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Review

Soil particle-size analysis up to 250μm by X-ray granulometer: Device set-up and regressions for data conversion into pipette-equivalent values

M.C. Andrenelli [a,*], V. Fiori [b], S. Pellegrini [a]

[a] CRA-ABP, Centro di Ricerca per l'Agrobiologia e la Pedologia, Piazza M. D'Azeglio 30, 50121 Firenze, Italy
[b] UNISS, Dipartimento di Ingegneria del Territorio, Sezione di Geopedologia e Geologia applicata, Viale Italia 39, 07100 Sassari, Italy

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ABSTRACT

The particle-size distribution is a fundamental soil physical property commonly used for attributing the textural class and, given its strict linkage with both several soil edaphyc properties and geomorphology processes, it represents a routine lab determination.

Over recent decades, various new methods for grain-size analysis have been developed, among them, the X-ray granulometer represents the unique innovative technology based on the Stokes' law, directly comparable with the standard methods (pipette and hydrometer). The authors illustrate the possibility of employing the Micromeritics SediGraph 5210 device for analyzing soil particles up to 250μm. This aim involves some modifications to the soil sample preparation along with a proper set up of the apparatus in order to assure the conformity with the Stokes' law.

A data set of 180 Italian soils, distributed over the entire soil textural triangle, was analyzed by both the SediGraph and pipette methods. Two-thirds of samples were employed for the calibration phase while the remaining part was used for validation.

A set of six multilinear regressions, one for each analyzed grain-size class, was developed to convert the SediGraph data into pipette-equivalent values. Regardless of the high significance level ($p<0.001$) of all the regressions, the coefficient of determination was always larger than 0.87, with the only exception of very fine sand (50–100μm) fraction ($R^2=0.64$).

No regressions were needed when SediGraph clay content is ≥68%; in such a case no conversion was required because SediGraph results match with the pipette data.

Contents

* Corresponding author. Tel.: +39 0552491246; fax: +39 055241485.
E-mail address: mariacostanza.andrenelli@entecra.it (M.C. Andrenelli).

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1. Introduction

The measurement of particle size is a basic analysis in many activity sectors, such as pharmaceutical, ceramics and steel industry, and soil science. Hence, the need has arisen to develop cutting-edge technologies able to improve the analysis reliability reducing the test duration. In soil science, in particular, the particle size analysis is one of the routine lab determinations, because information on soil texture is crucial for examining topics upon soil chemical and mineral compositions (Murashkina et al., 2007), soil organic carbon content (Zhang et al., 2005), hydraulic characteristics (Bittelli et al., 1999; Gawlik et al., 1999), tillage and land management (Basic et al., 2001), plant growth (Lipiec et al., 2007), and erosion and desertification phenomena (Su et al., 2004; Warrington et al., 2009). Truthful and comparable data on soil texture are crucial for such studies (Vdovic et al., 2010).

The conventional and most widespread method for determining the particle size analysis of soils is the "pipette method" (Gee and Bauder, 1986), which includes the sedimentation of the fine fractions (silt and clay) and the sieving of the coarse particles (sand). The main advantages of its employment are the possibility of comparing the results provided by different laboratories, its consolidated analytical procedure, the low cost of the apparatus and the acceptable data accuracy. Nevertheless, such a method has some important drawbacks: the prolonged analysis time, mainly when the clay fraction has to be investigated in detail, the impracticality of attaining a continuous granulometric curve, the quality results dependent on laboratory technique and operator skillfulness; in addition, a large amount of soil sample ($\geq$10 g) is required (Beuselinck et al., 1998).

To overcome these limitations, during the last decades new high-tech instruments able to increase the efficiency of grain size analysis were developed; among the most common apparatuses there are the ones based on X-ray attenuation (e.g., Micromeritics SediGraph), laser diffractometry, transmission electron microscopy (TEM), image analysis and electroresistance particle counting (e.g., Coulter counter).

Compared to the pipette method, they cover a wider but variable range of grain sizes, from the minimum diameter of 0.02 μm (laser technology) to the maximum diameter of 10,000 μm (TEM apparatus), require a small sample quantity to be tested, and need short analysis time. The use of these new techniques raises the question of how comparable the results are with respect to those obtained by more conventional methods (e.g., pipette or hydrometer). It is worth pointing out that only the X-ray attenuation technique provides the results in terms of mass percentage, easily comparable with those of the pipette, while all the other device outputs are expressed in terms of volume percentage.

The X-ray sedimentometer, represented by different updating of the Micromeritics SediGraph technology (Micromeritics Instrument Corp., Norcross, GA, USA), was available to soil scientists since more than four decades; nevertheless, the comparison with the standard pipette method provided conflicting results (e.g., Watts et al., 2000). Welch et al. (1979) observed divergences of the measurements but did not succeed in explaining satisfactorily the reason, while Weaver and Grobler (1981) found that the sedimentometer systematically gave finer particle size distributions compared with other methods (pipette, hydrometer, microscope); however, the discrepancies in results obtained with the diverse apparatus were not clear and the microscopic analysis of the samples supported the results obtained by the SediGraph method. Lara and Matthes (1986) suggested employing this technique as a valid alternative to the pipette because they found excellent agreement, especially for samples having a large percentage of clay-sized material, although Stein (1985) noted that errors might be quite large for samples containing about 50% and more of montmorillonite. Actually, Stein (1985) observed that under certain conditions the viscosity can increase so far that no further sedimentation is possible. This author also observed hindered settling effects in case of too high concentrations of the suspension that may have caused the overestimation of the clay fraction as a consequence of an increased particle–particle interaction. Buchan et al. (1993a) agree with the X-ray sedimentometer overestimation of the fine fraction compared to the pipette, attributing such differences to either different sample preparation techniques or to the presence of specific minerals such as Fe-oxides, mostly concentrated in the fine fractions, which attenuated the X-rays more than the other silicate particles.

All the comparative studies between X-ray sedimentometer and pipette have been carried out in investigating the performance of the two techniques below 63 or 50 μm; the sand fraction (>50 and 63 μm according to USDA and German classification schemes, respectively) was always determined by sieving, so that possible discrepancies between the two approaches may be related to differences in soil sample quantity and/or to different techniques used in soil preparation (Müller et al., 2009). In order to avoid such drawbacks, Delaune et al. (1991) employed the same soil sample quantity (30 g) for both the analytical procedures, then collecting 20 cm^3 and 50 cm^3 from the same suspension for pipette and X-ray sedimentometer analyses; in such a case, therefore, the sand weight was unique. Nevertheless, such a procedure was imposed following the analyses by the SediGraph in the use of a large amount of soil sample and did not reduce the time for washing and weighting sand fractions, separately determined by sieving.

Only few attempts to convert X-ray sedimentometer data into pipette measurements, or vice versa, by means of regression equations have been carried out. The former effort was carried out by Delaune et al. (1991) finding highly significant linear-regressions for coarse and fine silts, and clay by considering 34 soil samples ranging from 1 to 95% of sand and between 8% and 72% of clay contents. Afterward, Sporlein et al. (2004), based on a data set of 30 soil samples, obtained highly significant linear-regressions for coarse, medium and fine silts, and clay. The more recent work by Müller et al. (2009), based on a higher number of soil samples (n = 482) developed independent multi-regressions for converting X-ray sedimentometer data into pipette-equivalent results. In addition, the authors considered further elaboration to guarantee that the sum of all the estimated fractions was equal to 100%, a condition not always verified by the equations elaborated by previous studies.

Up to now, no attempt has been carried out about the possibility of employing the Micromeritics SediGraph X-ray sedimentometer for analyzing a part of the sand fraction too, though the apparatus is able to determine particles up to 300 μm. In particular, Coates and Hulse (1985) observed that the SediGraph was best suited to sand-free samples with abundant clay-sized material finer than 16 μm. In that regard, the common dispersing solution employed for preparing the soil sample (i.e., distilled water with Na-hexamethaphosphate) does not allow, at the SediGraph working temperature (35 °C), the maintaining of the Reynolds number beyond 0.21, as requested by the laminar flow condition.

Nevertheless, the possibility of increasing the analysis size range would significantly and conveniently reduce the soil sample preparation time, meanwhile decreasing the possible errors due to the operator. Coates and Hulse (1985) also outlined that the advantage of employing the SediGraph as an alternative to the traditional