

A Simple Method for Estimating Particle Numbers Using a Laser Diffractometer

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Abstract

Hitherto the method of laser diffraction has been used only for determining particle size distribution (PSD) of mixtures. This paper presents an extension of that measurement approach to include the determination of the number of particles. The described method may find practical application in a variety of disciplines, including biology (e.g. for determination of population sizes of micro- and mesofauna), agricultural sciences (e.g. with relation to the study of soils), and Earth science (e.g. with relation to sediments and lands), among others.

Keywords: laser diffraction, particle size distribution (PSD), counting of particles in suspension, soil

Introduction

The geometrical characterization of particles can be described by shape, size, and particle size distribution (PSD), among others things [1-3]. The estimation of the PSD can be based on the calculation of the individual sizes of the particles, or can be measured independently [4, 5]. PSD is an important quantity that is widely used in different laboratories and industries, for instance in laboratory work associated with soil microbiology [6, 7], the food industry [8, 9], and agriculture sciences [10-13].

One of the modern methods of PSD determination is laser diffraction. This method is increasingly used in the research laboratories of glaciologists, geomorphologists, hydrologists, and geologists [14, 15], and published studies report that the method has been used for determining the grain size composition of soils [16-20]. The laser diffraction method is based on the measurement of a laser beam scattered at measured particles. The fewer particles to be measured, the bigger the angle of the scattered beam. The scattered light is registered on the detectors, and the software calculates particle size distribution (PSD).

Commercial utilization of the laser diffraction method for the determination of grain size composition in suspensions began in the second half of the last century. A number of manufacturers have become competitive in developing new apparatus that permit the determination of PSD with increasing accuracy and over a broadening range of particle sizes. The dynamic increase in available apparatus has made the method more popular, but at the same time it causes difficulties in comparing results obtained with apparatus of various generations and of different brands. This has led to the need for methodical research.

Analysis of the available literature shows that, to date, the laser diffraction method has been used solely for the determination of volume particle size distribution (in the sense of a cumulative distribution function, i.e. a curve defining the probability of finding a particle from a selected size range within the set of all particles) [20]. Knowledge of PSD, however, does not provide information about the number of discrete particles in a given mixture (information about the number of particles or cells is required by microbiologists, for instance). Even the recalculation of the volume PSD to number PSD does not solve the problem of counting the particles, but gives only the distribution; the total number of particles in the measuring system remains unknown. Moreover, the recalculation proce-

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ture may be biased and can introduce significant errors, since the more the shape of the measured particles differs from an ideal sphere, the bigger the error becomes.

The objective of this paper is to present a simple method for estimating the number of particles (simultaneously with PSD determination) using laser diffraction. This method is an extension of the possibilities of laser diffraction. The first part of the paper describes measurements on the model system (glass beads), followed by validation of the developed model for a real system. We used loess as an example, but any other particles can be used.

Materials and Methods

Three fractions of glass spheres were used in the first part of the study (Table 1, Fig. 1). For each fraction, calculation was made of the number beads that make up a combined weight of 1 gram (Table 1). The calculations were performed using a Morphologi G3 optical microscope (Malvern Instruments Ltd, UK) equipped with image analysis software and 987 \times magnification. The pho-

Table 1. Properties of glass bead fractions and loess used in the study.

Fraction number	Fraction size as declared by manufacturer (mm)	Number of beads/loess particles with combined weight of 1 g
1	212-300	54,530
2	425-600	5,520
3	710-1180	856
loess	50-100	3.01 \cdot 10 ⁶

tographed and counted spheres for every fraction were weighed on a balance (Radwag, Poland) with an accuracy of $\pm 10^{-4}$ g. This procedure permitted, in the course of further study, determination of the number of spheres via the determination of weight.

The PSD for each glass beads fraction was measured individually (Fig. 2). For practical purposes these distributions do not overlap. To check the possibility of counting

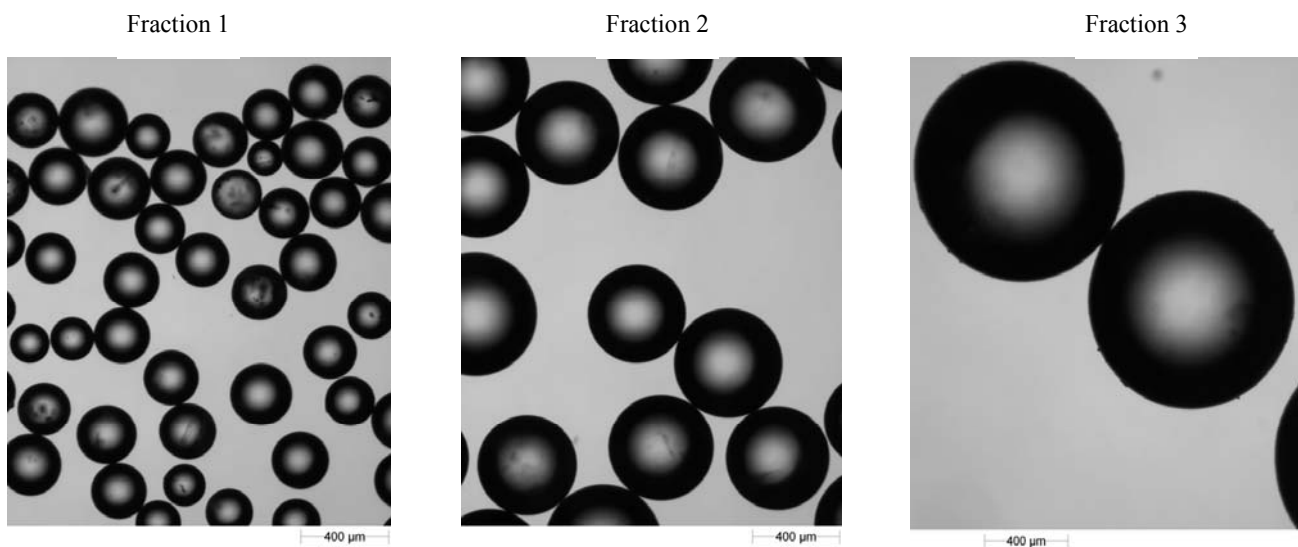


Fig. 1. Images of glass beads from the fractions under study.

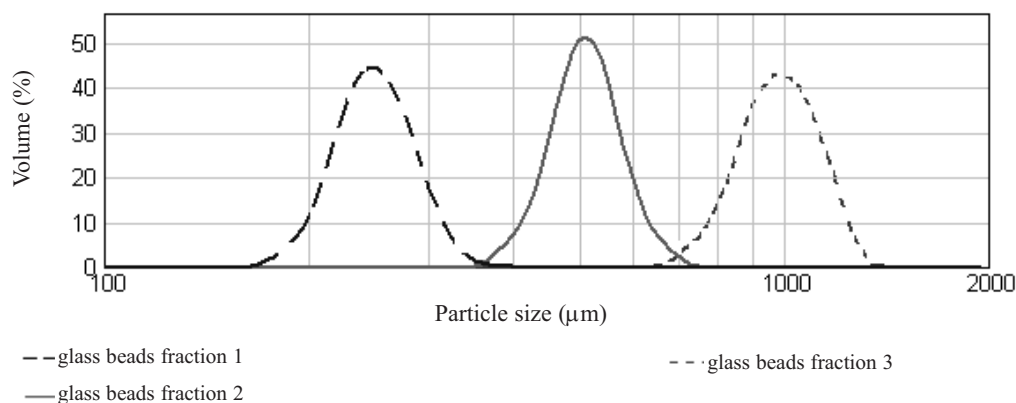


Fig. 2. The PSD of individual fractions of glass beads. The distributions were obtained for each fraction separately and then plotted on a common graph. The graph represents distributions obtained from calculations made according to the Mie Theory (MT), algorithm SM, and algorithm SSR.

particles using a laser diffractometer the following mixtures were measured: fractions 1 and 2, 1 and 3, and 2 and 3, all mixed 1:1 by weight; in addition, the mixtures of fractions 1 and 3 (because of their large size difference) also were measured in weight proportions 1:3 and 3:1.

The sieved fraction (50-100 μm) of loess was used for the validation of the method. The PSD of the loess is shown in Fig. 3. The loess was taken from Czeslawice (Lublin Upland, SE Poland) at a depth of 1.5 m (bedrock). Loess is relatively homogenous and its particle shape and size are relatively reproducible. The number of the loess particles in 1 gram was counted using the same procedure as for the glass beads (Table 1).

Two different quantities of glass beads were each added to two loess samples (these loess samples had the same mass – Table 3). The amounts of loess and glass beads were chosen on the basis of the height of the peaks of preliminary measurements of the mixtures using laser diffraction.

All measurements of PSD were performed using the Malvern Mastersizer 2000 apparatus (measurement range 0.02-2000 μm). Because the size of a measured particle was not too big, a Hydro MU dispersion unit was used to make the measurements [21] in a suspension of approximately 800 mL volume of distilled water, at 2,500 rpm stirrer speed to avoid the formation of air bubbles in the vortex. The apparatus operates at two laser light wavelengths: red (633

nm) and blue (466 nm). Individual measurements, taken as the determination of a single PSD, were conducted for 30 s, during which time the apparatus recorded 30,000 ‘snapshots’ of diffraction images and determined a single distribution from them. The adjustment was selected on the basis of previous experience [22]. The measurements were repeated 10 times.

In the course of measurement, the Mastersizer 2000 apparatus registers source data, in this case the intensity of light falling on sensors installed in the apparatus. This permits subsequent conversion of these data to PSD using Fraunhofer theory and/or the Mie Theory, and different algorithms. The Fraunhofer Theory is recommended for particles larger than 50 μm . The Mie Theory is better for smaller particles; however, definition of optical properties (refractive and absorption indices) is necessary in association with the Mie Theory [23].

For calculations the Mie Theory was chosen; the following index values were applied for both glass and for loess: refractive index = 1.52 and absorption index = 0.1. The values of optical indices for glass were chosen from the Mastersizer 2000 software. The values for loess were chosen on the basis of previous experiments [22].

The software supplied with the Mastersizer 2000 apparatus permits calculation of source data obtained on the sensors using various algorithms (irrespective of the prior

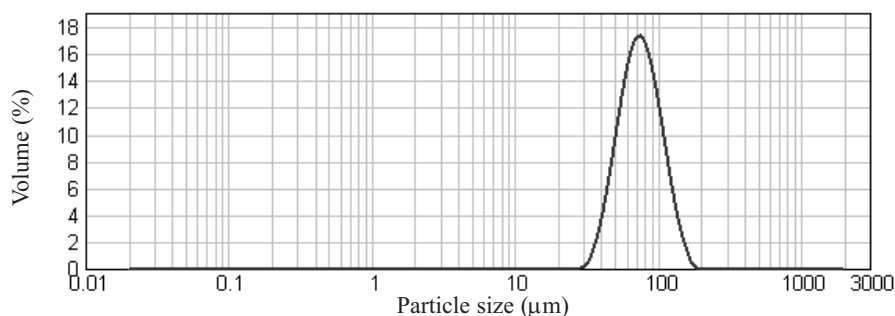


Fig. 3. PSD of loess material. The graph represents distributions obtained from calculations made according to the Mie Theory, algorithm SM, and algorithm SSR.

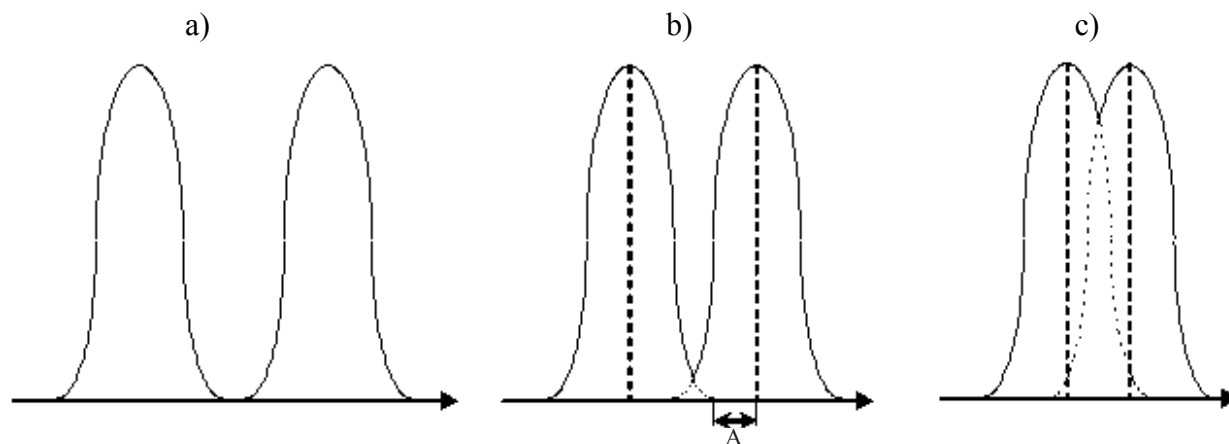


Fig. 4. Conditions for the application of the method of standard addition for determining the number of particles with the use of laser diffraction: a) Optimal situation – distributions are discrete (do not overlap); b) Conditional situation – assuming symmetry of distributions, the distributions overlap to an extent such that A is greater than zero; c) The error in calculating the number of particles in the case of such large distribution overlap is very high, making the utilization of such a case impossible in practice.

choice of optical theory). The choice of algorithm depends on the properties of the objects studied and on the objective of the research. Taking into account the geometrical properties of the measured particles, the multi-narrow modes (the model appropriate for samples consisting of one or more very narrow modes) and the spherical shape mode (the model appropriate for samples consisting of spherical particles) were used for measurements.

Results and Discussion

Condition for the Application of Method of Standard Addition

Because our focus here is to propose a method of estimating the number of particles using a laser diffractometer, we do not intend at this point to discuss other methodological problems that may need to be solved to obtain reliable results; some of these have been described in our previous paper [22].

The number of particles in a mixture was calculated using the method of standard addition. The method in this case consisted of adding a known number of glass spheres (called beads from here on) having diameters significantly different from the size of the spheres to be measured (later called particles). In practice, the PSDs of the particles to be measured and of the added beads should be discrete or only slightly overlapping (Fig. 4).

Determination of the Equivalent Diameter

The diameters both of beads and particles must be known for calculating the number of particles by the laser diffraction method. Because it is not possible to find truly monodisperse fractions (i.e. the peaks of PSD always have non-zero widths), the diameter corresponding to the maximum of the peak will be called the equivalent diameter (the equivalent diameter is the abbreviation because it should be called sphere equivalent mode diameter).

Mastersizer 2000 software does not allow the value of the peak maximum to be read. When the cursor of the mouse is placed at the top of the peak, information about the range and volume of the fraction appears. For instance, a measurement reading: "25.04% between 216.8 mm – 294.5 mm" means that the particles in the specified size range occupy 25.04% of the total volume. The equivalent diameter of the peak maximum is calculated as the arithmetic mean of the range limits (in the above example, $[216.8 \text{ mm} + 294.5 \text{ mm}]/2 = 255.65 \text{ mm}$). Therefore, Table 2 shows the arithmetic means for the range.

Calculation Method of the Number of Particles

The volume of a single equivalent bead of the known fraction, V_1 (adopted as the internal standard), was calculated from:

$$V_1 = \frac{1}{6} \pi d_1^3 \quad (1)$$

...where d_1 is the equivalent diameter calculated as the arithmetic mean of the range determined by the software at the maximum of the peak for the known fraction (added beads).

The volume of the known fraction (added beads), $V_{\text{Known_fraction}}$ was calculated as:

$$V_{\text{Known_fraction}} = n_1 V_1 \quad (2)$$

...where n_1 is the number of beads of the known fraction.

The share of both known and unknown fractions in the mix were read from the software, and the volume of the fraction studied was calculated from the ratio:

$$V_{\text{Unknown_fraction}} = \frac{a}{b} V_{\text{Known_fraction}} \quad (3)$$

...where: a is the percentage of volume occupied by the studied fraction (measured particles), read from the software, b is the percentage of volume occupied by the known fraction (added beads), read from the software.

Taking into account that the volume $V_{\text{Unknown_fraction}}$ calculated from eq. (3) can be written as:

$$V_{\text{Unknown_fraction}} = n_2 V_2 \quad (4)$$

...where: n_2 is the number of particles on the studied fraction, given by:

$$n_2 = \frac{V_{\text{Unknown_fraction}}}{V_2} \quad (5)$$

V_2 is the volume of a single equivalent particle of the studied fraction, given by:

$$V_2 = \frac{1}{6} \pi d_2^3 \quad (6)$$

Testing the Method on the Glass Beads

Comparison of the results of numbers of particles calculated with the readout of particle diameter from the middle of the range displayed on the graph obtained for the mixtures is presented in Table 2.

The general conclusion to be drawn from this stage of investigations is that the method works. The conditions are: selection of the optimal fractions (narrow distributions with little or no overlap) and optimal proportions of both fractions. The best results were obtained for fractions 1 and 3 when the amount of smaller fraction was bigger (3 times by weight) than the coarser one (line 4 in Table 2). The fact that laser diffraction gives the volume distribution can be an explanation of this result. One big particle takes up a much

Table 2. Results of calculation of the number of particles in the studied fraction and relative error referenced to the number of particles determined by weight.

Number of line	Fraction numbers in mixture	Equivalent diameter (mm)	Ratio of fraction weights in the mixture	Number of beads of the known fraction	Calculated fraction	Number of particles on the studied fraction	Standard deviation	Relative error %
1	1	249.25	1:1	382,250	1	384,073	29,368	0.47
	3	998.40						
2	1	249.25	1:1	442,320	1	474,609	6,623	7.30
	2	558.4						
3	2	558.4	1:1	60,940	2	61,199	7,898	0.42
	3	998.40						
4	1	249.25	3:1	409,640	1	409,127	44,656	-0.13
	3	998.40						
5	1	249.25	1:3	136,470	1	146,679	5,790	7.48
	3	998.40						

Table 3. Results of calculation of number of loess particles and relative error referenced to the number of particles determined by weight. The glass beads (fraction 212-300) were used as the internal standard.

	Equivalent diameter (mm)	Mass (g)	Number of beads of the known fraction	Calculated fraction	Number of particles on the studied fraction	Standard deviation	Relative error %
Loess	78.09	1.7166	5.17·10 ⁶	loess	5.01·10 ⁶	0.20·10 ⁶	-3.09
Glass beads	249.25	0.5302					
Loess	78.09	1.7166	5.17·10 ⁶	loess	5.09·10 ⁶	0.15·10 ⁶	-1.55
Glass beads	249.25	1.1746					

greater volume than many small particles. Increasing the number of smaller particles evens out the proportions. Relatively larger errors occur in systems containing a small number of smaller particles (line 5 in Table 2 confirms this explanation). It is difficult to explain the reason for the high value of relative error in the mixture of fractions 1 and 2 (line 2, Table 2).

Validation of the Method on the Loess Particles

The results obtained for loess particles are presented in Table 3. The relative error at the level of a few percent is quite good; note that this error can be minimized by selecting the best ratio between measured particle and internal standard.

The above results should be treated as only one possible mode for using the described method. The laser diffraction method for counting the measured particles can be used in many applications: for example, in biology for estimating the number of algae or protozoa, or in different industries for estimating the numbers of particles of interest to them.

Conclusions

1. A method has been developed for estimating the number of particles in suspension using laser diffraction, by adding an internal standard (a known number of particles of specific size) to the sample under study.
2. The presented method for estimating the numbers of particles and their size distribution may find practical applications in numerous fields, such as biology (e.g. for determination of population sizes of micro- and meso-fauna), agricultural sciences (e.g. with relation to study of soils), Earth science (e.g. with relation to sediments and lands) and in different industries. In most such applications, an error of measurement of the number of particles of the order of several percent is acceptable (up to 10%), especially considering the speed, low cost, and ease of carrying out the measurement procedure.

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