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Shall we abandon sedimentation methods for particle size analysis in soils?

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ABSTRACT

For many years papers have been published showing differences between sedimentation-based methods against laser diffraction. Differences were found especially in the fine texture ranges and regression equations were presented to convert data obtained between different methods. In this paper we aimed at understanding which method is closer to an independent measurement of particle size. We selected a new, automated image analysis technique as a reference method. Since with this new method each individual particle is photographed, its pixels counted and its shape analysed, we assumed this method as the reference method against which test the accuracy of sedimentation methods and laser diffraction. Comparison showed that laser diffraction was in better agreement with the independent optical methods, indicating that the sedimentation methods tend to largely overestimate the finer fractions of the distribution. Considering the results presented in this research, and the methodological disadvantages of sedimentation methods, we propose to employ laser diffraction as a standard method for particle size analysis in soils.

1. Introduction

Particle size distribution (PSD) of soils is a basic property that affects a large number of soil variables and processes, such as soil water retention, hydraulic conductivity, thermal properties and geo-mechanical properties. A large variety of methods to measure the PSD have been developed (Allen, 1981; Gee and Or, 2002; Goossens, 2008).

The pipette method (P) has been recommended as a standard for measurement in mineral soils (International Standards, ISO 11277, 2009). This method has been used for many decades and the obtained data populated soil maps and databases worldwide. Another method based on sedimentation theory is the Sedigraph (S) (Micromeritics Instrument Corp., Norcross, GA, USA), which measures the X-ray absorption and results are equally expressed as percent by mass. Although sedimentation methods are still the standard, they have many disadvantages: (a) small range and limited number of size classes when compared to other techniques such as laser diffraction, (b) lack of reliable data at smaller sizes ($< 2\,\mu m$) due to Brownian motion effects on sedimentation times (Loveland and Whalley, 2001), (c) long analysis time and, (d) assumptions about the homogeneous density and sphericity of all the particles (Clifton et al., 1999). Due to these limitations, many researchers investigated the potential of developing other techniques.

Among the alternative available techniques, Laser Diffraction (LD) is becoming widely utilized, since it has several advantages (Allen, 1981). Sample analysis by LD is fast, covers a wide range of size classes, provides many data points allowing for obtaining a detailed PSD and the amount of soil needed for the measurement is small. Many researches have been performed in Laser Diffraction. Haynes (2008) studied the effects of the Refractive Index (RI) on PSD analysis. Eshel et al. (2004) performed several tests on the variation of PSD obtained from LD and reported that a value of RI = 1.53 was suitable for most soils. This is consistent with reporting of Jonasz (1987), who reported that the scattering cross-section for a particle increases with the real part of the RI. Ozer et al. (2010) presented similar values of RI and AC of 1.55 and 0.1 for laser diffraction in naturally soils. Moreover Eshel et al. (2004) showed “overestimation” of clay content with sedimentation methods when compared to laser diffraction.

Studies have been performed to assess the most suitable medium of suspension and method of dispersion for a Malvern laser sizer on sandy soil (Chappell, 1998). Vdovic et al. (2010) investigated the effects of sample pre-treatment and performed a comparison among different methods. Storti and Balsamo (2010) investigated the effect of dispersing methods and properties of the dispersion units for different volumes and pump speed on the PSD results for sands.

Újvári et al. (2016) discussed the importance of grain size analysis

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for Quaternary studies. Schulte and Lehmkuhl (2017) exploited the differences in LD results given by the application of the Mie and Fraunhofer theory, to highlight the enrichment of fine-grained material by post-depositional chemical weathering.

The Mie theory considers the complex refractive index that depends on the mineral composition, therefore allowing for more detailed analysis and information about mineralogical composition, when compared to the simplified Fraunhofer approach. Indeed, the Mie scattering theory and the Fraunhofer diffraction theory are used to obtain the relationship between particle size and light intensity distribution pattern, therefore are used to compute what kind of light intensity distribution patterns are produced by particles of various sizes. The Mie solution of the Maxwell′s equations describes the scattering of an electromagnetic wave by an homogeneous sphere or by stratified spheres, therefore it strictly applies to spherical, homogeneous particles and it requires knowledge (or assumptions) about the real (i.e. refraction) and imaginary (i.e. attenuation) parts of the complex Refractive Index (RI). It is a more rigorous solution accounting for particle properties such as color and mineralogical composition affecting refraction and attenuation. The Fraunhofer diffraction equation is a simplified equation used to model the diffraction of waves when the diffraction pattern is viewed at a long distance from the diffracting object. de Boer et al. (1987a, 1987b) pointed out that LD is often claimed to operate on the principle of Fraunhofer diffraction, while this is only true if particles are large compared to the wavelength of light or if the ratio of the refractive indices of the disperse and continuous phases is clearly different from unity. In their paper, they presented and discussed the differences between Fraunhofer and Mie theories. Di Stefano et al. (2010) analyzed 220 soil samples from Sicily using laser diffraction and sedimentation methods. They found no significant differences in using ultrasound or not, as a sample pre-treatment. They showed no significant differences in sand content between sedimentation methods and laser diffraction, while they confirmed the overestimation of clay percentage with sedimentation methods. Roberson and Weltje (2014) presented a methodological framework for the inter-comparison of different particle-size analyzers. They concluded that laser diffraction based instruments were best able to distinguish between different populations of particle-size distributions at a 95% confidence level.

Overall, after almost five decades of research, laser diffraction for particle size analysis is progressively becoming an established technique. The standard for laser diffraction analysis of particles is the ISO 13320, although specifics about soil sample preparation and dispersion are not detailed.

Another technique that is gaining popularity is digital imaging (DI), usually by employing optical microscopy. Carter and Yan (2005) presented a study where DI was used for particle shape determination, while Carter et al. (2006) compared image analysis and laser diffraction for powder used in industrial applications, in the range between 25 and 150 μm. When DI and LD were compared in this range, they showed good agreement between methods. Chen et al. (2013) obtained similar particle size distributions, where comparison of DI and LD, providing comparable PSDs, in the sand fraction. The authors stressed the limitation of their device in measuring particles with a lower limit of 24.83 μm. Pieri et al. (2006) employed image analysis of images obtained from transmission electron microscopy (TEM) and found that when compared to the pipette method, the latter overestimated the amount of clay. Tinke et al. (2008) compared DI from static image analysis (as well as scanning electron microscopy (SEM) for morphological studies) against LD and they found good agreement between the methods. One of the main limitations of DI is the investigation of small number of plates, on which particles are dispersed. Given that image analysis systems are commonly assembled in laboratories with camera and digital acquisition systems (Pieri et al., 2006; Chen et al., 2013), the number of observed particles is small with respect to the whole sample. This problem determines a limit on the representativeness of the whole sample, which distribution is described assuming that a limited number of plates represent the sample as a whole. In this case for instance, the presence of a few large particles, over a relatively small total number, can bias the distribution. Veghte and Freedman (2014) investigated aspect ratios of different minerals, by using scanning electron microscopy and image analysis techniques.

To elucidate the effect of experimental differences and assumptions among different methods, it is convenient to employ a method that does not rely on the same fundamental assumption. In this case a method that does not rely on the sphericity of the particles.

Digital Imaging derives the diameter of each particle by using different geometrical parameters, after counting the pixels occupied by the particle on the digital image. This is an important advantage since allows for an independent comparison among methods; however, the main limitation is usually the manual acquisition and digital analysis of each sample plate, limiting the number of acquired images and therefore making the measurement either biased or not representative of the sample. To overcome this problem, new commercial automated morphological methods based on optical microscopy are now available, where millions of particles can be photographed and analyzed at various levels of magnification. Moreover, the possibility of photographing and digitally analyzing each single particle, allows for investigation of many parameters related to the shape of particles, therefore performing a more in-depth analysis of the effect of particle shape on the distribution and on the potential errors introduced by the different methods. Overall, while many comparative studies have been performed thus far, a complete comparative analysis using new optical methods that provides automated measurement of millions of individual particles have not yet been presented. Moreover, for many years there have been questions about the “overestimation” of clay particles from sedimentation methods. In this paper we aimed at understanding which method is closer to an independent measurement of particle size and if laser should be proposed as the standard method for particle size analysis. The objective of this study is therefore to perform a comparative analysis where sedimentation methods and laser were compared and validated against an independent, new, automated optical technology.

2. The soil samples

In this study eleven soil samples collected in different Italian pedoclimatic environments, were analyzed. Table 1 lists information on the sampling sites in terms of geographical coordinates, elevation, administrative region and parent material, while Table 2 shows basic chemical and physical analysis.

In particular, the soils from Sardinia developed on Pleistocene alluvial deposits and are characterized by a xeric moisture regime and by a free internal drainage; little sodium is present on the exchange complex, and a moderate development of argic horizon may occur. The light pink-red colour observed in these samples is typical of well-developed soils.

Soils from Lombardy developed on the Pleistocene fluvial and fluvioglacial deposits of the high Po valley; these are clay or silt calcareous gravel debris deposits, under sub-humid/humid climate regime with mean annual rainfall of around 900 mm. All the soils are freely drained and deep to hard rock. Typically, the Ap horizon is characterized by loamy texture and brown yellowish colour.

Soils from Tuscany, despite the quite homogeneous texture, differ for parent material, geomorphology, climate and land use. Soil 163, classified as Aquic Haploxerepts, developed on steep slope composed of marine clayey deposits, which land use is abandoned olive orchard, subjected to important rill and sheet erosion phenomena. The surface Ap horizon is characterized by a very low hydraulic conductivity, and the occurrence of redox mottles below 0.10 m. Soil 181, classified as Typic Haploxeralfs, is developed on colluvial deposits of siliceous metamorphic rocks. Soil 189, classified as Aquic Haploxerepts, is developed on steep slopes made of travertine subjected to sheet erosion. The