sciendo
DOI: 10.2478/ssa-2018-0003

SOIL SCIENCE ANNUAL
Vol. 69 No. 1/2018: 17–27

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Soil grain size analysis by the dynamometer method – a comparison to the pipette and hydrometer method

Abstract: The aim of the presented work was to compare the results of grain size distribution measurement by an innovative dynamometer method, developed by the authors, with results obtained by the pipette and hydrometer methods. Repeatability of results obtained in the dynamometer method was also determined. The content of three fractions with equivalent diameters <0.002 mm, 0.002–0.063 mm and 0.063–2.0 mm was measured. The results were compared using ordinary linear regression and additionally in the repeatability analysis by RMA (reduced major axis regression). It was found that the proposed dynamometer method is characterized by good result repeatability with no systematic errors when compared with the pipette method. The RMSE (root mean square error) value when referring to the pipette method calculated for the three fractions considered in total was 4.9096 and was lower than the analogous for the hydrometer method, which amounted to 5.4577. Values of determination coefficients in the comparison of dynamometer method and pipette method are within the range of 0.9681–0.9951 for the different fractions. It was found that slightly larger differences in relation to the pipette method occurred for the fractions <0.002 mm and 0.002–0.063 mm, and smaller for the fraction 0.063–2.0 mm. Similarly, greater differences between repetitions in the dynamometer method were observed for the fraction <0.002 mm, and smaller for the 0.063–2.0 mm fraction. Possible sources of errors in the dynamometer method were discussed, as were proposals for their reduction.

Keywords: grain size composition, dynamometer method, pipette method, settling velocity

INTRODUCTION

Soil grain size composition is the basic property used to predict other physical features (Trzecki 1974, 1976; Gimenez et al. 2001, Walczak et al. 2006, Lamorski et al. 2014, Brogowski and Kwasowski 2015). It is most often determined by sieve-sedimentation methods. These methods have been developed for several dozen years (Bouyoucos 1927, Köhn 1928, Casagrande 1934) and now allow achieving results with satisfactory repeatability and validity (Rząsa and Owczarzak 2013). In terms of dimensions below 0.1 mm, they use differences in settling velocities of soil particles, with different equivalent diameters (Gee and Bauder 1986).

Diversified grain settling velocity results in measurable changes in the density of the suspension both in terms of time and distance from the suspension surface. Measurements of the suspension density are currently performed in a variety of ways: suspension is taken up and evaporated (Indorante et al. 1990, Gee and Or 2002), measured with a hydrometer (Komornicki and Jakubiec 1978, Ryżak et al. 2009), measurement of X–ray absorption (Buchan et al. 1993) and even measuring the pressure of the suspension at a given depth (Zhang and Tumay 1995, Kovács et al. 2004, Durner et al. 2017).

Sedimentation analysis is supplemented with sieve analysis due to the settling velocity of grains with diameters greater than 0.1 mm preventing sample absorption or measuring its density by means of a hydrometer in a given time. Therefore, two separate physical phenomena (sedimentation and sieving under dry and wet conditions) are used to determine the soil grain size distribution. The set of fractions determined by the sieve method is defined by the number and size of meshes used in the screens.

Recently, a new method has been proposed (Kaszubkiewicz et al. 2017) for determining the suspension density and hence the soil grain size distribution. The method is based on the measurement of the apparent weight of a float immersed in the suspension over time. The apparent weight measurement is performed using a sensitive piezoelectric dynamometer.

The change of a float's position in the suspension during the measurement is in fractions of a millimeter. Measurements of the suspension density with a float can therefore be performed at a strictly selected depth

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with a frequency of up to 0.1 s. In this article, this will be called the dynamometer method.

The use of new measurement method obviously raises the problem of comparability of results with that obtained so far, the quantity of accidental and systematic errors and test result repeatability. The first tests of the method showed its satisfactory compliance with the results obtained in the pipette method and correctness of results for artificially prepared soil mixtures (Kaszubkiewicz et al. 2017).

The aim of this study is to evaluate the conformity of the results of the dynamometer method with the hydrometer and pipette method for broader experimental material covering the soils of different textural groups.

MATERIALS AND METHODS

Samples representing 59 genetic horizons of soils of varied structure and genesis have been selected for the research and measurement evaluation. In total, in terms of grain size distribution (measured using the hydrometer method), these samples belonged to the following granulometric groups according to PTG (PTG 2008): sands-16, sands, loamy sands-4, sandy loams-5, loams-5, clay loams-5, silty clay loam-1, silt loams-12, clays-7, heavy clays-4. The samples were characterized by median values of diameters (d_{50}) ranging from <0.002 mm to 0.245 mm. The average median value calculated for samples for which the value of d_{50} (45 samples) could be read was 0.099 mm and the standard deviation was 0.081. For 14 samples the value of d_{50} was below 0.002 mm, which made it impossible to calculate (the extrapolation method was omitted as unreliable).

The samples contained less than 1% of $CaCO_3$ (as determined by Scheibler's method) and less than 1% of organic carbon content (as determined by wet oxidation method).

In all samples, the grain size distribution was determined by Casagrande's hydrometer method modified by Prószyński (according to PN- ISO 11277, 2005) by pipette method in accordance with Köhn (Gee and Bauder 1986) and by the dynamometer method. In the case of pipette and hydrometer method, the methodology described in the work of Ryżak et al. (2009) has been applied. The dynamometer method is described in Kaszubkiewicz et al. (2017). It consists in determining the density changes of the suspension at a depth z in time t by measuring the apparent weight of the float immersed in it.

Taking change in density into consideration, the content of individual soil fractions can be calculated using the Stokes equation (1850). In relation to the method described in the above cited work, the following modifications were used: the shape of the float was changed and at the same time its volume was increased to 41.48 cm^3 and weight (in the air) to 49.26 G, a thin metal low stretch wire was used instead of a monofilament to hang the float and continuous temperature measurement was introduced. Changes were also made to the software, taking into account the time elapsing from the end of mixing to pressing the automatic measuring switch, and calculation of the mean of several results for short measurement times was improved.

The dynamometer measurements were made again for 23 samples at an interval of 24 hours to determine repeatability. In the dynamometer method the content of fraction <0.002, 0.002–0.004, 0.004–0.006, 0.006–0.008, 0.008–0.016, 0.016–0.02, 0.02–0.032, 0.032–0.05, 0.05–0.063, 0.063–0.1 mm was determined. The content of fraction 0.1–0.25, 0.25–0.5, 0.5–1.0 and 1.0–2.0 mm was determined entirely by sieve method. Content of fractions <0.002, 0.002–0.063, and 0.063–2.0 mm used for comparisons of the three methods were calculated by appropriate summation.

With the hydrometer method the standard set of fractions <0.002, 0.002–0.006, 0.006–0.02, 0.02–0.05, 0.05–0.1 mm was determined. The content of fractions 0.1–0.25, 0.25–0.5, 0.5–1.0 and 1.0–2.0 mm was determined by sieving.

The fraction of particles with equivalent diameters below 0.063 mm was calculated by interpolation using a particle distribution model, which was a renormalized lognormal function (Buchan 1989, Buchan et al. 1993, Esmaeelnejad et al. 2016). Subsequently, the content of fraction <0.002, 0.002–0.063, and 0.063–2.0 mm was determined by appropriate summation.

With the use of pipette method, the content of fractions <0.002, 0.002–0.063, and 0.063–2.0 mm was measured.

RESULTS AND DISCUSSION

Compliance with the pipette method

The pipette method is considered the reference when determining grain size distribution. It is essentially used to validate other methods (Syvitski 1991, Allen 1997, Orzechowski et al. 2014). The results obtained are characterized by high repeatability. It uses a simple and understandable mathematical model of the phenomenon of sedimentation (Stokes 1850, Dietrich 1982). Its main shortcomings are, of course, considerable labour and time consumption. It was also treated as a reference method in the presented work.

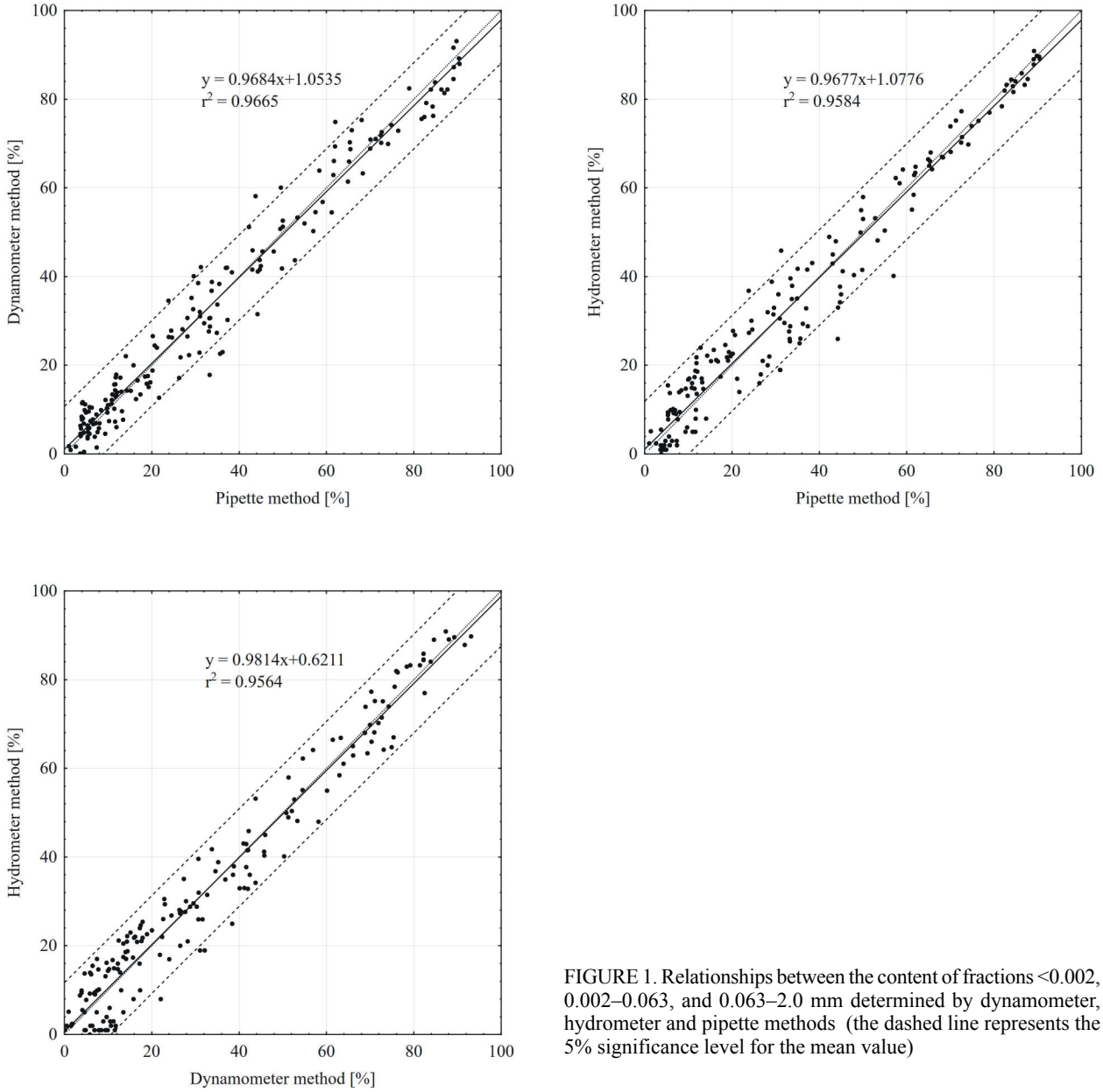

FIGURE 1. Relationships between the content of fractions <0.002, 0.002–0.063, and 0.063–2.0 mm determined by dynamometer, hydrometer and pipette methods (the dashed line represents the 5% significance level for the mean value)

TABLE 1. Comparison of results obtained using the dynamometer and pipette method

Fraction mm	No. of samples	Regression equation	Correlation coefficient	Std. error of slope coefficient	Root mean square error	Std. error of regression	Critical value*
0.063–2.0	59	y=0.9625x+0.2739	0.9951	0.0127	3.7275	3.2931	-0.2846
0.002–0.063	59	y=0.9992x–0.5335	0.9681	0.0343	5.4107	5.4750	0.5340
<0.002	59	y=1.0217x–1.4148	0.9699	0.0340	5.3985	5.1183	-1.3848
All fractions	177	y=0.9684x–1.0535	0.9831	0.0136	4.9096	4.8634	-1.0879

* x value (fraction content measured in the pipette method) for which y = 0

The content of fraction <0.002, 0.002–0.063, and 0.063–2.0 mm were determined directly using the pipette method. The determinations were made for 59 samples obtaining a total of 177 measurements. Suspensions for dynamometer and pipette measurements were prepared separately. The results of the comparison for the pipette and dynamometer methods are presented in Figure 1and 2. Selected statistical parameters are summarized in Table 1.

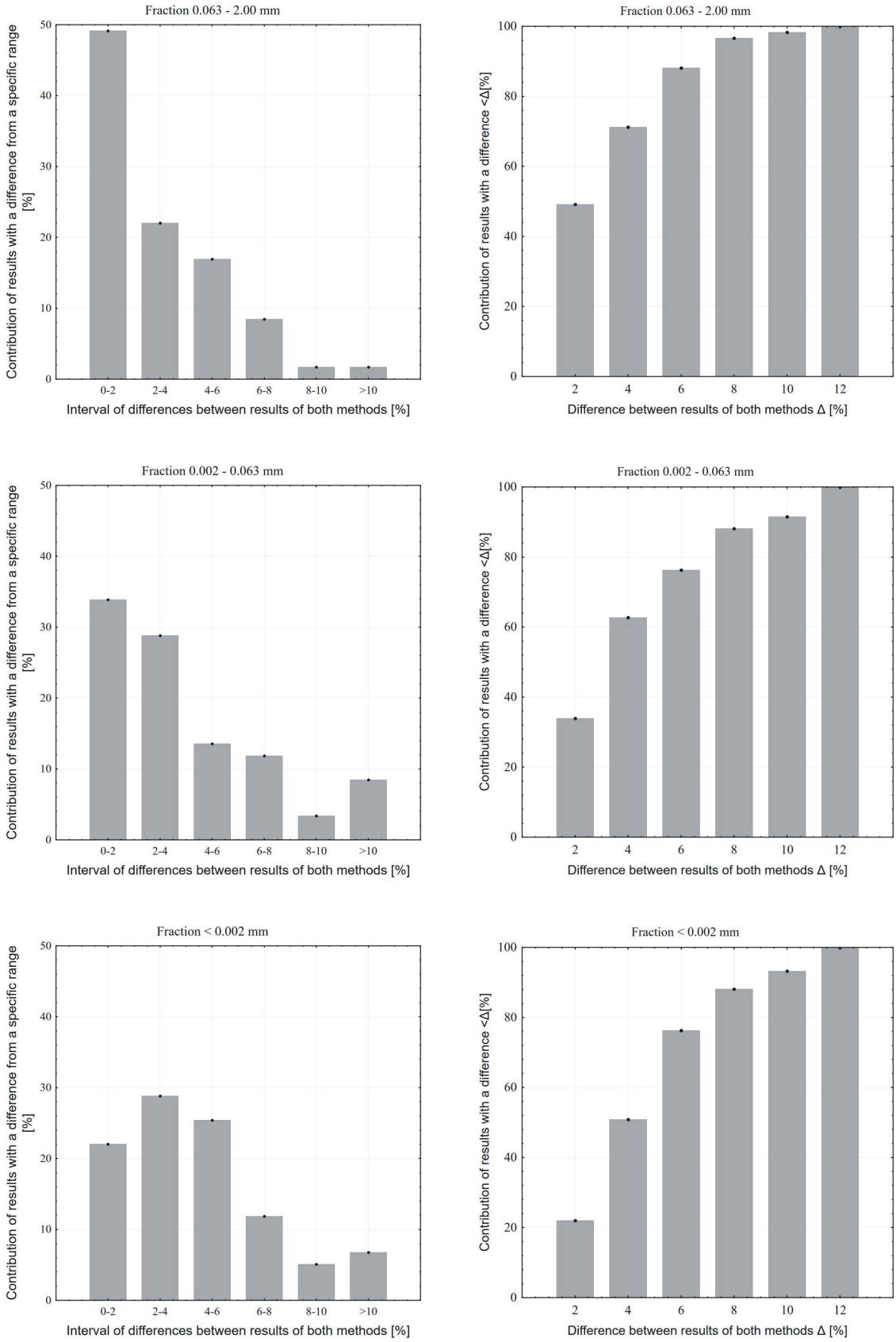

FIGURE 2. Distribution functions and histograms of differences (absolute values) between the results obtained with the use of pipette and dynamometer method for the three tested fractions

TABLE 2. Comparison of results obtained using the hydrometer and pipette method

Fraction mm	No. of samples	Regression equation	Correlation coefficient	Std. error of slope coefficient	Root mean square error	Std. error of regression	Critical value*
0.063–2.0	59	y=0.9204x+5.4505	0.9957	0.0113	4.4060	2.9203	-5.9222
0.002–0.063	59	y=0.8959x+4.2248	0.9588	0.0352	5.9668	5.6116	-4.7159
<0.002	59	y=1.0483x-3.8113	0.9704	0.0345	5.8603	5.2002	3.6355
All fractions	177	y=0.9685x+1.0489	0.9791	0.0152	5.4577	5.4228	-1.0829

* x value (fraction content measured in the pipette method) for which $y = 0$

Trend lines for the relationship between the results of both methods only slightly deviated from the line $y = x$. The trend line equation for all fractions together is $y = 0.9684x–1.0535$, and the correlation coefficient is 0.9831. The results for individual 3 fractions look similar when analysed separately. Trend line slope coefficients for the all fractions are within the range of 0.9625–1.0217 and are significant at $p < 10^{-6}$. The root mean square error was the highest for the 0.002–0.063 fraction and was 5.4107, and the lowest for the 0.063–2.0 mm fraction was 3.7275. For 25% of measurements, the absolute difference between the results obtained by both methods (all fractions evaluated together) was less than 1.36% and for 50% of the results did not exceed 3.03%, and for 75% of measurements it was less than 5.29%. Differences greater than 10.50% were found in 5% of measurements.

The observed scheme of results indicates the lack of major systematic errors in the dynamometer method and the occurrence of some random errors that require elimination or at least a reduction in the course of further development.

For the hydrometer method evaluated in the analogous system, with the acceptance of the results of the pipette method as the reference, the following results were obtained. The trend line equation for all fractions together is $y = 0.9685x–1.0489$, and the correlation coefficient is 0.9791 (Table 2, Fig. 1). The slope coefficients for the three analysed fractions are more diverse than for the dynamometer method. They are within the range of 0.8959–1.0483 and are significant at the level of $p < 10^{-6}$. The root mean square error was the highest for the 0.002–0.063 fraction and was 5.9668, and the lowest for the 0.063–2.0 mm fraction was 4.4060.

The course of the trend line and critical values indicate systematic underestimation of fraction <0.002 in all samples by about 3–4% in relation to the pipette method and overestimation of fraction 0.002–0.063 mm in sandy soils and also overestimation of fraction 0.063–2.0 mm in all tested samples (Table 2, Fig. 2).

For 25% of measurements, the absolute difference between the results obtained by both methods (all fractions evaluated together) was less than 1.64%, for 50% of the results it did not exceed 3.41%, and for 75% of the measurements it was less than 6.02%. In 5% of measurements, differences greater than 10.28% were detected.

Both dynamometer and hydrometer methods showed compliance with the results of the pipette method at a similar level, with the correlation coefficients differing slightly as did the mean square errors. The presented dynamometer method does not show systematic deviations, and errors are accidental. On the other hand, instead of random errors, the hydrometer method has also a systematic error, which, in relation to the pipette method, underestimates the fraction of the clay at the same time overestimating the content of sandy fractions. Warzyński et al. (2018) arrived at similar conclusions.

Repeatability of results obtained with the dynamometer method

The repeatability of results obtained in the dynamometer method was evaluated for the same fractions as described above. The repeat measurement was carried out 24 hours after the previous one, in the same suspension after possible supplementation of small water losses associated with evaporation. Measurements of individual fractions were made at the same depths and after the same times. Possible small differences in the experimental conditions could be related to temperature changes in the laboratory.

In a system of this kind, random factors such as ground vibrations transmitted to the device, changes in temperature during the measurement, instability of the electronic components of the measurement system (drift error) and other factors difficult to determine may have influenced the results. The only systematic factor that could affect the results would be the possible breakdown of aggregates or even grains during the period between the first and the second measurement.

The tests were repeated for 23 samples and in total 69 fractions were analysed. The results are presented in Figure 3. The statistical parameters are summarized in Table 3. As can be seen, the trend lines for the relationship between measurements for 0.002–0.063, 0.063–2.0 mm fractions and for all the fractions together practically do not differ from the $y = x$.

The only trend line for the comparison of two measurements of fraction contents <0.002 mm clearly deviates from the line $y = x$. The slope coefficients of the trend line are significant at the level of $p < 10^{-6}$. The value of the correlation coefficient for the total of 3 fractions is 0.9947. For 25% of the results, the difference between the first and the second measurement

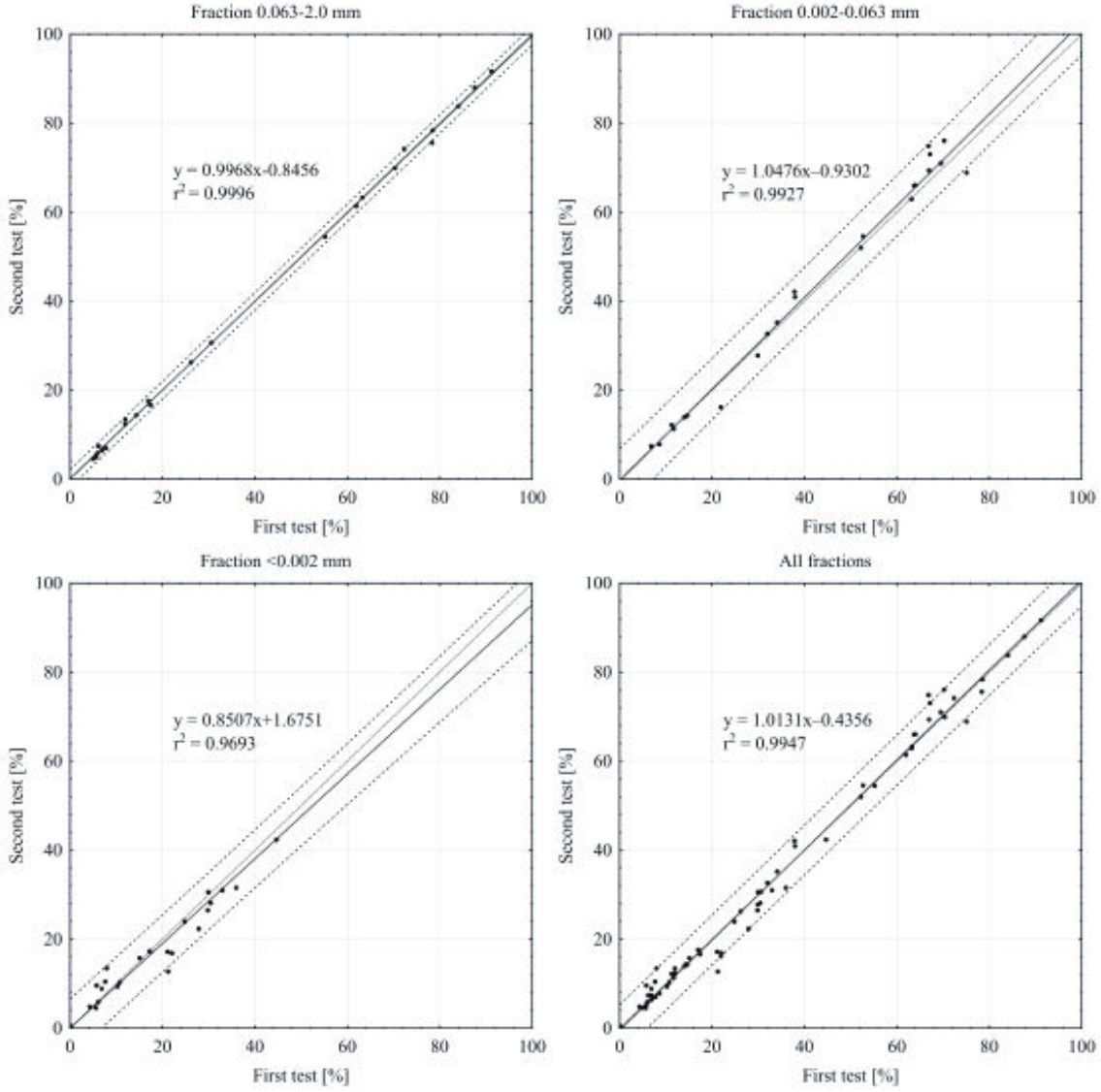

FIGURE 3. Comparison of results obtained in repeated measurements, for these same soils, with the use of dynamometer method

TABLE 3. Comparison of repetitions in the dynamometer method

Fraction mm	No. of samples	Regression equation	Correlation coefficient	Std. error of slope coefficient	Root mean square error	Std. error of regression	Critical value*
0.063–2.0	23	y=0.9968x–0.8456	0.9996	0.0061	0.9043	0.9395	-0.0848
0.002–0.063	23	y=1.0476x–0.9302	0.9927	0.0279	3.3731	3.1323	0.8879
<0.002	23	y=0.8507x+1.6751	0.9693	0.0471	3.3019	2.6956	-1.9691
All fractions	69	y=1.0131x–0.4356	0.9947	0.0128	2.7747	2.7944	0.4299

* x value (fraction content measured in the pipette method) for which y = 0

was less than 0.43%, for 50% of the results it was less than 0.84% and for 75% it was lower than 2.40%. In 5% of measurements, differences greater than 5.88% were found.

Analysis of repeated cumulative particle size distribution function (PSD–not included in the paper) it was revealed that the differences between measurements are twofold. In the first case, a single point on the PSD curve in one of the repetitions clearly stands out from the second repetition and additionally from the trend for the whole curve. In the second, we deal with a more systematic deviation of the course of both curves at a certain distance.

Achieving even better repeatability of measurements by eliminating random errors would most probably be possible after improving the insulation of the system from ground vibration and by stabilizing the laboratory room temperature.

The ordinary regression analysis used above is based on the assumption that an independent variable (fraction content measured with pipette method) is measured without error. The dependent variable is loaded with a measurement error.

In the case when both variables x and y are loaded with measurement errors, it is suggested the Reduced Main Axis Analysis is used instead of the usual regression (Smith 2009, Harper 2014). The simple regression in RMAA is determined in this way, so that the sum of the rectangular triangle fields between the measuring points and the straight one is the smallest. Using this regression model, the equations listed in Table 4 were obtained. As can be seen an analysis of results using both methods give almost the same parameters of regression equation.

The influence of some factors on the results of measurements

Under real conditions during sedimentation process, each particle experiences a different fluid resistance due to variable arrangements of adjacent particles, emerging local pressure gradients, eddies caused by larger, rapidly drooping grains, water countercurrents and wall effects (Ham and Homsy 1988, Syvitsky 1991, Nguyen and Laad 2005).

Variable fluid resistance due to the presence of other particles and the water movement caused by them means that settling velocity of grains depends on suspension concentration and composition. The actual settling velocity w is therefore different from the one calculated from the Stokes equation, and the difference increases with the suspension concentration. The literature contains many equations describing the relationship between w_0 and w considering the concentration of suspension and the size and shape of settling particles (van Rijn 1989, Cheng 1997, Ahrens 2000). The most commonly used equation binding both speeds is the empirical Richardson-Zaki formula (Richardson and Zaki, 1954):

$$\omega = \omega_0(1 - c)^m$$

where c is the volume concentration of the suspension, and m is the parameter determined experimentally. The Richardson-Zaki formula is used for suspensions with a volume concentration of $0.05 < c < 0.5$. For suspensions with $c < 0.05$ Batchelor (1982), (Batchelor and Wen 1982) introduced a modified formula:

$$\omega = \omega_0(1 - nc)$$

where the value of parameter n is from 5.5 to 6.5 (Silva et al. 2015).

Therefore, it can be expected that the results obtained in sedimentation methods, where the volumetric suspension concentrations used are at the level of 0.01–0.03 will depend on the applied sample determining the concentration of the suspension and thus the actual grain settling velocity.

The deviation from the Stokes equation will be greater in heavy soils due to the higher concentration of fractions remaining in the suspension for a long time. For example, for soil containing 50% of clay fraction at a weight of solid phase in the suspension equal to 60 g and the parameter value $n = 5.5$, the ratio ω/ω_o is 0.94.

The phenomenon is very complicated in nature because grain settling velocity depends on the grain concentration in suspension, and this on the grain size distribution, which is yet to be measured. The obvious solution of the problem would be to reduce the sample's weight and concentration, but this would require measuring the density changes that are smaller than now. This would increase the significance of random errors.

There was an attempt at an initial assessment of this phenomenon in terms of its impact on the results obtained in the proposed measurement method. The content of selected fractions ($f < 0.05$ and $f < 0.015$ mm) was measured by means of the dynamometer method at several depths after times calculated with the Stokes' equation.

As can be seen in Figure 4 and as shown in the paper of Kaszubkiewicz et al. (2017), results of measurements of the same fraction in the dynamometer method may vary by 2–4% depending on the measurement depth, and depending on the sample weight varies by 1 to 6%. Larger differences could be observed for larger depths of measurements.

As results from the Richardson-Zaki formula, the interaction of grains cause that its settling velocity is lower than calculated from the Stokes' formula. Therefore, the adopted measurement times are too short and, in the moment of measurement, in the suspension there are still grains with equivalent diameters larger than the assumed ones.

It can be expected that the difference between the assumed and the actual diameters will be greater for the fractions with larger equivalent diameters because they are measured in a suspension with a higher concentration, and therefore with larger deviations of the actual and calculated settling velocity.

A possible solution to the problem may be a reduction of the weight of soil sample and its differentiation due to initial organoleptic assessment. As a result, more homogeneous sedimentation conditions for different samples can be achieved.

The description of phenomena causing some differentiation of results for the same fraction obtained at different depths requires further theoretical and experimental research, which the authors plan to carry out in the near future.

Theexact approach to determine time of particle settling would also require determination of specific density of soil solid phase. It can be assumed that at low contents of organic matter, its impact on density is small (Blake and Hartge 1986). The impact of differentiated mineral density and hence the different density of fractions included in a single soil (Mocek et al. 2009) is not included in any of the available methods.

TABLE 4. Comparison of repetitions in the dynamometer method with the use of RMA analysis

Fraction	Equation
0.063–2.0	y=0.997x+0.069
0.002–0.063	y=1.055–1.258
<0.002	y=0.878x+1.185
All fractions	y=1.022x–0.008

However, one should not expect effects related to the particle's motion phase until reaching the final settling velocity when they accelerate after shaking the suspension (Allen 1997). According to Gee et al. (2002) the time required for a particle with a diameter of 5 mm to reach 99% of the final settling velocity is 0.017 ms, and for a particle of 1000 μm–1000 ms.

The deviation of particle shape from spherical has a significant influence on the settling velocity. In the literature on the subject, many empirical equations can be found to calculate the settling velocity of real soil grains (Gibbs et al. 1971, Ahrens 2000, Jimenez and Madsen 2003).

Various indexes describing the shape of grains are used such as the Coreya index (Jimenez and Madsen 2003) or the Janke index (Janke 1966). Any deviation from the spherical shape results in a decrease of the settling velocity in relation to the spherical grain of the same volume as the tested ones. On the other hand, the surface smoothness turns out to be less important (Baba and Komar 1981).

A simple solution to this problem seems to be the use of an equivalent diameter concept as the diameter of the sphere falling at a speed equal to the real shape grain. However, this is a solution that has a significant role in the emerging discrepancies between the sedimentation and optical methods (Polakowski et al. 2014).

The above comments apply equally to all three sedimentation methods, and the differences between the methods result mainly from the use of different sample weights and different measurement depths.

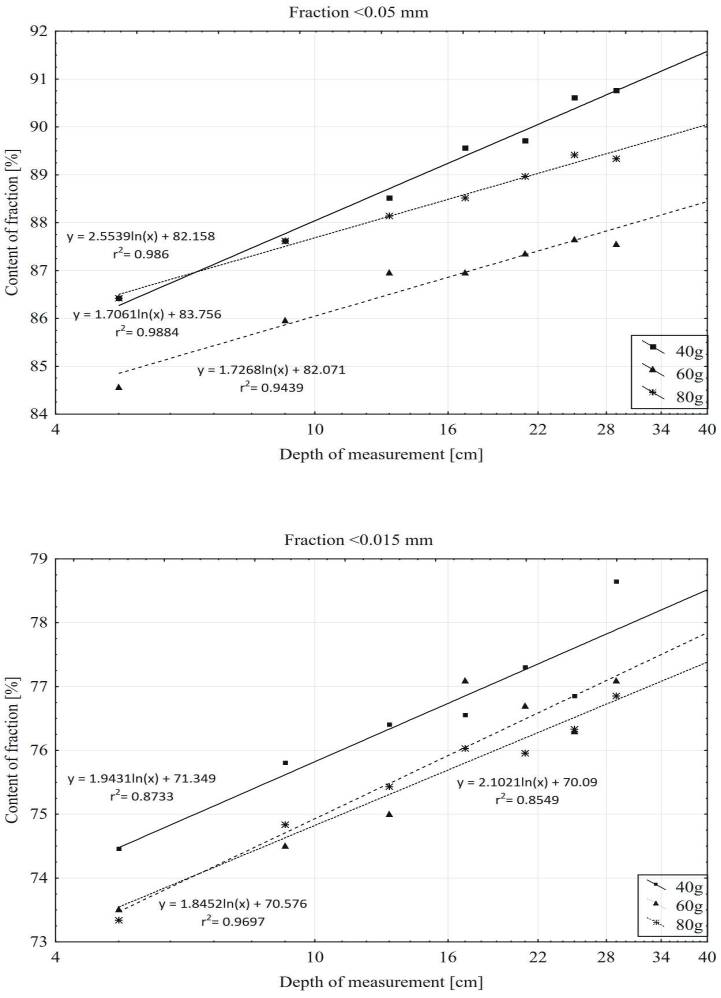

FIGURE 4. Comparison of results obtained for these same fractions, for different samples weights and at different depths (single selected sample)

SUMMARY

From a wide range of methods of grain size analysis none can be considered ideal (Goossens 2008), and their evaluation depends on the criteria used.

The method for determination of grain size distribution in the range of 0.002–0.1 mm diameters proposed by Kaszubkiewicz et al. (2017) gives a satisfactory compatibility of results with the results of the pipette method considered as a reference as well as the hydrometer method. There is no need to enter the calibration of dynamometer method to achieve the convergence of results with the pipette method.

The observed differences in results in relation to the reference method are accidental in nature, but no systematic differences were observed. Systematic differences were observed for the hydrometer method, which, in relation to the reference, underestimates the clay fraction content and overestimates the content of fraction 0.002–0.063 mm for sandy soils.

The dynamometer method shows good reproducibility of the results, with a slightly higher dispersion for the clay fraction and for the silt fraction (0.002–0.063 mm).

Subsequent improvements of the method both in terms of its physical aspects and the use of improved computational algorithms should lead to further improvement of compliance with reference and repeatability of results.

In order to reduce the effects of interaction between sedimenting particles on the results of the analysis, it is reasonable to standardize the concentration of suspensions for soils with different fine fraction contents. For this purpose, the team is going to introduce variation of sample weight depending on the organoleptic evaluation of soil texture.

Summarizing the main advantages of the developed method, these are, in addition to the consistency of the results with the reference method, a direct record of the results in digital form, the capacity to analyse multiple fractions with arbitrarily chosen ranges of diameters and the reduction of the analysis time in relation to other sedimentation methods.

ACKNOWLEDGMENTS

The research was carried out with the financial support from the Intelligent Development Operational Program, contract No. POIR.02.03.02-02-0008/16-01, project title: Significant improvement of the innovative device for measuring the grain size composition of fine-grained materials.

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Received: February 8, 2018
Accepted: May 28, 2018
Associated Editor: J. Rejman

Analiza składu granulometrycznego gleby metodą dynamometryczną – porównanie z metodą pipetową i areometryczną

Streszczenie: Celem przedstawianej pracy było porównanie wyników składu granulometrycznego zmierzonego za pomocą innowacyjnej metody dynamometrycznej, opracowanej przez autorów, z wynikami uzyskanymi w metodzie areometrycznej i traktowanej jako referencyjna, metodzie pipetowej. Określono również powtarzalność wyników uzyskiwanych w metodzie dynamometrycznej. Mierzono zawartość trzech frakcji o wymiarach <0,002 mm, 0,002–0,063 mm i 0,063–2,0 mm. Wyniki porównywano z zastosowaniem regresji liniowej, a przy analizie powtarzalności dodatkowo za pomocą analizy RMA (reduced major axis). Stwierdzono, że proponowana metoda dynamometryczna charakteryzuje się dobrą powtarzalnością wyników i brakiem błędów systematycznych przy porównaniu z metodą pipetową. Wartość RMSE (root mean square error) przy odniesieniu do metody pipetowej obliczona dla 3 frakcji rozpatrywanych łącznie wynosiła 4,9096 i była mniejsza od analogicznej obliczonej dla metody areometrycznej, dla której wyniosła 5,4577. Wartości współczynników determinacji przy porównaniu metod dynamometrycznej i pipetowej mieszczą się, dla różnych frakcji, w granicach 0,9681–0,9951. Stwierdzono, że nieco większe różnice wyników w relacji do metody pipetowej występują przy pomiarze frakcji <0,002 mm i 0,002–0,063 mm, a mniejsze dla frakcji 0,063–2,0 mm. Podobnie większe różnice pomiędzy powtórzeniami w metodzie dynamometrycznej zaobserwowano dla frakcji <0,002 mm, a mniejsze dla frakcji 0,063–2,0 mm. Przedyskutowano możliwe źródła błędów w metodzie dynamometrycznej i określono ewentualne sposoby ich redukcji.

Słowa kluczowe: skład granulometryczny, metoda dynamometryczna, metoda pipetowa, prędkość opadania ziaren