Determination of soil texture: Comparison of the sedimentation method and the laser-diffraction analysis

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Abstract
Laser-diffraction analysis (LDA) is a rapid automated method achieving highly resolved frequency distributions of particle sizes. Recently, LDA has come into use in environmental sciences. However, in the size range of silt and clay deviations from the particle-size analysis with the standard pipette method, which is regarded as the reference method for soil-texture classification, have been reported. Therefore, this study concentrates (1) on the verification of systematic relations between both methods using a series of soils of Lower Saxony (Germany) and (2) on the general applicability of the laser-diffraction method to soil-texture classification as well as (3) texture-based estimates of air capacity, available field capacity, and permanent wilting point. The comparison of LDA with the pipette method demonstrated highly significant linear correlations in each of the particle-size fractions from clay to coarse silt. The slope of regressions ranged from 0.4 with fine silt to 3.1 with clay. If the clay content derived from LDA was applied to texture classification, the resulting textural classes differed from the standard textural classes, except for purely sandy samples with a clay content of <5%. However, the linear-regression model enabled an approach of the LDA-based clay content to values produced with the standard pipette method. Using this transformation, a texture classification became practicable in many cases, but, despite of a high significance level between LDA and pipette method, still led to wrong textural classes in several cases. A comparison with regression models from other regions in Europe showed both similarities and discrepancies, even for similar substrates. Hence, the laser-diffraction analysis cannot be used for the texture classification of soil samples without verification by the standard pipette method.

Key words: clay content / pipette method / texture classification / particle-size fractions

Accepted October 24, 2008

1 Introduction

Soil texture is a fundamental soil characteristic closely linked to more complex soil properties such as the main characteristics of the soil water supply, the cation-exchange capacity, and the hydraulic conductivity. As a consequence, empirical relations could be established to predicting these complex properties from soil texture. There are various international and national systems of determination and nomenclature in use. In this study, the system in Germany as defined in the German Soil Survey Manual (Ad-hoc-AG Boden, 2005) with four major texture groups ("Bodenarten-Hauptgruppen") and 31 soil-textural classes ("Bodenarten") was applied.

The standard procedure in soil science to analyze the particle-size distribution is a combination of sieving and sedimentation techniques, the latter mainly performed with an areometer or a pipette apparatus (Smith and Mullins, 1991). The sieve-pipette method is internationally applied as commonly used standard (Gee and Bauder, 1986; DIN ISO 11277, 2002). On the base of the relative mass of clay, silt, and sand fractions obtained with this method, the soil texture can be classified systematically using an appropriate nomenclature.

With the development of modern automatically measuring instrumentation for grain-size analysis (e.g., SediGraph, Coulter Counter), several attempts were made to apply these new techniques which are less complex and less time-consuming than the traditional method in soil science (Singer et al., 1988; Syvitski, 1991). Furthermore, new techniques such as the laser-diffraction analysis (LDA) offer the determination of a highly resolved frequency distribution of particle sizes (Syvitski, 1991) with no additional operating cycles.

Detailed grain-size analysis can be of advantage for soil-genetic studies or investigations of particle redistributions (Muggler et al., 1997). Buurman et al. (2004) used laser diffractometry to analyze the stratigraphy of European volcanic soils. Westerhof et al. (1999) studied the formation of micro-aggregates under diverse soil-management systems with LDA. However, it was shown that the results of the laser-diffraction method often differed considerably from those analyzed with the pipette method, especially within the fractions of clay and fine silt, implying that the grain-size distribution evaluated with the LDA cannot be accepted without criticism. Hence, problems associated with the use of LDA measurements specifying the soil-textural class which is based on the sedimentation method are to be expected. Syvitski (1991) and Allen (1997) have also shown that the different physical
 basics of the two approaches, sedimentation and laser diffractometry, imply diverse results.

1.1 Sedimentation method

The sedimentation method is based on the Stokes’ equation which describes the settling velocity of particles in a suspension as a function of the effective particle radius, particle density, liquid density, and dynamic viscosity of the liquid. This equation requires the following postulations (Smith and Mullins, 1991; Allen, 1997):

– particles are of spherical shape,

– the velocity is constant with laminar flow (Reynolds’ number <1),

– no interaction occurs between particles and particle and vessel wall,

– no interaction occurs between particles and the liquid.

Stokes’ equation is applicable for a particle-size range that is limited by the Reynolds’ number at the upper boundary and by the Brownian motion at the lower boundary (approx. <1 μm), following the concept of equivalent diameter for nonspherical particles (Smith and Mullins, 1991). The sedimentation method is generally used to separate particles by equivalent diameters in fractions such as silt and clay. Corresponding to a selected diameter, an aliquot of the settling sample suspension is taken from a given depth at a given time and used for calculations on the mass base.

1.2 Laser-diffraction analysis

The laser-diffraction analysis (LDA) is based on the diffraction of a laser beam when it strikes a particle, whereby the diffraction angle increases with decreasing particle size. The intensity distribution of the scattered laser beam in several directions behind the particles in a sample cell can be detected with concentric arranged sensors, each assigned to a special particle-size range. The corresponding relative particle-size distribution in the suspension is calculated on volume base.

The LDA method implies the following limitations and conditions (Syvitski, 1991):

– calculation is based on spherical shape,

– Fraunhofer theory or Mie theory is used,

– very small particles produce very high diffraction angles which could exceed the detection area of the sensors,

– particles are passing the laser beam several times to produce averaged two-dimensional particle diameters in the cross-sectional area.

The Mie theory describes the radiation in and around a homogeneous particle in a homogeneous medium that does not absorb light, taking into account the processes of diffraction, refraction, and absorption which all contribute to the scattering pattern. The particle itself can be transparent or totally absorbing, whereby its optical properties (refractive index) are part of the calculation. The Fraunhofer theory is an approximation for nontransparent particles with diameters above the wavelength used (in general \( \varnothing > 10 \lambda \)) independent of the optical parameters when diffraction becomes dominating (Allen, 1997). For particle sizes below the laser wavelength, the full Mie theory has to be applied.

Several articles have been published dealing with methodical research concerning particle-size analysis and laser diffractometry in general (Wu et al., 1993; Bowen et al., 2002; Dur et al., 2004), as well as addressing technical-based uncertainties (McCave et al., 1986) to high measuring reproducibility with further developed LDA techniques (Loizeau et al., 1994).

1.3 Laser-diffraction analysis and soil texture

Environmental and soil-science investigations comparing sedimentation with laser techniques demonstrated that diverging results between sedimentation and LDA methods increased with decreasing particle size, consequently mainly in the clay fraction (Beuselinck et al., 1998; Buurman et al., 2001; Eshel et al., 2004). Konert and Vandenberghe (1997) presented a study comparing sieve and pipette methods with the LDA method based on a variety of sediments in The Netherlands and certified reference materials. Sand fractions of sediments showed only slightly differences between the two methods, whereas the clay fraction was clearly underestimated by laser diffraction. They concluded that the pipette fraction <2 μm was corresponding with the laser fraction <8 μm. As the main reason for this divergence, the deviations from spherical shape was given, but deviations in the particle density from the quartz density that is assumed in the sedimentation method should also be considered. In samples consisting of quartz, only the discrepancies between the two methods can be neglected over the whole size range (Beuselinck et al., 1998; Pabst et al., 2000). However, regarding the particle-size distribution with respect to soil texture, no information is given in literature about the applicability of LDA for soil-texture classification.

The aim of this study was to verify systematic relations between the LDA and sedimentation method for representative soils of the regional zone in N Germany covering the range of frequent substrates with varying clay and sand contents. Furthermore, it should be validated whether or not results achieved with LDA could be used in conjunction with the German classification system (Ad-hoc-AG Boden, 2005) to determine soil texture. The characteristics of soil water supply will be included as an example of texture-related complex properties. A comparison between the combined standard sieve-pipette method (SM based on sedimentation) and a combined sieve-laser method (LM based on LDA) was realized with 16 soil samples from Lower Saxony.
2 Material and methods

2.1 Material

The selected soil samples of different textural classes cover a wide range of clay percentage and are largely representative for the lowland region of Lower Saxony. Sixteen soil samples were taken from soils which developed on quaternary sediments such as loess loams, glacial fluvial deposits, river sediments, and clay layers. In accordance with DIN ISO 11277 (1994), all samples were air-dried, carefully crushed, homogenized, and passed through a 2 mm sieve to gain fine-earth material.

Simultaneously, the water content of air-dried soil was determined by oven drying at 105°C for 24 h. The OM content was determined by oven drying at 105°C for 24 h. The OM content was calculated on the base of the total C content, given by a CNS Analyzer (Euro EA, Hekatech). Both values were used to convert the air-dry mass of the sample to the absolute dry mass of humus-free mineral soil.

2.2 Sample pretreatment

All carbon-free soil samples were pretreated in the same way prior to the grain-size analysis. An amount of 10 g air-dried fine-earth was prewetted with water in a 1000 mL Duran glass bottle. A volume of 30 mL of H2O2 was added to remove organic matter (OM) in an overnight reaction at room temperature followed by 2 h at 105°C. The remaining soil was dispersed with 25 mL 0.05 M Tetrasodium Diphosphate and shaken for 1 h in aqueous suspension. Three replicates were prepared for the sedimentation method as well as for the laser method.

Table 1: Four particle-size fractions of silt and clay, based on soil free of OM and water.

<table>
<thead>
<tr>
<th>Size (μm)</th>
<th>Fraction</th>
<th>Abbreviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>20–63</td>
<td>coarse silt</td>
<td>gU</td>
</tr>
<tr>
<td>6.3–20</td>
<td>medium silt</td>
<td>mU</td>
</tr>
<tr>
<td>2–6.3</td>
<td>fine silt</td>
<td>fU</td>
</tr>
<tr>
<td>&lt;2</td>
<td>clay</td>
<td>T</td>
</tr>
</tbody>
</table>

2.3 Sieving and preparing of soil suspension

Using three sieves with the mesh sizes of 630, 200, and 63 μm, the wet sieving of the sand fractions (63–200; 200–630; 630–2000 μm) was carried out before the sedimentation and LDA analysis. The collected fine-sand, medium-sand, and coarse-sand fractions were dried at 105°C and related to dry mass of humus-free mineral soil. The suspended fine material (<63 μm) was collected in a 1000 mL Duran glass bottle and diluted to 850 mL with deionized water at room temperature. Sieving prior to suspension analysis was not only in accordance with the DIN standard (DIN ISO 11277, 2002), but allowed a better handling of the laser analysis (cf., section 2.5).

2.4 Sedimentation analysis

The 850 mL suspension (section 2.3) served for the sedimentation analysis (<63 μm) which was performed with a modified pipette apparatus according to Köhn (1928) and the DIN standard (DIN ISO 11277, 2002). Calculation of the settlement velocity was accomplished by postulating an average particle density of 2.65 g cm–3 for the Stokes’ equation. Temperature-dependent values of density and dynamic viscosity of pure water were applied.

Each glass bottle containing the soil suspension was first agitated by hand to produce a homogeneous suspension which was then set onto a pipette table for sedimentation. After the corresponding sedimentation time, an aliquot of the suspension was taken with a pipette, having three horizontally arranged (120°) openings at the tip, at a given depth and rinsed into a glass beaker of 100 mL.

Aliquots (approx. 18 mL) of soil suspension <63 μm, <20 μm, <6.3 μm, and <2 μm were collected successively in this way and subsequently dried at 105°C. According to the German classification, four particle-size fractions, based on soil free of OM and water (section 2.2), were derived from this procedure (Tab. 1).

2.5 Laser-diffraction analysis of particle sizes

Laser-diffraction analysis was realized by wet dispersion in deionized water with a laser particle sizer (analysette 22, Fritsch). This equipment was operating with a convergent laser beam of λ = 638 nm hitting the sample measuring cell at two defined distances from the detector unit with 31 sensors. Consequently, a measurement range of 0.290–295 μm was adjusted. The results of light-intensity distributions were calculated as percentages of 62 particle-size fractions in exponentially increasing intervals, summed up to 100%. Since the Mie theory would have required detailed but unknown information about the optical properties of particles, the Fraunhofer theory was applied in the calculations by an empirical approach. Scattered light intensity which could not be detected by the sensor system, i.e., large angles caused by very small particles, was calculated based on an energy balance before and during measurement and added to the smallest-size interval (Mutter and v. Bernuth, 2001).

To conduct a measurement procedure, an aliquot of 20–40 mL of the homogenized soil suspension (section 2.3) was flushed into the sample chamber of the particle sizer, adjusting the turbidity in the measurement cell to the optimum detection range. In this context, sieving prior to laser detection was of advantage as there was no coarse material in the suspension left which could complicate the filling of a homogeneous aliquot and, furthermore, could obscure smaller particles from detection.

The relative amounts of the LDA particle-size fractions <2 μm, 2–6.3 μm, 6.3–20 μm, and 20–63 μm of the suspension were converted into relative amounts of dry and humus-free soil including the total mass of the sand fraction. In some samples, particles >63 μm were detected in the suspension.
To contribute to texture classification, those particles (63–80 $\mu$m) were added to the coarse-silt fraction holding onto the 63 $\mu$m sieve as reference.

2.6 Statistical analysis

The Kolmogorov-Smirnov test, modified by Kuiper, was used to control the normal distribution. With the paired t-test, the significance of the differences between sedimentation and laser method in the particle-size fractions (T, fU, mU, and gU) was proved, while the linear-regression analysis revealed the relation between size fractions derived from sedimentation and laser diffraction (Lozan and Kausch, 1998). Root mean squared error of estimate (RMSE) was calculated as a measure of prediction error.

2.7 Determination of soil texture

According to the German Soil Survey Manual (Ad-hoc-AG Boden, 2005), soil texture is based on the relations of clay (<2 $\mu$m), silt (2–63 $\mu$m), and sand (63–2000 $\mu$m). While in the standard sieve-pipette method, the clay content and the sum of the silt fractions (cf., section 2.4) were used for texture classification, in the sieve-laser method, clay and sand formed the basis for classification with silt as a completion to 100%. In consequence of the different methodological and statistical approaches, for the clay content three different kinds of texture were generated (Tab. 2).

2.8 Control of particle size with scanning electron microscopy

The clay-fraction suspension of a loess-loam sample (U01) collected during the sedimentation analysis was diluted to 1:20, dehydrated with a graded series of acetone (70, 90, and two changes of 100% v/v), coated with a gold layer, and subsequently analyzed with a scanning electron microscope (SEM) (Stereoscan 250, Cambridge) in combination with a digital image analysis (AnalySIS 3.2, Soft Imaging).

3 Results and discussion

3.1 Detection of size fractions

Table 3 provides the results of single particle-size fractions, the derived textural classes, and major groups of texture comparing the standard sieve-pipette method with the sieve-laser method. The variability coefficient of pipette and laser analysis was <7% in the pipette fractions and <3% in the laser fractions. For each particle-size fraction, normality can be assumed at a significance level $\alpha = 0.1$ based on the Kolmogorov-Smirnov-test.

Table 2: Methodological approaches to determine the clay content and soil texture.

<table>
<thead>
<tr>
<th>Method</th>
<th>Determination of clay</th>
<th>Texture</th>
</tr>
</thead>
</table>
| Standard sieve-pipette method (SM) | pipette analysis  
                             (standard clay content) | standard texture        |
| Sieve-laser method (LM)  | laser analysis  
                             (LDA clay content)      | LDA texture              |
| Sieve-laser method (cLM) | corrected laser analysis  
                             (LDA clay content is converted by linear regression) | corrected LDA texture |

It is shown in Tab. 3 that there was no difference detectable in the total sand content between the two method combinations. This is due to the uniformly used sieving procedure. Furthermore, it has been reported that sieving and laser-diffraction analysis led to nearly same values in the sand fraction of milled quartz as well as silty soil samples (Beuselinck et al., 1998). In this study, sand was a complementing feature for the textural classification of the soil.

While only negligible differences between the two methodological approaches in the coarse-silt fraction were detected, clay and the remaining silt fractions showed increasing differences associated with decreasing particle size (Tab. 3). Regarding all samples as one population, the paired t-test showed that the results of laser and pipette method significantly ($\alpha = 0.05$) differed in the fractions clay, fine silt, and medium silt, whereas the differences in the coarse-silt fractions were insignificant. The relation between laser and sedimentation analysis was a highly significant ($\alpha = 0.01$) linear regression in each size fraction (Fig. 1).

The coarse-silt fraction is of particular interest because it builds up a methodological intersection where the total sieving results for sand (>63 $\mu$m) were combined with the results of the suspensions. In the sieve-pipette method, both parts are mass-based, while the laser method is based on volume, and the relative LDA particle distribution had to be converted arithmetically into mass percentage. As presented in Fig. 1, a slope close to 1.0 for coarse silt is confirming the congruent results of both methods pipette and LDA, although the LDA included larger particles in some samples. In those cases, the LDA is an indicator of particle-shape effects on the sieving procedure (Allen, 1997). The restriction of the comparison to the upper limit of 63 $\mu$m in the LDA fractions improved the linear regression to: $y = 0.987x + 1.316$ ($R^2 = 0.940$). With respect to texture classification based upon sieving, the combining procedure could be neglected as a possible source of error.

For the clay fraction (T), the pipette method generated results approx. 3 times higher than the laser method, indicated by a slope of 3.09. A comparable effect was reported by Konert and Vandenbergh et al. (1997) for sediments in The Netherlands.
Table 3: Texture analysis for selected soil samples of Lower Saxony (N Germany) with different contents of organic matter (OM). Particle-size fractions of clay (T), fine silt (fU), medium silt (mU), coarse silt (gU), and sand (S) were determined by two methods: (1) standard sieve-pipette method and (2) sieve-laser method. Resulting textural classes and major texture groups (Ad-hoc-AG Boden, 2005) are labeled as “standard” and “LDA”.

<table>
<thead>
<tr>
<th>Sample characteristics</th>
<th>Sieve-pipette method</th>
<th>Standard texture</th>
<th>Sieve-laser method</th>
<th>LDA texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>no. code</td>
<td>substrate</td>
<td>OM (%)</td>
<td>T (%)</td>
<td>fU (%)</td>
</tr>
<tr>
<td>1 T01</td>
<td>clay</td>
<td>0.4</td>
<td>61</td>
<td>16</td>
</tr>
<tr>
<td>2 T02</td>
<td>clay</td>
<td>1.3</td>
<td>54</td>
<td>5</td>
</tr>
<tr>
<td>3 L01</td>
<td>fluvial sediment</td>
<td>2.0</td>
<td>25</td>
<td>9</td>
</tr>
<tr>
<td>4 L02</td>
<td>fluvial sediment</td>
<td>2.6</td>
<td>21</td>
<td>8</td>
</tr>
<tr>
<td>5 L03</td>
<td>glacial loam</td>
<td>0.1</td>
<td>17</td>
<td>5</td>
</tr>
<tr>
<td>6 L04</td>
<td>fluvial sediment</td>
<td>0.4</td>
<td>17</td>
<td>3</td>
</tr>
<tr>
<td>7 L05</td>
<td>fluvial sediment</td>
<td>0.8</td>
<td>26</td>
<td>10</td>
</tr>
<tr>
<td>8 U01</td>
<td>loess loam</td>
<td>0.4</td>
<td>21</td>
<td>3</td>
</tr>
<tr>
<td>9 U02</td>
<td>loess loam</td>
<td>3.0</td>
<td>18</td>
<td>4</td>
</tr>
<tr>
<td>10 U03</td>
<td>loess loam</td>
<td>4.6</td>
<td>13</td>
<td>3</td>
</tr>
<tr>
<td>11 U04</td>
<td>loess loam</td>
<td>0.8</td>
<td>12</td>
<td>5</td>
</tr>
<tr>
<td>12 S01</td>
<td>glacial sand</td>
<td>1.2</td>
<td>11</td>
<td>4</td>
</tr>
<tr>
<td>13 S02</td>
<td>glacial sand</td>
<td>0.9</td>
<td>9</td>
<td>3</td>
</tr>
<tr>
<td>14 S03</td>
<td>glacial sand</td>
<td>0.7</td>
<td>8</td>
<td>2</td>
</tr>
<tr>
<td>15 S04</td>
<td>glacial sand</td>
<td>0.1</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>16 S05</td>
<td>fluvial sand</td>
<td>0.2</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>
with a similar regression relation between pipette and laser fraction and classified as underestimation of the clay fraction.

In the medium- and fine-silt fraction slopes distinctly <1.0 give evidence that the laser method gained systematically higher values compared to the pipette method (Fig. 1). This was more pronounced in the fine-silt fraction than in the medium-silt fraction with slopes of 0.44 and 0.64, respectively.

3.1.1 Particle shape

Both, the sedimentation equation and the matrices solution to convert diffraction angles into particle sizes, are based strictly on spherical particles. Soil particles, however, show various other shapes from nearly spherical to platy. The laser-diffraction principle projects a mean particle diameter averaged out of different axes of view. Consequently, a platy particle is described by a cross-sectional area being larger than that of a sphere of equal volume (Jonasz, 1987). Concerning the sedimentation principle, platy or other irregular particles reach lower settling velocities than spheres of comparable volume. In this case, the particle diameter will be underestimated (Syvitski, 1991). Thus, the effect of shape is working in opposite direction in LDA and sedimentation method.

3.1.2 Particle mineralogy

Particle density of soil minerals is affecting the sedimentation method only, by means of the settling velocity from Stokes’ equation. A lower density makes a particle slower, a higher density makes it faster. However, the mineral composition of soil samples is variable over the whole range of particle sizes. Since no detailed information is available, the density of quartz which also represents an average of the common clay minerals in many soils (Jasmund and Lagaly, 1993) is used as a substitute.

In the LDA, the particle mineralogy is adopted as refractive index (RI) in the Mie theory, where it has a strong influence on the calculated size distribution (Eshel et al., 2004; Sperazza et al., 2004). Eshel et al. (2004) proposed a value of 1.53 as approximation to most soil minerals. However, especially the detected clay content may distinctly differ by varying RI values of 1.5 to 1.6. Thus, the correct setting of optical properties is substantial, but difficult to realize in routine analysis. It was shown by experience that the Fraunhofer theory could be extended to the clay-size fraction with acceptable precision (Konert and Vandenbergh, 1997).

3.1.3 Size limit of clay fraction

Considering the influence of shape on sedimentation, it is expected that a clay pipette fraction will contain also larger irregularly shaped particles. Investigations on kaolin by Pabst et al. (2000) using SEM analysis indicated that the clay fraction gained by pipette method included particles >2 μm. Konert and Vandenbergh (1997) also detected particles >2 μm by SEM in their study on sediments. In addition, Eshel et al. (2004) detected in three differing soil samples with the laser method that approx. 42% of the pipette-taken clay...
fraction was >2 µm. However, their samples included OM and, therefore, their results cannot be compared quantitatively to standard humus-free mineral soil samples.

In this study, the clay suspension of the sample U01 (Haplu-dalf subsoil) obtained with the sedimentation method was investigated with SEM and image analysis (Fig. 2). Out of 12 SEM micrographs, the diameters of the four biggest clay particles were measured. A maximum of approx. 2.1 µm was found along the crystallographic a- and b-axis within the clay-fraction suspensions.

The micrographs showed single clay particles and the predominance of particles of the fine-clay fraction. In addition, there was a tendency towards a globular shape of the smaller particles such as fine clay, whilst the coarse clay displayed the typical platy shape of phyllosilicates. This is due to the changing ratio of the crystallographic axis. Compared to the coarse clay, the a- and b-axis is reduced within the fine clay while the c-axis of the packed minerals remains at 80–200 nm. In contrast to the literature, these results indicate for a loess sample that the clay fraction extracted with the pipette method is reliable in terms of particle size. Therefore, the SEM gives evidence that shape as an influence factor is indeed well-known, but its extent still could and should be investigated in future.

3.1.4 Clay subfractions

An additional source of error is caused by clay subfractions such as fine clay. The influence of this fraction is not well known until now and, although often dominating the clay fraction, it is not detectable directly by laser diffraction. The resolution of laser diffractometry reaches its lower limit when nanoparticles are concerned, which are calculated by an energy balance of the laser beam instead of detection with the sensor unit.

In contrast to laser diffraction, particles of equivalent diameters of <2 µm are always collected in total using the pipette method (Agrawal et al., 1991). Schwertmann (1961) stated that approx. 60% (±15%) of the clay fraction was found to be <0.2 µm in a study with ten differing soil samples. Consequently, a considerable part of the clay fraction is not covered by the laser diffractometry with a lower range limit of 0.3 µm in standard equipment or 0.1–0.02 µm in modern analyzer instruments of various manufacturers.

Information about the detailed detection of the clay fraction was given by Dur et al. (2004) who compared laser detection of the clay fraction of a fine loamy soil with transmission—electron microscopy image analysis. In the size range of 0.04–0.8 µm, accordance of data between both methods was found if the standard laser calculation procedure for spheres was modified for platy discs. Bowen et al. (2002) associated variations between LDA and image analysis to shape factors for mica platelets of the size of 10–1000 µm. Thus, the influence of the particle shape as a main factor for size-detection methods becomes obvious.

3.2 Determination of soil-textural classes and estimates of soil water supply

3.2.1 Soil-textural classes

Standard texture and LDA texture of the 16 samples are listed in Tab. 3. With respect to the standard method, the investigated soils represented all of the major texture groups: Two soils belong to the clay group, four to the loam, five to silty sand each (Tab. 3).

Comparing standard textural classes with “LDA texture” classes (Tab. 3), growing discrepancies with increasing clay content became obvious. The samples in the major texture group “sand” partially showed accordance with the textural classes derived from the LDA method, whereas in the major texture group “silt”, consistency was found rather on the level of the group than on the level of textural classes. In the major texture groups “loam” and “clay”, no correlation was found, neither on the level of the major texture groups nor in the textural classes. Both groups, loam and clay, could not be identi-
fied with the LDA method in any case. Consequently, the LDA-derived clay content cannot serve as a direct substitute for pipette clay content for the texture classification. However, to become applicable for standard texture classification, the LDA clay content was transformed into a pipette like clay content by means of a linear-regression correlation.

Table 4 presents the results applying four linear-regression models (LRM) developed on different data sets:

- LRM (1) for the sediments of Lower Saxony of this study (Fig. 1).
- LRM (2) for diverse sediments of The Netherlands (Konert and Vandenberghe, 1997),
- LRM (3) for loess samples of Belgium (Beuselinck et al., 1998), and
- LRM (4) for marine sediments of The Netherlands (Buurman et al., 2001).

Furthermore, (5) the amount of the <8 µm laser fraction was used as adequate for the standard clay content as it was suggested by Konert and Vandenberghe (1997). All presented regression models (Tab. 4) describe highly significant relationships on a significance level \( \alpha = 0.01 \). The predicting error RMSE of the linear regression of various sediments of the Netherlands LRM (2) was in accordance with the linear-regression model obtained for the samples of Lower Saxony LRM (1). This is an indication of similar quaternary sedimentation history, similar sample pretreatment, and methodological procedure. The linear regression for the loess from Belgium LRM (3) produces a considerable RMSE of 9.5 so that it was not suitable for the samples of Lower Saxony, not even for the apparently corresponding loess-loam samples no. 8–10. The influence of material origin was demonstrated by Buurman et al. (2001) comparing laser-diffraction and pipette method on three different kinds of sediments from The Netherlands and related soils: marine sediments, fluvial sediments, and loess. As a result, the linear-regression model for the clay fraction differed clearly between all three sediment types. Using these three models for the samples of Lower Saxony, the best result to predict pipette clay fraction was achieved with the marine sediment model (LRM [4] in Tab. 4).

Table 4: Determination of clay content—comparison between standard pipette method and corrected LDA analysis derived on the basis of laser measurement by several estimation methods and sample bases: LRM (1) of this study, LRM (2) (Konert and Vandenberghe, 1997), LRM (3) (Beuselinck et al., 1998), LRM (4) (Buurman et al., 2001), and (5) laser fraction <8 µm. RMSE was calculated with pipette fraction as reference.

<table>
<thead>
<tr>
<th>Sample base:</th>
<th>Sample</th>
<th>Clay (%)</th>
<th>Corrected LDA clay (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>soil samples</td>
<td>1 T01 60.8 63.5 60.2 51.7 52.5 71.7</td>
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<td></td>
</tr>
<tr>
<td>soil samples</td>
<td>2 T02 53.5 35.4 35.0 26.4 29.2 29.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lower Saxony</td>
<td>3 L01 25.2 26.8 27.3 18.5 22.0 30.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lower Saxony</td>
<td>4 L02 21.4 22.0 23.0 14.2 18.0 25.4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lower Saxony</td>
<td>5 L03 17.4 17.4 18.8 10.0 14.2 22.4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lower Saxony</td>
<td>6 L04 17.2 20.3 21.4 12.7 16.6 21.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lower Saxony</td>
<td>7 L05 25.6 28.8 28.9 20.2 23.5 32.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lower Saxony</td>
<td>8 U01 20.5 15.4 17.0 8.2 12.5 17.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lower Saxony</td>
<td>9 U02 18.0 20.5 21.7 12.9 16.8 22.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lower Saxony</td>
<td>10 U03 13.0 13.0 14.9 6.0 10.5 12.6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lower Saxony</td>
<td>11 U04 11.9 20.0 21.1 12.4 16.3 11.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lower Saxony</td>
<td>12 S01 10.7 14.3 16.0 7.2 11.6 16.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lower Saxony</td>
<td>13 S02 9.1 7.9 10.3 1.4 6.3 11.7</td>
<td></td>
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<tr>
<td>Lower Saxony</td>
<td>14 S03 7.9 6.9 9.5 0.6 5.5 9.0</td>
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<tr>
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<td>15 S04 1.9 1.6 4.6 –4.3 1.1 4.3</td>
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<td></td>
</tr>
<tr>
<td>Lower Saxony</td>
<td>16 S05 0.8 0.0 3.2 –5.7 –0.2 3.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lower Saxony</td>
<td>RMSE: reference 5.41 5.81 9.52 7.09 7.60</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* LRM: [Corrected LDA clay content] = \( m \times [\text{LDA clay content}] + b \); correlation coefficient \( r \)
The fluvial sediment model and the loess model, which would be expected to gain better accordance, are not demonstrated here because they distinctly failed with a RMSE of 12.5 and 13.6, respectively. Obviously the fraction of particles <8 μm of the laser method (5) did not adequately represent the standard clay content in the soil samples of Lower Saxony and could not be used as a simplified estimation method.

Even highly significant regression models can only produce reasonable approaches if the sample to test corresponds to the statistical population. In this database of soils of Lower Saxony, sample no. 2 (T02) did not fit in the average, as could be noticed from its secluded position in the linear-regression graphs of clay and coarse silt (Fig. 1). This also directs special attention to the origin of the soil material and the correct allocation to an appropriate correlation model.

Table 5: Diverging textural classes of six soil samples of Lower Saxony (N Germany), obtained by three different methods of texture determination.

<table>
<thead>
<tr>
<th>Sample no. code</th>
<th>Standard texture</th>
<th>LDA texture</th>
<th>Corrected LDA texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 T02</td>
<td>Tu2</td>
<td>Ut3</td>
<td>Tu3</td>
</tr>
<tr>
<td>8 U01</td>
<td>U14</td>
<td>Uu</td>
<td>U13</td>
</tr>
<tr>
<td>11 U04</td>
<td>U13</td>
<td>Uu</td>
<td>U14</td>
</tr>
<tr>
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<td>S13</td>
<td>S12</td>
<td>S14</td>
</tr>
<tr>
<td>14 S03</td>
<td>S13</td>
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<td>Su3</td>
</tr>
<tr>
<td>16 S05</td>
<td>Ss</td>
<td>Ss</td>
<td>Su2</td>
</tr>
</tbody>
</table>

Using the linear-regression model for Lower Saxony (LRM [1]) for the prediction of the standard clay content which is used as one parameter for the classification of soil texture, a mean error of estimate (RMSE) of about 5% clay had to be taken into account. Nevertheless, the “corrected LDA texture” classified in this way could meet the textural class found with the standard method in ten out of sixteen soil samples. In five samples, texture classification still drifted (Tab. 5), whereas in the sandy sample S05, a different texture compared to standard texture was found with the corrected LDA method only. Table 5 lists the six samples with diverging textural classes, comparing standard texture, LDA texture, and corrected LDA texture. The discrepancies described above imply that the use of LDA-derived textural classes for the estimation of complex soil properties will produce consecutive errors.

3.2.2 Texture-based estimates of soil water supply

Based on classes of texture, soil bulk density, and content of OM, the main characteristics of soil water supply were estimated according to the German Soil Survey Manual (Ad-hoc-AG Boden, 2005). Figure 3 presents exemplarily for a medium soil bulk density and textural class (Ad-hoc-AG Boden, 2005). For each sample, textural class was determined with three different methods: SM = standard sieve-pipette method, LM = sieve-laser method, cLM = corrected sieve-laser method.

Figure 3: Estimation of the soil-water characteristics: permanent wilting point (PWP), available field capacity (AFC), and air capacity (AC), based upon medium soil bulk density and textural class (Ad-hoc-AG Boden, 2005). For each sample, textural class was determined with three different methods: SM = standard sieve-pipette method, LM = sieve-laser method, cLM = corrected sieve-laser method.

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<tr>
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<td>Su2</td>
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</table>

Produced differences within the air capacity from –7 to +2 vol.-% and within available field capacity from –13 to +2 vol.-%. The use of the corrected LDA texture (classified with cLM) instead of standard texture produced differences within the air capacity from –4 to +12 vol.-%, and within available field capacity from –12 to +3 vol.-%. In each comparison, the maximum differences were assigned to one sample: to the clay sample no. 2 with the LDA texture and to the sandy sample no. 16 with the corrected LDA texture (Fig. 3). There was no general tendency noticeable in the differences for AC and AFC caused by the corrected LDA texture. Due to these uncertainties, LDA-based textural classes cannot be used as base values for the estimation of soil water characteristics.

4 Conclusion

The laser-diffraction analysis (LDA) provides a detailed particle-size distribution with 62 intervals for soil suspensions with particles <63 μm. Converting these intervals into the four standard size fractions of clay (T), fine silt (fU), medium silt (mU), and coarse silt (gU), growing discrepancies from the standard pipette method become apparent with decreasing particle size. The differences between the two methods are systematic and could be described by highly significant linear regressions on an α level of 0.01.

The main reason for the discrepancies is a result of the basic premiss of the two approaches, LDA and pipette method, whereby all particles are of spherical shape which is not met by natural soil material. For model quartz material, e.g., both methods produce similar results (Beuselinck et al., 1998), natural soil samples, however, are of heterogeneous mineral
composition and irregularly shaped. The growing discrepancies from coarse silt to clay of this study indicate growing discrepancies from the ideal quartz sphere that is assigned with the coarser fractions, with decreasing particle size to the clay fraction which is dominated by mostly platy clay minerals. Furthermore, large amounts of the clay fraction cannot be detected by the LDA due to optical reasons (laser wavelength, Fraunhofer theorem, detection limit), while the pipette method collects all particles below the defined equivalent diameter.

Both methods offer improvements such as particle-density correction for the sedimentation velocity and refractive index in Mie theory. However, if these were taken into account, the difficulty is to decide what definite value would represent the complete and often heterogeneous mixture of shapes and minerals in real soil.

The uncorrected use of the LDA clay content led to wrong classification within the textural classes in 14 of 16 soil samples. Only the two sandy samples with a clay content of <5% correspond with the standard method. As a consequence, clay contents from laser-diffraction analysis are not directly usable for the texture classification of soils.

However, LDA clay content could serve as a base for soil textural–class determination with minor deviation if it is converted by a linear regression into a pipette like clay content (corrected LDA clay content). The selection of an appropriate converting system, however, is rather complex as demonstrated by the use of several models described in literature. For the clay-size fraction, both distinction from and accordance with soils of other regions can be found. Application of regression systems based upon apparently similar substrates can lead to severe miscalculations. In addition to the character of the soil substrate, the sample preparation as well as the measurement procedure should be taken into account.

Therefore, for the determination of soil texture of unknown soil samples with LDA-derived and with regression-transformed size fractions, a validation with the standard method is essential in any case to find an appropriate model supplying estimated values with minor deviations.

Acknowledgment

We would like to thank the Fritsch GmbH (Idar-Oberstein, Germany), T. von Dobeneck (Marine Geophysics, University of Bremen), and T. Mörz (Marine Engineering Geology, University of Bremen) for providing the laser-diffraction equipment.

References


