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Inherent factors limiting the use of laser diffraction for determining particle size distributions of soil and related samples

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ABSTRACT

A number of reports have proposed that recently developed laser diffraction instruments show potential for automating the measurement of particle size distributions (texture) in soil and related materials, but relationships to standard sieve-sedimentation measurements have been poor and inconsistent. Measurements with a commercially available laser diffraction instrument (Horiba LA-920) on five soils having a wide range of textures (silty clay to sand) showed wide variability for proportions of clay, silt and sand when the weight of the sample was varied resulting in unacceptable consistency and conformity with hydrometer measurements. Detailed measurements were subsequently made on systematic combinations of four known silt size fractions (0–5, 38–45, 75–90 and 125–150 μm diameter), to explore the reasons for the variable results. Samples weighing less than 0.10, 0.15 and 0.20 g of the 38–45, 75–90 and 125–150 μm diameter fractions, respectively, were not detected by the instrument. Calculations of the probable numbers of particles in these samples ranged from 0.06×10^6 to 1.0×10^6. There were, however, 2445×10^6 particle in a 0.05 g of 0–5 μm diameter sample, and was detected by the instrument. This showed that there is a threshold of the number of particles required for detection. Alternatively, similar and greater weights of small diameter particles result in potential saturation of the detector. Saturation occurred when laser light transmission was low (<20%). Even before saturation of the detector, an interaction was detected when combinations of small and large particles were mixed such that the proportion of small particles tended to be over-estimated and large particles under-estimated. Percentage laser light transmission measurements were inadequate to guide development of calibrations to correct for threshold/saturation limits and light competition problems. It was concluded that the geometric change in numbers of particles as the size decreases in a given weight of sample is too large to allow the use of current laser diffraction instrumentation for particle analyses of soil and related samples where the sizes in the distribution are from clay to sand or even with a narrower range of sizes (e.g., clay or silt).

1. Introduction

Texture, the distribution of different particle size fractions, is a fundamental characteristic of soil which has a profound influence on physical, chemical and biological processes. Sieving and sedimentation methods have been widely adopted to measure the proportions of sand, silt and clay fractions (Day, 1965; Kroetsch and Wang, 2008). These methods are not difficult to conduct and do not require expensive instrumentation, but require considerable time and labor, which discourages measurements on numerous samples. In most soil studies, particles size distribution measurements are limited to basic soil characterization. Several methods have been proposed to automate this measurement, including use of X-ray attenuation, electrical resistance, transmission electron microscopy, image analysis and light scattering, with considerable focus on commercially available instruments based on light scattering (Konert and Vandenberghe, 1997; Loizeau et al., 1994; McCave et al., 1986; Pieri et al., 2006; Segal et al., 2009). There were early concerns about the effectiveness of a laser diffraction instrument (Malvern 3600 E) for determining the particle size distributions of sediments and it was concluded that "the promise of an instrument that has both a wide spectrum and is precise in the silt range is not met", however, interest in this technology continued. Loizeau et al. (1994), who used two laser diffraction instruments (Coulter LS-100 and Malvern Laser Particle Sizer 2600), concluded that "reproducibility of the results on natural sediments appears to be satisfactory, but the method underestimated the fraction of clay particles with an efficiency of detection (36–70%) proportional to the clay content determined from pipette analysis". They concluded that the lack of correspondence between the laser and pipette methods was partly due to calculations of light scattering data output (i.e., Fraunhoffer versus Mie equations) and differences in units of measurement (volume for laser and weight for pipette). Konert and Vandenberghe (1997) also noted differences between laser and sieve measurements and concluded that these were

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of "methodological origin" with the "shape and sphericity" of the particles having an influence in the differences. Similarly, Buurman et al. (2001) concluded that the shape of the particles (e.g., platy versus spherical) influenced the correlation between laser instrument and pipette measurements such that correlations need to be specific for each type of material, but they noted that deriving these relationships would be "tedious". Zobeck (2004) and Pieri et al. (2006) also observed that measurements by laser diffraction and sedimentation did not always agree very well and suggested that the differences were due to methodology and the shape and characteristics (mineralogy, reflective index) of the particles. Arriaga et al. (2006) noted that "the light diffraction technique does not have a perfect agreement with the pipette method", and they recommended that measurements based on sieve-pipette methods were superior. Despite the evidence that there is usually a poor comparison of particle size distribution measurements by laser diffraction and sedimentation (seiving, pipette, hydrometer) methods, many studies concluded that laser diffraction could provide acceptable measurements but calibrations would be necessary (Buurman et al., 1997, 2001; Eshel et al., 2004; Muggler et al., 1997; Zobeck, 2004). All of these studies concluded that the calibration would not be universal and would have to be specific to factors such as profiles, toposequence, geological origin and mineralogy.

Commercial laser diffraction instruments are now readily available to offer potential automation of particle size distribution measurements of soils. However, since there are numerous reports that there were significant limitations, we decided to conduct a thorough evaluation on its effectiveness. During initial measurements on soil samples ranging from fine to very coarse texture, we observed that instrument output for coarse samples showed very little signal for sand-size particles. Further investigation and consultation with the instrument manufacturer showed that a much larger sample had to be introduced into the instrument to have sufficient numbers of these large particles to be detected, i.e., a threshold quantity. However, we surmised that there could be a potential to saturate the detector as the sample size introduced into the instrument was increased. This led us to examine the effect of sample size and the limitations of the instrument to measure materials that had a broad distribution of different proportions of various sizes of particles. We focused on particles that were smaller than sand size since sand is often determined by sieving before silts and clays are determined by sedimentation using either hydrometer or pipette. The objective was to determine the effectiveness and limitations of a laser diffraction instrument to measure distributions of a range of particle sizes, and to explore potential methods to calibrate the measurements.

2. Materials and methods

The particle size analysis instrument used in this study was Horiba LA-920. This instrument includes two light sources, a He-Ne laser (632.8 nm) coupled with a beam expander and a blue monochrome tungsten lamp (405 nm). The light from these two sources is directed on the sample in a detection cell where it is circulated with a high-output centrifugal pump to ensure consistent dispersion and flow of all of the particles. The instrument also has an ultrasonic probe that can be used to enhance dispersion of the sample in the detection cell. The laser light that is dispersed and scattered by the particles in the cell is focused through a condenser lens onto a specially designed forward ring silicon diode array with 75 separate detectors. The scattered light from the tungsten lamp is examined by six wide-angle silicon diode detectors that are arranged to sense the signal at angles up to 138° from forward direction. This configuration results in 87 simultaneous detections that are processed by Mie theory and equations to calculate the distribution of particle sizes from 0.02 to 2000 μm. The percentage transmission of the laser and tungsten lamps are also reported.

Five soil samples were obtained from previous projects that provided material that ranged from silty clay to sand particle size distributions according to measurements by a hydrometer method (Kroetsch and Wang, 2008) to test the response of laser instrument measurements when different sample weights were added. The distributions were classified according to the Canadian standards where clay, silt and sand ranged from 0–2, 2–50 and 50–2000 μm diameter, respectively. The air dried samples had previously been sieved through a 2 mm screen, thus did not contain particles larger than that.

Four commercially available samples of defined sizes were used, 3 μm silicon oxide particles (part #4806SF) from Nanostructural and Amorphous Materials Inc., Houston, Texas (www.nanoamor.com), and 38–45 μm (cat. #GP0042), 75–90 μm (cat. #GP0083) and 125–150 μm (cat #GP0138) glass particles from Whitehouse Scientific Ltd., Chester, UK (www.whitehousescientific.com). The 3 μm sample was considered to be 0–5 μm in this study. The study involved measurements on 625 combinations of 0, 0.05, 0.10, 0.15 and 0.20 g of each of the four particle sizes (total weights of the sample introduced into the instrument ranged from 0.05 to 0.80) plus another 35 measurements on selected combinations of these fractions where the total weight of the sample was always 0.80 g. In order to derive the expected output for the various combinations of each of the particle sizes, the output for each of the four size particle sizes measured individually was used to extrapolate what the signal from the combinations would be by mathematically combining output according to the proportion of the size fractions added. This method of calibrating the instrument ensured that any variability in the sizes of each fraction was taken into account by extrapolating actual measurements by the instrument used in the study.

The approximate number of particles that would be present in a given weight of the different diameter sizes of samples was calculated with the equation of Rosinski et al. (1956):

$$N/g = 1/0.5235d^3 \times \rho \times 10^{-12},$$

where N = number of particles per gram, d = particle median or geometric mean diameter in microns and ρ = particle density in grams per cubic centimeter. Particle density was assumed to be 2.5. For samples that had a known range in diameter (e.g., 125–150), the average diameter (e.g., 137.5) was used. Although these numbers were approximate, they were considered to be realistic and would reflect the relative differences between samples of different sizes.

All statistical analyses (regressions, etc.) were conducted with Statistix 9 (Analytical Software, Tallahassee, FL).

3. Results

Increasing the size of the sample added to the instrument changed the measurement of the distribution of clay, silt and sand fractions of all five soils that were examined (Fig. 1). With the silty clay, silty clay loam, silt loam and sandy loam soils, the proportion of clay increased with increased weight of sample, whereas the proportion of the sand tended to decrease. Since the proportion of each fraction changed with sample weight, the values differed variably from hydrometer measurements for each size fraction. Transmitted laser light also varied with the weight of the sample. For example, 0.05 g samples of silty clay, silty clay loam, silt loam, and sandy loam produced laser light transmissions of 65%, 84%, 94%, and 88% respectively. For 0.75 g samples of these soils it was 0.2%, 0.9%, 20%, and 7% respectively. In contrast to these four samples, the proportion of the sand fraction in the sandy soil increased with increased weight of sample. There was also considerably greater transmission of laser light with a 0.75 g sample of sandy soil compared with the other four soils that contained a greater proportion of fine particles. It is apparent