was air dried in the laboratory and mechanically crushed. Subsequently, the sample was sieved through a  $\varnothing 2 \text{ mm}$  sieve to discard any skeleton. An amount of 0.4 kg was weighed from the sieved soil, and this was mixed with distilled water and a dispersant – a solution of sodium hexameta-phosphate and sodium carbonate in a ratio of 35.7 : 7.9 g per 1 l of distilled water. The resulting suspension was mixed thoroughly and allowed to stand for 24 h. After that, the suspension was boiled for 1 hour with occasional stirring. After cooling, the sample was poured into a sedimentation cylinder and filled with distilled water to complete 5 l. The suspension was then prepared for the experiment.

Prior to the start of the experiment, the suspension in the settling cylinder was thoroughly mixed to form a homogeneous state. From this point on, the sedimentation time was counted. At specified times, a pair of suspension samples at constant heights  $H_1$  and  $H_2$  were taken from the sedimentation cylinder using pipettes. The scheme of the experiment is shown in Fig. 1 where  $H_2$  is 5 cm and  $H_1$  is 15 cm above the bottom of the settling vessel. The investigated path of the soil microparticle  $\Delta H$  is 10 cm. At the same time, the temperature of the suspension was checked. Sampling time intervals were chosen in a way that their increase in the semi-logarithmic representation was linear. The velocities were calculated based on the particle motion for  $d$  (90). A total of 204 samples were analyzed.

The texture of the samples was determined by laser diffraction on a Mastersizer 2000 by MALVERN Instruments. Laser diffraction measures particle size distributions by measuring the angular variation in intensity of light scattered as a laser beam passes through

a dispersed particulate sample. Large particles scatter light at small angles relative to the laser beam and small particles scatter light at large angles. The angular scattering intensity data is then analyzed to calculate the size of the particles responsible for creating the scattering pattern, using the Mie theory of light scattering. The particle size is reported as a volume equivalent sphere diameter. The device is able to work both dry and wet way. The grain size analyses in this work were performed exclusively by the wet method using the Hydro 2000MU dispersion unit. The dispersant was distilled water. Prior to each analysis, the suspension samples exposed to ultrasound for 5 minutes in order to enhance dispersion. The measurement range indicated by the manufacturer is between 0.02 and 2000  $\mu\text{m}$ .

## Results and discussion

On the left sides of Figures 2 to 7, the grain distributions in the examined soil layers at the lower  $H_2$  level at the beginning of the experiment and at the end of the experiment are shown. The grain size distributions for the upper sampling levels  $H_1$  are shown on the right. When comparing the grain distribution at the upper and lower sampling point at the start of the experiment (red lines), it is clear that the distributions are the same. The suspension was homogeneous at the start of the experiment. There were no inhomogeneities that

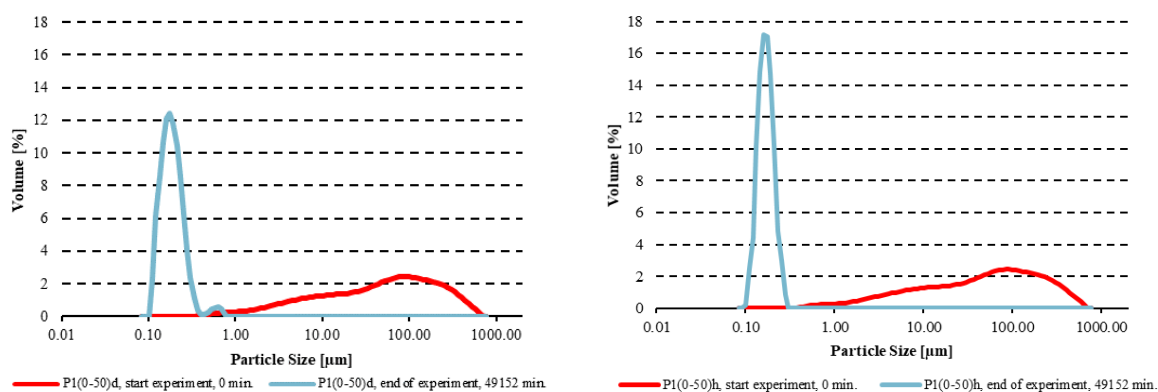
could cause errors in measuring the settling rate of soil microparticles. From the comparison of the grain size distribution at the upper and lower sampling point at the end of the experiment (blue lines), some courses are identical (Fig. 4 and Fig. 6). Identical courses indicate that the soil suspension was homogeneous at the end of the experiment. This means that a dynamic sedimentation equilibrium was reached. Small differences in the distribution curves indicate that sedimentation of the smallest particles was still taking place at the end of the experiment. Clearly different course in the left side of Figure 7, i.e. Senné locality, layer 200 to 250 cm under the ground, was probably caused by the agitation of the suspension during sampling at the lower  $H_2$  level.

Table 2 shows the sedimentation rates of soil microparticles from the investigated localities. For comparison, there are also sedimentation rates calculated by the Stokes equation. The sedimentation rates determined by laser diffraction measurements for  $d(90)$  and particle sizes  $> 2 \mu\text{m}$  are smaller than the rates calculated by the Stokes equation. The match is higher after introducing an empirical correction factor of 1/5 compared to the original value of 2/9 for the spherical shape of microparticles.

Figure 8 shows a graphical comparison of the soil microparticles settling velocities measured by laser diffraction and Stokes velocities. The graphical comparison shows a good coincidence of the Stokes

**Table 1.** Basic characteristics of sampling points

Locality	Coordinates	Thickness of the subsoil layer [cm]	Site designation	Soil type	d(10)	d(50)	d(90)	d(10)	d(50)	d(90)
					start of measurements			end of m., upper level		
Poľany 1	N 48°39'53.41" E 22°02'53.22"	0–50	P1(0–50)	clay - loam	3.886	53.968	272.059	0.131	0.168	0.217
		50–100	P1(50–100)	clay	4.389	57.31	218.781	0.133	0.174	0.228
Poľany 2	N 48°28'06.41 E 21°58'3.32"	0–50	P2(0–50)	silt-loam	2.258	16.935	84.432	0.13	0.178	0.249
		100–180	P2(100–180)	sandy-loam	2.062	12.136	41.793	0.131	0.173	0.226
Senné	N 48°39'3.41" E 22°02'53.22"	100–150	S(100–150)	clay	1.417	5.057	25.456	0.131	0.179	0.250
		200–250	S(200–250)	clay	1.821	10.371	118.514	0.14	0.197	0.311



**Fig. 2.** Grainsize distribution at the lower sampling level  $H_2$  (left) and at the upper sampling level  $H_1$  (right) at the start and at the end of the experiment in the locality Poľany 1, layer 0–50 cm under the surface.

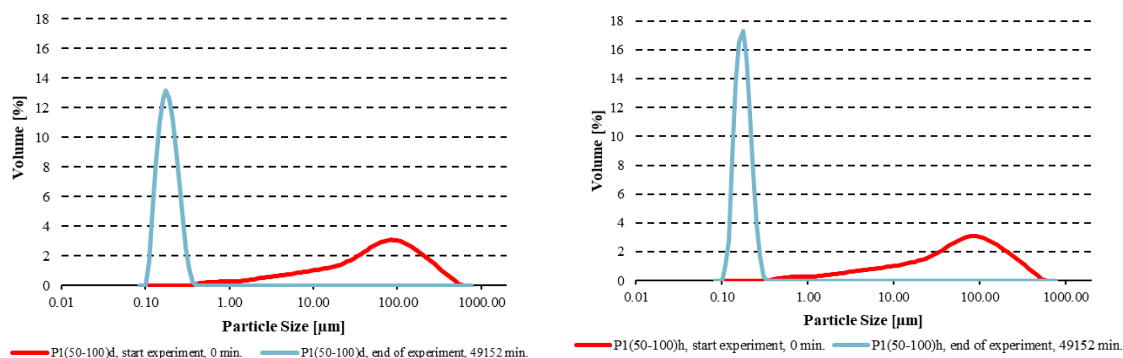


Fig. 3. Grain-size distribution at the lower sampling level  $H_2$  (left) and at the upper sampling level  $H_1$  (right) at the start and at the end of the experiment in the locality Polany 1, layer 50–100 cm under the surface.

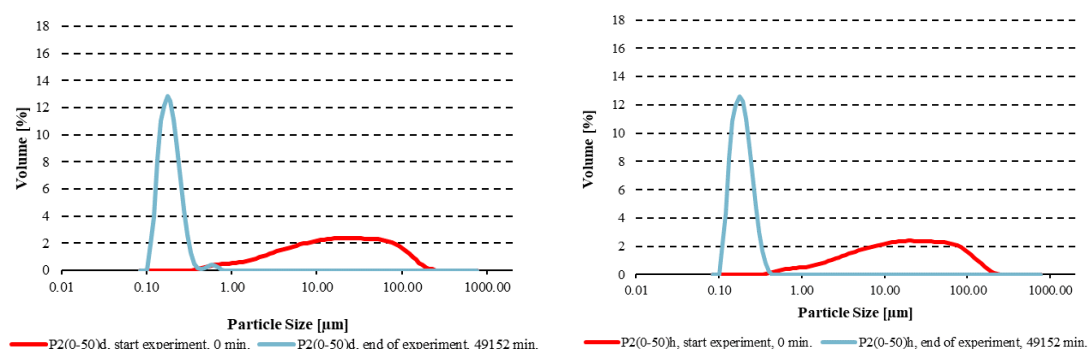


Fig. 4. Grain-size distribution at the lower sampling level  $H_2$  (left) and at the upper sampling level  $H_1$  (right) at the start and at the end of the experiment in the locality Polany 2, layer 0–50 cm under the surface.

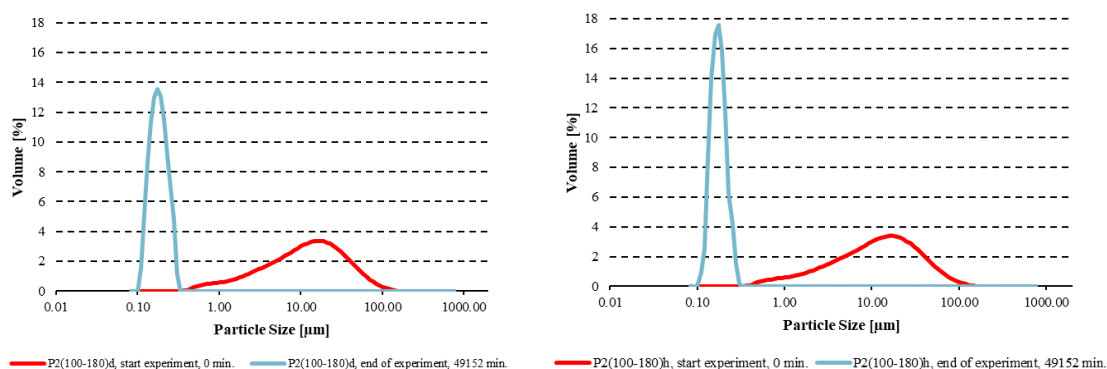


Fig. 5. Grain-size distribution at the lower sampling level  $H_2$  (left) and at the upper sampling level  $H_1$  (right) at the start and at the end of the experiment in the locality Polany 2, layer 100–180 cm under the surface.

calculations using the empirical correction factor 1/5 with the measured velocities using laser diffraction. High coincidence is only in the interval 2–10  $\mu\text{m}$ . As soil microparticles shrink below 2  $\mu\text{m}$ , the difference between rates calculated by the Stokes equations and laser diffraction analysis increases. From the above, it is assumed that laser diffraction is more accurate for determining settling rates for particles < 2  $\mu\text{m}$ . This is

also due to the fact that the diffraction angles decrease as the particle sizes increase. This makes small differences more difficult to detect and the resolution of laser diffraction decreases. Particle shrinkage causes greater intensity in light scattering. This expands the measurement range of laser diffraction and makes it more precise for smaller particle size. The use of traditional methods of grain-size analysis and sedimentation rate

measurement is limited at very small grain sizes. Dispersion system gradually becomes a colloid in which the diffusion effect increases. Under certain conditions, sedimentation equilibrium occurs. Measurements to date

have shown that Stokes' law can be applied for soil colloids with particles sized up to 2  $\mu\text{m}$ . For investigating the movement of smaller particles laser diffraction-based techniques are more convenient to be used.

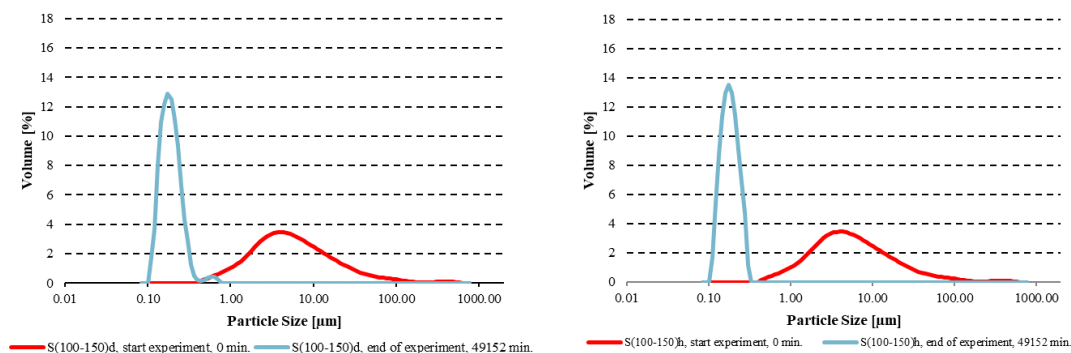


Fig. 6. Grain-size distribution at the lower sampling level  $H_2$  (left) and at the upper sampling level  $H_1$  (right) at the start and at the end of the experiment in the locality Senné, layer 100–150 cm under the surface.

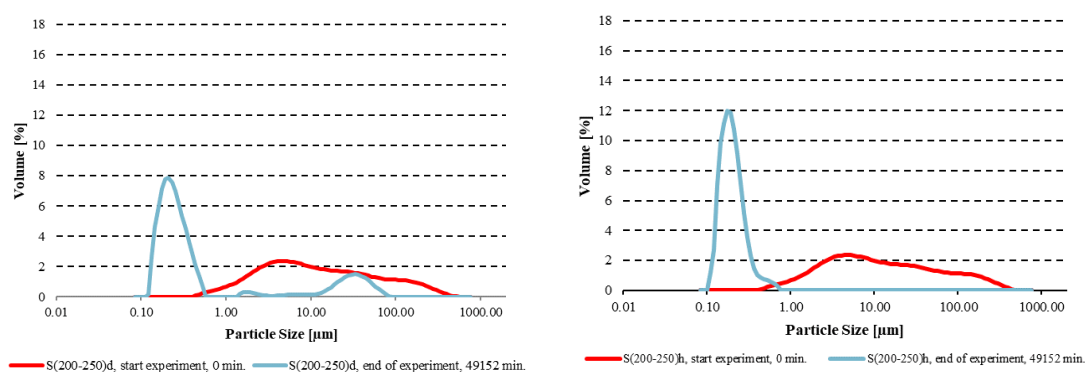


Fig. 7. Grain-size distribution at the lower sampling level  $H_2$  (left) and at the upper sampling level  $H_1$  (right) at the start and at the end of the experiment in the locality Senné, layer 200–250 cm under the surface.

**Table 2.** Measured settling rates of soil microparticles of different sizes Comparison of sedimentation speed of soil microparticles measured by laser diffraction with speed calculated according to Stokes

soil microparticle size [ $\mu\text{m}$ ]	10.0	7.0	5.0	3.0	2.0	1.0	0.9	0.8	0.7	0.6	0.5	0.4	0.3
Localities	Settling rate of soil microparticles [ $\mu\text{m min}^{-1}$ ]												
Poľany P1(0–50)	511.8	304.5	159.3	51.0	30.9	18.3	17.8	17.4	16.9	16.8	12.7	10.2	8.5
Poľany P1(50–100)	1146.1	605.4	279.3	101.8	70.0	37.8	34.2	27.6	21.9	19.0	16.8	15.0	6.3
Poľany P2(0–50)	1030.2	442.3	243.3	75.9	38.1	17.0	16.2	15.5	14.9	11.0	9.3	8.7	8.2
Poľany P2(100–180)	1152.6	605.5	312.4	74.0	51.3	24.6	20.7	17.8	15.7	11.0	9.8	8.9	8.1
Senné S(100–150)	2067.2	1092.2	388.5	95.1	55.0	16.3	14.3	11.4	10.3	9.4	8.7	8.0	4.6
Senné S(200–250)	1918.9	502.3	266.1	80.5	37.8	9.8	8.2	7.5	6.9	5.6	4.2	3.8	---
Stokes $K=2/9$	5403.4	2647.7	1350.9	486.3	216.1	54.0	43.7	34.6	26.5	19.5	13.5	8.6	4.9
Stokes delta $K=1/5$	1185.0	580.7	296.3	106.7	47.4	11.9	9.6	7.6	5.8	4.3	3.0	1.9	1.1



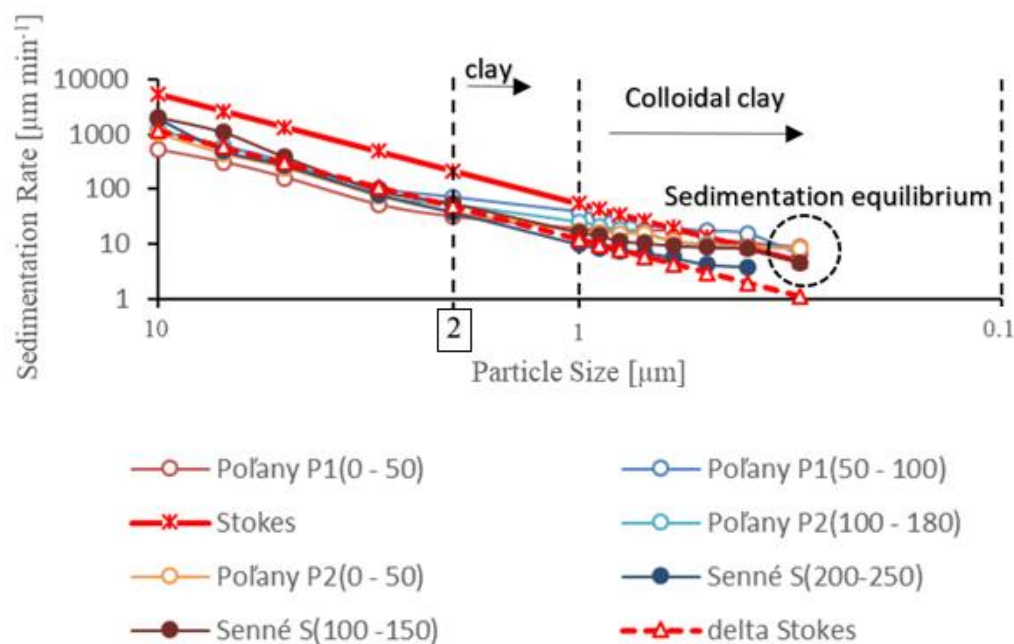


Fig. 8. Graphical comparison of sedimentation rates of soil microparticles measured by laser diffraction with the rates calculated by the Stokes equation.

## Conclusion

The availability of modern instruments for research practice opens up new possibilities for research. The contribution summarizes the traditional theoretical background and shows new possibilities for investigating the speed of soil microparticles in water. Previous analysis has been gained on the MALVERN Instruments Mastersizer 2000. This device allows measurements for particle size lower limit of 0.02  $\mu\text{m}$ . An experimental method is proposed in this contribution for the use of this device for measuring the sedimentation rates of soil microparticles. It is based on measuring the time during which soil microparticles of a defined diameter pass certain distance. The size of soil microparticles in space and time is defined by probability. Probability is defined in the form of grain-size distribution functions. The proposed method was investigated in an experiment. The settling rates of soil microparticles sampled at three localities in the East Slovakian Lowland were monitored. The results proved the applicability of the presented method for the analysis of soil microparticles (especially clay microparticles) sedimentation. The advantage of the proposed method is its robustness, elimination of human factor errors and measurement speed.

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