



Literature Review - Measuring and interpretation of settling velocity and particle size

TKI MUSA project



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Summary

This report is part of the research of the MUSA project. In the MUSA-research project, a consortium contractors, consultants and research organizations join forces to increase the understanding of sand-mud dynamics by means of fieldwork campaigns and lab experiments, and to implement this knowledge in engineering tools and advanced models for the prediction of mud and sand transport and associated morphology in tidal conditions with both currents and waves.

Part of the laboratory experiments is the determination of the particle sizes and settling velocities for the finer part of the samples ($<63 \mu\text{m}$). At WaterProof-laboratory, there are two sedimentation methods available for this analysis; the Hydrometer and the TAP-method.

This report describes the procedures and results of both methods for the same samples, to see if there are (significant) differences between the two methods. Additionally, this report studies the differences of these two methods with other available methods such as the Laser-Diffraction method, by looking into their accuracy ranges. Finally, several recommendations are made regarding the methodology that is used.

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1 Introduction

1.1 Background

This report is part of the research of the MUSA project. In the MUSA-research project, a consortium contractors, consultants and research organizations join forces to increase the understanding of sand-mud dynamics by means of fieldwork campaigns and lab experiments, and to implement this knowledge in engineering tools and advanced models for the prediction of mud and sand transport and associated morphology in tidal conditions with both currents and waves. Part of the laboratory experiments is the determination of the particle sizes and settling velocities for the finer part of the samples (<63 μm). At WaterProof-laboratory, there are two sedimentation methods available for this analysis;

- Hydrometer-method;
- TAP-method.

This report describes the procedures and results of both methods for the same samples, to see if there are (significant) differences between the two methods. Additionally, this report studies the differences and accuracies of these two methods with other available methods such as the Laser-Diffraction method.

To this end, this report firstly describes several methods that are commonly used to determine particle sizes and accuracy ranges for different sediment sizes, based on an extensive literature review (chapter 2). Then, the methods and procedures used at WaterProof-laboratory for the two sedimentation methods are explained in chapter 3. Chapter 4 gives an overview of the laboratory tests for these two methods and the accuracies involved. Finally, chapter 5 explains the conclusions of this reports and gives several recommendations for the methods and analysis used in the determination of the particle sizes for the finer part of the samples (<63 μm).

1.2 Research questions

The most basic research questions are:

- 1 How to define the particle size of fines with a platy shape?
- 2 What is the effective diameter of the available methods (Laser-Diffraction method and Sedimentation methods: Hydrometer method and Pipet/TAP method) and how are these diameters related to each other?
- 3 What is the accuracy of both sedimentation methods in measuring the particle size distribution of fines < 63 μm ?
- 4 What is the most accurate method to determine the settling velocity of fine sediments < 63 μm ?

2 Sampling and analysis methods for particle size and settling velocity

2.1 Introduction

Sedimentation and erosion studies require knowledge of the sediment bed characteristics based on collection and analysis of field samples (most generally consisting of clay, silt and sand particles as well as some calcareous and organic materials).

Bed samples are usually taken from a survey vessel using a:

- grab (Van Veen Grab) for samples of the upper 0.1 m of the surface;
- box-corer for undisturbed samples of the upper 0.5 m of the surface;
- piston corer and vibro-corer for undisturbed samples with lengths up to 6 m.

The characterization of the sediment bed samples involves the following aspects:

- description (and photographs) of the sampling method,
- visual features (and photographs) of the samples immediately after sampling;
- determination of content of calcareous and organic materials;
- particle size distribution of primary particles and analysis methods used (including pre-treatment procedures and methods);
- settling velocity distribution of the primary particles and flocs in native (sea) water and analysis method used.

This chapter gives an overview of the sampling and analysis methods which are available and presents the results and accuracy involved.

2.2 Particle size distribution of primary particles

Sand, silt and clay are generally defined by their grain size based on the size distribution of the primary particles (psd), either as mass percentage or volume percentage.

Multiple definitions exist to differentiate sand silt and clay, see Table 2.1 and Table 2.2.

Table 2.1: Sand, silt, clay definitions and US-Wentworth scale.

Classification method	Sand [mm]	Silt [mm]	Clay [mm]
American Geophysical Union scale, Wentworth scale	0.063 – 2	0.04 – 0.063	< 0.004
ASTM D422	0.075 – 4.75	0.05 – 0.075	< 0.005
B.S. 1377	0.060 – 2	0.02 – 0.060	< 0.002
NEN 5104	0.063 – 2	0.02 – 0.063	< 0.002

Table 2.2: Wentworth scale.

Size [mm]	Wentworth Grade	Phi-Scale	Sediment	Sedimentary rock
> 256	Boulder	-8	Gravel	Conglomerate
256 – 64	Cobble	-6		
64 – 4	Pebble	-2		
4 – 2	Granule	-1		
2 – 1	Very coarse sand	0	Sand	Sandstone
1 – 1/2	Coarse sand	1		
1/2 – 1/4	Medium sand	2		
1/4 – 1/8	Fine sand	3		
1/8 – 1/16	Very fine sand	4		
1/16 – 1/32	Coarse silt	5	Mud	Mudstone
1/32 – 1/64	Medium silt	6		
1/64 – 1/128	Fine silt	7		
1/128 – 1/256	Very fine silt	8		
< 1/256	Clay	> 8		

Various methods are available to determine the particle size distribution (psd) of the primary particles:

- Sieving method for the sand fraction > 63 μm (mass-based psd); the size of spherical and ellipsoid sand particles is mostly measured by using the sieve method with a rectangular mesh size (between 63 and 2000 μm); the size of fine particles < 63 μm and especially the very silt fraction < 32 μm cannot easily be measured using the sieve-method;
- Scanning electron microscopy (SEM-method); the shape and size of clay-type particles can be studied qualitatively, but also quantitatively using many samples for representativeness (Conley, 1965; Lu et al., 2000);
- Sedimentation methods (Pipet/TAP-method; Hydrometer-method; Sedigraph-method; mass-based psd derived from settling of sediment in short columns, see Figure 1.2) in combination with the Stokes-diameter equation for the fine fraction > 63 μm ;
- Laser-light diffraction method (Malvern particle sizer; LD-method; Lu et al., 2000; Haverbeke, 2013; Yang et al., 2019; Ibanez Sanz, 2018; volume-based psd) which is most appropriate for spherical particles of silt and sand and less for the flaky clay particles and mixtures with a wide range of sediment sizes, where smaller particles are shaded by larger particles (Ibanez Sanz, 2018);
- Particle count methods based on measuring changes of electrical resistance of particles with different sizes (Coulter counter).

Ideally, the analysis of the particle size distribution should be done on samples with and without the presence of calcareous (shells and shell fragments) and organic materials so that the influence of these materials can be determined. This involves sample pre-treatment, such as:

- removal of coarse materials > 2 mm by sieving;
- removal of calcareous materials using a HCl-solution (Hydrogen Chloride acid);
- removal of organic materials using a H₂O₂-solution (Hydrogen Peroxide) or by heating (loss on ignition);
- dispersal (loosening) of primary particles using anti-flocculation solutions (Calgon; Peptisator) in distilled water for 24 hours; sample shaking for 15 minutes may be used; ultra-sonic vibration in a water bath may also be used (optional), but not longer than 10 minutes to prevent breaking of sediment particles (Deltares, 2014).

Given the wide range of instruments and methods for measuring the particle size distribution (psd), it is not surprising that the results of these methods may differ substantially, particularly for the finer platy/flaky clay-type particles, which is discussed hereafter.

2.2.1 LD-method

The Laser-Diffraction method (LD) is a sophisticated (expensive) method for rapid analysis of many samples, as small wet subsamples can be used. However, because of the small sample size involved, many samples of the same base sample should be used, as only 1 or 2 subsamples may not be sufficiently representative for the bulk sample/soil. The method is very accurate for spherical (silt, sand) particles, but the accuracy of this method for the more flaky/platy clay-type particle is not yet clear. The sediment concentration of the test sample should be smaller than about 0.7 g/l, otherwise the particle size is reduced (slightly) due to instrumental errors. The $d_{50,LD}$ is 5% smaller for a larger sample concentration (4 g/l instead of 0.2 g/l), see Ibanez Sanz (2018). Ibanez Sanz (2018) has also found that the LD-method yields erroneous results for mixtures of mud and sand, because smaller particles are shaded by the larger particles in the subsample used during testing by LD.

The LD-method takes both the particle shape, size and its optic properties into account. A particle will scatter the incident light and through a number of detectors, the intensity and shape of this scattering pattern can be measured. The Fraunhofer or the Mie theory can be used to interpret the obtained pattern. Larger particles will scatter strongly over small angles, small particles will do so more weakly and over greater angles (Figure 2.1). Smaller particles pass through the light source more than once (they are suspended in a closed water circuit) with a different orientation each time, resulting in an 'equivalent spherical diameter', which is a sphere producing the same scattering pattern. The measuring range is 0.04 to 2000 μm .

To determine the particle size of the primary particles, the diluted sample is pre-treated with a chemical anti-flocculant (Peptiser/Calgon) and/or ultrasonic stirring. The sample concentration should be as uniform as possible, which is achieved by continuous recirculation of the sample volume (by pumping) during the measurement time. A sample dispersion unit is available for sample preparation. It is remarked that due to extensive ultrasonic treatment, sand particles may break up, resulting in an overestimation of the finer fraction. Sensitivity tests (Deltares, 2014) show that ultrasonic treatment should not be longer than about 10 minutes.

LD gives a particle diameter for a sphere with equivalent cross-sectional area averaged over all particle orientations. As spheres have the largest volume compared with disc-type particles with the same cross-sectional area, the particle sizes derived from LD are expected to be larger than the actual volume-equivalent sphere diameters. Thus, LD leads to overestimation of the particle size of platy particles.

In the case of very small disc-type or plate-type clay particles, it is not so clear what type of diameter is measured by the LD-method. Brown and Felton (1985) have found that the projected area of the plates (from which the LD-diameter d_{LD} is derived) as measured by the LD-instrument is equal to 0.25 times the total area of the plates. The total area of the plates is defined as the summation of 2 flat sides and two half edge sides. Using this theory and defining the equivalent diameter of the plate as d_{plate} and the thickness of the plate as δ_{plate} , the LD-diameter can be derived as follows:

$$\frac{1}{4} \pi d_{LD}^2 = \frac{1}{4} [2 * \frac{1}{4} \pi d_{plate}^2 + 2 * \frac{1}{2} * \delta_{plate} \pi d_{plate}]$$

This leads to:

$$d_{LD}^2 = d_{plate}^2 [0.5 + (\delta_{plate}/d_{plate})] \text{ or,} \\ d_{LD} = d_{plate} [0.5 + (\delta_{plate}/d_{plate})]^{0.5} \quad (2.1)$$

Assuming that δ_{plate}/d_{plate} is in the range of 0.01 to 0.1, the LD-diameter can be found:

$$\delta_{plate}/d_{plate} = 0.1, \text{ it follows that: } d_{LD} \cong 0.77d_{plate} \text{ or } d_{plate} = 1.3d_{LD} \quad (2.2a)$$

$$\delta_{plate}/d_{plate} = 0.01, \text{ it follows that: } d_{LD} \cong 0.71d_{plate} \text{ or } d_{plate} = 1.4d_{LD} \quad (2.2b)$$

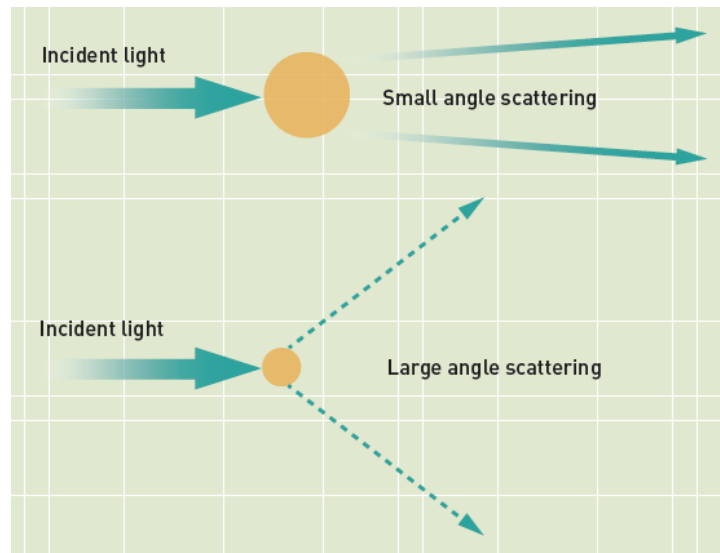


Figure 2.1: Light scattering at small and large angles; LD-method.

The LD-method is known to underestimate the clay fraction ($d < 2 \mu\text{m}$), as the fines are partly shadowed by the larger particles during testing of the subsample. This can be partly overcome by separating the finest fraction from the bulk by means of a settling test, in which the finest fraction remains in suspension after one hour (Ibanez Sanz, 2018). The subsample can also be washed over a $63 \mu\text{m}$ -sieve. The ratio of the mass of the finest fraction to the overall bulk mixture can be calculated, and the particle size distribution (psd) can be corrected with the psd of the finest fraction.

2.2.1.1 Experimental results

Blott and Pye (2006) have found that the LD-method gives larger particle diameters (up to 20%) for ellipsoid sand particles than the sieve-method. These non-spherical sand diameters can pass through the sieve mesh with their smallest diameter whereas the diameter from laser diffraction is derived from the volume of the particles. Deltares (2012) found discrepancies up to 50% for sand particles.

Haverbeke (2013) also found that the sieve method produces smaller d_{50} -values (about 15% to 20% for medium fine sand of 0.15 to 0.3 mm) than the LD-method

Ibanez Sanz (2018) found the following results comparing grain sizes from the sieve-method and the LD-method:

$d_{50,LD} = 175 \mu\text{m}$ and $d_{sieve} = 137.5 \mu\text{m}$ for narrow sand fraction 125-150 μm ; $d_{50,LD} = 60 \mu\text{m}$ and $d_{sieve} = 49 \mu\text{m}$ for narrow silt fraction 45-53 μm .

It was also found that the LD-method is dependent on the sediment concentration of the subsample. The $d_{50,LD}$ was 5% smaller for a larger sample concentration (4 g/l in stead of 0.2 g/l)

Based on available results for sand particles $> 63 \mu\text{m}$: $d_{LD} \cong 1.2$ to $1.3 d_{sieve}$.

Di Stefano et al. (2010) have found that the percentage of clay based on the LD-method may be 50% smaller compared to that measured by sedimentation methods.

Yang et al. (2019) have tested 613 soil samples from a typical cropland in the region northeast of Beijing (China) using the LD-method (Malvern Mastersizer 2000) and other methods. Sample pre-treatment was the same for all samples (drying, sieving, removal of organic and calcareous materials and dispersal with sodiumhexameta-phosphate-solution). Particle size distributions were also measured with Sieve-Pipet (SP) method (sieving for fraction $> 100 \mu\text{m}$) and Scanning

Electron Microscopy (SEM-method). SEM was used for 100 samples and LD for 277 samples based on random selection. The results of Yang et al.(2019) are presented in Table 2.3.

The sand content $> 53 \mu\text{m}$ measured by LD is almost the same as that of the Sieve-method (within experimental error). The silt and sand content of all fractions $> 20 \mu\text{m}$ are measured correctly by both methods LD and SP (about 53%). This means that the fraction $< 20 \mu\text{m}$ is also correctly measured by both methods. However, LD underestimates the clay content $< 2 \mu\text{m}$ and thus overestimates the fine silt content 2-20 μm compared to the SP-method.

The SEM-method of Yang et al. (2019) was used to determine the psd of the clay fraction with a spherical Stokes diameter $< 2 \mu\text{m}$ (based on the Pipet-Sedimentation method). The maximum plate diameter detected with SEM is 4.8 μm . The SEM-images of clay particles are shown in Figure 2.2. The clay content based on the PS-method reduces to 4.4% if all plate-type clay particles $> 2 \mu\text{m}$ are removed from the sample. Thus, the Pipet-Sedimentation method severely overestimates the clay content in terms of plate diameter, because the Stokes sphere diameter is much smaller than the plate diameter. Based on SEM, most (50% of the counts) of the clay particles are in the range of 0.2 to 0.5 μm , about 30% in the range of 0.5 to 1 μm and about 20% in the range of 1 to 2 μm . Based on the counts, the mean overall plate diameter is about 0.5 μm . Using a volume approach (with plate thickness equal to 0.1 plate diameter; $\delta \cong 0.1 d_{\text{plate}}$), the equivalent sphere diameter is found to be about 1.3 μm by Yang et al. (2019)

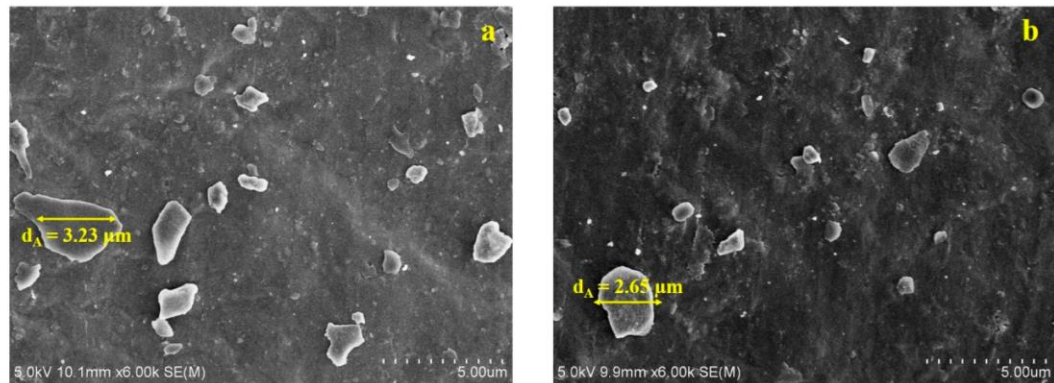


Figure 2.2: SEM-images of plate-type clay particles $> 2 \mu\text{m}$ at magnification of 6000 (d_A = equivalent plate diameter), (yang et al., 2019).

Table 2.3: Sediment fractions measured with different methods (Yang et al. 2019).

Size class as equivalent sphere diameter or plate diameter [μm]	Percentage of sediment in a size class [%]		
	Sieve-Pipet method	Laser-Diffraction method	SEM-method
100-2000	15	17	-
53-100	12	13	-
20-53	26	22	-
2-20	23	42	-
<2	25	6	4.4

2.2.2 Sedimentation methods

Particle size analysis of the fine sediment fraction using sedimentation methods is more laborious and involves the drying, weighing, rewetting and loosening of the sediment particles with deflocculant (sodiumhexameta-phosphate-solution) including or excluding the removal of calcareous and organic materials (sample pre-treatment).

Using a settling tube, the effective settling velocity is measured, which can be converted to an equivalent spherical diameter from the Stokes' settling equation.

The Sedigraph is used at the Soil Mechanics Laboratory of Wiertsema (NL). This instrument has a small cell-type container, which is filled with a mud suspension and the mud concentrations of the settling sediments are determined by direct (pre-calibrated) x-ray absorption inside a narrow beam. Using the known settling height, the decreasing mud concentrations in time are converted to settling velocities and to equivalent (spherical) sediment diameters with the Stokes settling formula.

The Hydrometer method and the TAP method are used at WaterProof BV. Laboratory. The settling column of the Hydrometer is filled with a high-concentration mud suspension in controlled temperature conditions. The decreasing sediment density as function of time is measured by using a floating body in the column with settling mud particles. The effective settling distance is the distance between the centre of gravity (centre of bulb) of the floating body and the surface of the suspension, which decreases as function of time. The settling column of the TAP-method is filled with a low concentration suspension. Small subsamples are taken by using a tap at about 7 cm above the bottom of the settling column. The concentrations of the subsamples are determined by filtration, drying and weighing.

The equivalent spherical settling diameter $d_{\text{sphere, stokes}}$ follows from the Stokes settling equation, which reads as:

$$w_s = (s-1) g (d_{\text{sphere, stokes}})^2 / (18 \nu) \quad (2.3)$$

with:

w_s = settling velocity,

$s = \rho_s / \rho$ = relative density,

ρ_s = sediment density (2650 kg/m³),

ρ = fluid density,

ν = kinematic viscosity coefficient,

d_{sphere} = equivalent spherical settling diameter of Stokes.

Two cases are defined for platy clay-type particles:

- 1 The settling velocity of a disc-type particle falling with its flat side normal to the vertical direction;
- 2 The settling velocity of a disc-type particle falling with its thin side normal to the vertical direction.

2.2.2.1 Effect of plates on settling velocity

The differences in measurement methods as introduced above are strongly influenced by the platy shape of clay particles affecting the scattering and settling processes. We therefore analyze this shape in more detail, provide relationships between the settling speed and plate diameter, and interpret differences in settling speed observed with different instruments and reported in the literature.

The settling velocity of a disc-type particle (with thickness defined as δ_{plate} and the plate or disc diameter defined as d_{plate}) falling with its flat side normal to the vertical direction can be derived from the drag force and the submerged volume weight.

The drag force is defined as (Clarkson University, 2020):

$$F_{\text{drag}} = 8 \rho \nu d_{\text{plate}} w_s \quad (2.4)$$

and the gravitational force is:

$$G_{\text{plate}} = 0.25 \pi d_{\text{plate}}^2 \delta_{\text{plate}} (\rho_s - \rho) g \quad (2.5)$$

The settling velocity follows from equilibrium of both forces resulting on:

$$w_s = \pi (s-1) g \delta_{\text{plate}} d_{\text{plate}} / (32\nu) \quad (2.6)$$

The settling velocity of a disc-type particle (with thickness defined as δ_{plate} and the plate or disc diameter defined as d_{plate}) falling with its thin side normal to the vertical direction can be derived from the drag force and the submerged volume weight.

The drag force is defined as:

$$F_{drag} = 5.3 \rho v d_{plate} W_s \quad (2.7)$$

and the gravitational force is:

$$G_{plate} = 0.25 \pi d_{plate}^2 \delta_{plate} (\rho_s - \rho) g \quad (2.8)$$

The settling velocity is:

$$W_s = \pi (s-1) g \delta_{plate} d_{plate} / (21.3v) \quad (2.9)$$

The plate diameter can be related to the spherical Stokes-diameter by assuming equal settling velocity, giving:

- disc with flat side normal to vertical direction: $d_{plate} = 0.75 (\delta_{plate}/d_{plate})^{-0.5} d_{sphere, stokes}$ (2.10)

- disc with thin side normal to vertical direction: $d_{plate} = 0.62 (\delta_{plate}/d_{plate})^{-0.5} d_{sphere, stokes}$ (2.11)

Assuming δ_{plate}/d_{plate} to be in the range of 0.02 to 0.1, it follows that: $d_{plate} = 2$ to $5 d_{sphere, stokes}$

Thus, if the settling velocity of very small plate-type clay particle is measured and converted to an equivalent spherical diameter using the Stokes settling velocity formula, the actual plate diameter of these particles is about 2 to 5 times larger than the equivalent spherical diameter.

Experimental results of Lu et al. (2000) suggest a value $d_{plate} \cong 5 d_{sphere, stokes}$ (see below).

Based on Equations (2.1), (2.10), (2.11), a general expression is:

$$d_{sphere, stokes}/d_{LD} \cong 1.6 (\delta_{plate}/d_{plate})^{0.45} \quad (2.12)$$

Pabst and Berthold (2007) have given:

$$d_{50, sphere}/d_{50, LD} = 2.5 (\delta_{plate}/d_{plate})^{0.5} \quad (2.13)$$

Both results are plotted in Figure 2.3, showing large discrepancies.

Measured data of Haverbeke (2013), Lu et al. (2000) and Ibanez Sanz (2018) are also shown, assuming that the ratio δ_{plate}/d_{plate} is most likely in the range of 0.02 to 0.1. Yang et al. (2019) observed $\delta_{plate}/d_{plate} \cong 0.1$ for clay particles based on Scanning Electron Microscopy (SEM).

Overall, Equation (2.12) of this study is in better agreement with the data than Equation (2.13).

The measured data are discussed in more detail hereafter. Lu et al. (2000) compared the hydrometer-sedimentation method (H-method), Laser-Diffraction method (LD) and Scanning Electron Microscopy-method (SEM for kaolinite material from Georgia (USA)). The average major dimension (disc diameter) of platy kaolinite particles was between 1 and 3 μm based on SEM. The particle size measured with the LD-method was $d_{50, LD} = 2.5 \mu m$ with a percentage clay $< 2 \mu m = 45\%$. The Hydrometer-method measured $d_{50, stokes} = 0.55 \mu m$ with a percentage clay $< 2 \mu m = 75\%$. Thus, the LD-method measured a value close to the average major dimension (disc diameter) of the platy kaolinite particles, whereas the Hydrometer-method yields a much smaller (almost 5 times) spherical Stokes settling diameter: $d_{50, sphere} \cong 0.22 d_{50, LD}$ for kaolin-clay.

Ibanez Sanz (2018) compared the Hydrometer-method, the Sedigraph-sedimentation method based on X-ray detection and the LD-method for clay-type samples. It was found that:

$$d_{50, sedigraph-stokes} \cong 0.2 d_{50, LD} \text{ and } d_{50, hydrometer-stokes} \cong 0.65 d_{50, LD}$$

The Sedigraph-method produced a relatively high percentage of fines ($55\% < 2 \mu m$) compared to the Hydrometer method ($40\% < 2 \mu m$) and LD-method ($20\% < 2 \mu m$). Finally, Vitton and Sadler

(1997) and Hilbrand and Row (1995) also found that the sedimentation methods seriously underestimate the major dimension (disc diameter) of the clay-type particles.

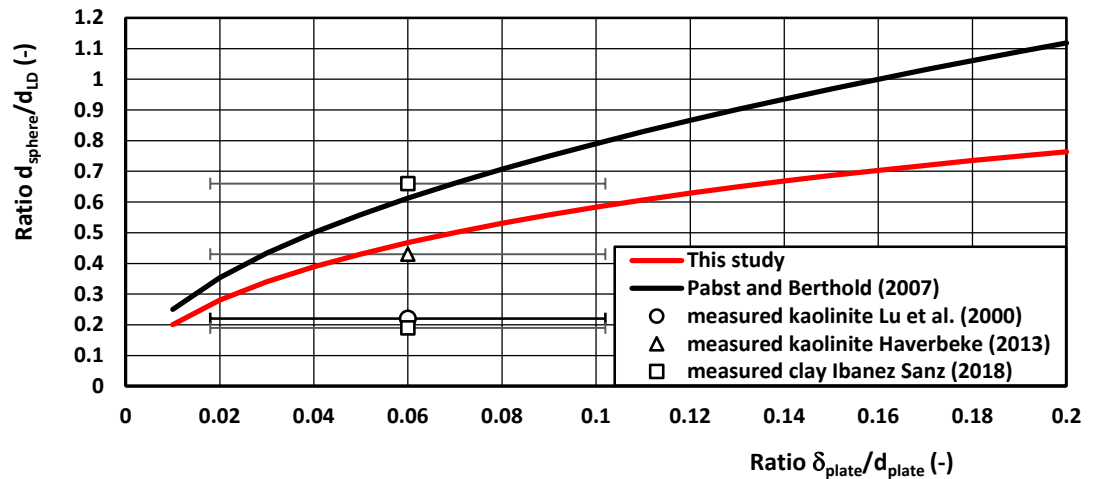


Figure 2.3: Ratio of sphere diameter and LD-diameter as function of d_{plate}/δ_{plate} .

2.2.2.2 Experimental results

Coates and Hulse (1985) have done a detailed comparison of the Pipet-method (see Section 3.4) and the Hydrometer-method (see Section 3.3).

Two different soil samples were used:

- Sample A: high-plasticity mud with $p_{sand}=6\%$, $p_{clay < 2 \mu m}=24\%$;
- Sample D: mud of grinded quartz with $p_{sand}=26\%$, $p_{clay < 2 \mu m}=7\%$.

Pipette analyses were conducted by taking withdrawals of a precise pipet volume from a suspension column at known depths and times after stirring ceases. Approximately 15 g of mud in 1 L of water was used. Sample pre-treatment included disaggregation, subsampling, dispersion in a Calgon solution, wet sieving at 63 μm and transfer of the suspension to a 1 L graduated cylinder. Hydrometer analyses were conducted in accordance with the method of the American Society for Testing Materials (D 422) using an ASTM 152H hydrometer. The samples were dried and weighed initially. Subsamples of 30-40 g were used in distilled water of 1 L with Calgon solution. Sand fraction was removed by wet sieving over 63 μm .

Figure 2.4 shows comparison results of the Hydrometer-method and the Pipet-method. Analysis of the results can be summarized as:

- **Sample A**
 - Hydrometer-method: $d_{50}=16 \mu m$; $p_{clay < 2 \mu m}=28\%$;
 - Pipet-method: $d_{50}=22 \mu m$; $p_{clay < 2 \mu m}=24\%$;
- **Sample B**
 - Hydrometer-method: $d_{50}=22 \mu m$; $p_{clay < 2 \mu m}=12\%$;
 - Pipet-method: $d_{50}=30 \mu m$; $p_{clay < 2 \mu m}=6\%$.

The Hydrometer-method leads to smaller particle sizes and larger clay fractions. The relative difference of the d_{50} due to experimental errors is up to 35% for both samples. The relative difference for the percentage of clay < 2 μm is up to 20% for the muddy sample A and about 50% to 100% for the more silty sample D.

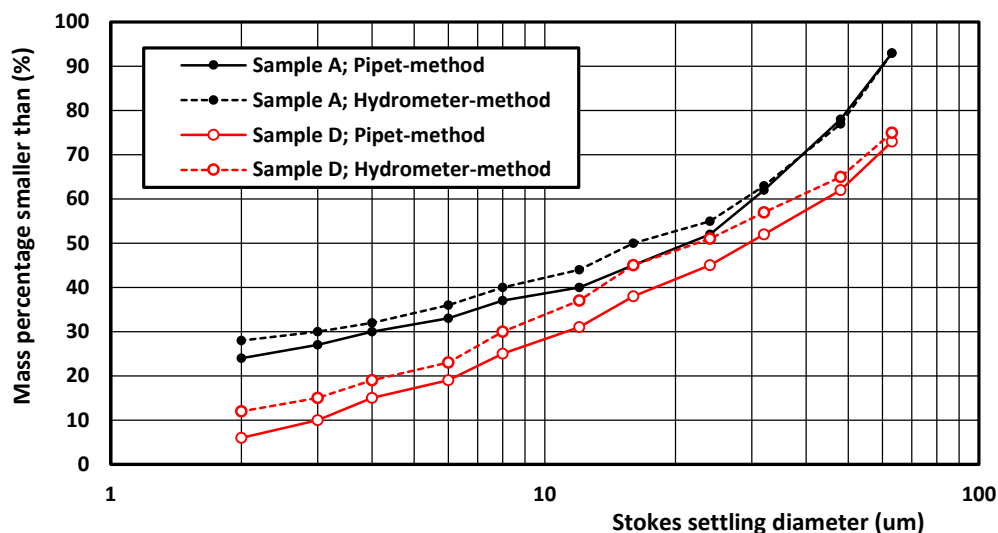


Figure 2.4: Comparison of particle size distributions based on Hydrometer-method and Pipet-method (Coates and Hulse, 1985).

Lu et al. (2000) studied the accuracy of the hydrometer-sedimentation method (H-method) for fine-grained silt and clay particles. Sedimentation methods are based on the Stokes' settling equation. The validity of this equation for fine sediment is based on the assumption of laminar flow around spherical particles during settling in a high-concentration suspension. The maximum concentration should not be larger than about 50 g/L to prevent hindered settling effects (Weatherly, 1929). A major problem is that clay-type particles are platy rather than spherical resulting in deviations between the spherical particle size from the Stokes' equation and the actual disc-type particle size of the platy clay particles. This problem was addressed theoretically and experimentally by Lu et al. (2000). They presented analytical expressions relating the actual disc-type particle size of clay to the Stokes' diameter. Based on this, it was shown that a spherical diameter of 10 µm has the same settling velocity as a disc-type particle with diameter 16 µm and thickness of 2 µm and a disc-type particle of 32 µm with thickness of 1 µm. These findings were confirmed by two experiments under controlled conditions.

The first experiment concerns the particle size (psd) of standard kaolinite from Georgia (USA). It is widely accepted that the actual average major dimension (disc diameter) of platy kaolinite particles is between 1 and 3 µm based on results from Scanning Electron Microscopy (SEM). The particle size was measured with the Laser-Diffraction method (LD-method; Malvern particle sizer) and with the Hydrometer-method (H-method). Sample preparation was identical for both methods (drying, weighing, and dispersal in 4%-solution of sodiumhexameta-phosphate).

The results are, as follows:

- H-method: $d_{50, \text{stokes}} = 0.55 \mu\text{m}$; percentage clay < 2 µm = 75%.
- LD-method: $d_{50, \text{LD}} = 2.5 \mu\text{m}$; percentage clay < 2 µm = 45%;

Thus, the LD-method reflects the average major dimension (disc diameter) of the platy kaolinite particles, whereas the H-method yields the much smaller (almost 5 times) spherical Stokes settling diameter. Thus, $d_{\text{LD}} \cong 5d_{\text{sphere, stokes}}$ for kaolin-clay.

Haverbeke (2013) compared the psd of kaolin from St. Aultell (UK) measured with the Hydrometer-method and the LD-method. He found:

- H-method: $d_{50, \text{stokes}} = 3 \mu\text{m}$; percentage clay < 2 µm = 35%.
- LD-method: $d_{50, \text{LD}} = 7 \mu\text{m}$; percentage clay < 2 µm = 12%.

Thus, $d_{\text{LD}} \cong 2.3d_{\text{sphere, stokes}}$ for kaolin-clay.

Vitton and Sadler (1997) and Hilbrand and Row (1995) also found that the sedimentation methods seriously underestimate the major dimension (disc diameter) of the clay-type particles.

The other experiment of Lu et al. (2000) concerns the comparison of the psd of pulverized mica (solid density of 2760 to 2880 kg/m³) measured with the Hydrometer-method (H-method) and with the sieve method (S-method). The pulverized mica was mechanically sieved to retain the fraction between 43 and 75 μm which consisted of platy particles based on SEM. The results of both methods are:

- S-method: $d_{50}=60 \mu\text{m}$;
- H-method: $d_{10}=13 \mu\text{m}$, $d_{50}=26 \mu\text{m}$, $d_{90}=35 \mu\text{m}$.

Thus, the H-method producing a spherical diameter underestimates the disc-type diameter of the S-method by a factor of 2.3 or $d_{\text{sieve}} \cong 2.3d_{\text{sphere,stokes}}$ for coarse silt. The analytical expression in the paper of Lu et al. (2000) for disc-type particles is in good agreement with the result of the S-method for a disc with diameter of 60 μm and thickness of about 3 μm.

Ibanez Sanz (2018) has compared the Hydrometer-sedimentation method (H), the commercial Sedigraph-sedimentation method based on X-ray detection (S) and the LD-method (LD) for a clay-type sample, as follows:

- H-method: $d_{50,\text{stokes}} = 5 \mu\text{m}$; percentage < 2 μm= 40%; percentage < 8 μm= 55%;
- S-method: $d_{50,\text{stokes}} = 1.5 \mu\text{m}$; percentage < 2 μm= 55%; percentage < 8 μm= 70%;
- LD-method: $d_{50,\text{LD}} = 7 \mu\text{m}$; percentage < 2 μm= 20%; percentage < 8 μm= 55%.

The Sedigraph-method produces a relatively higher percentage of fines, which has also been noticed by Van Rijn (2018) for mud from Noordpolderzijl (The Netherlands). Ibanez Sanz argues that the differences may be related to the preparation procedures involved. The H- and S-methods require that the clay sample is dried beforehand for weighing and pulverized again in a mortar which may result in smaller particles due to the grinding process.

Summarizing, it is concluded that:

- the plate diameter is about 2 to 5 times larger than the equivalent sphere diameter of Stokes derived from settling velocity data for platy fines < 8 μm ($d_{\text{plate}} \cong 2 \text{ to } 5 d_{\text{sphere,stokes}}$);
- the equivalent plate diameter is about 1.3 to 1.4 times the size derived from the LD-method for platy clay and silt particles ($d_{\text{plate}} \cong 1.3 \text{ to } 1.4 d_{\text{LD}}$);
- the equivalent sphere diameter of Stokes is about 0.2 to 0.6 times the diameter derived from the LD-method ($d_{\text{sphere,stokes}} \cong 0.2 \text{ to } 0.6 d_{\text{LD}}$) for platy clay-type particles with $\delta_{\text{plate}}/d_{\text{plate}} < 0.1$;
- the percentage of clay < 2 μm (in terms of equivalent spherical diameter) of muddy samples can be measured with by the Hydrometer-method and the Pipet-method; the inaccuracy is a factor of 1.2 (smaller or larger) for clay percentages > 25% going up to an inaccuracy of factor of 2 for small clay percentages < 10% percentages (Coates and Hulse, 1985).

2.3 Settling velocity distribution of primary particles and flocs

An alternative method for determining the particle size distribution is to directly measure the settling velocity distribution of the particles and flocs. The particles and flocs of the upper bed sediments have to some extent taken part in the dynamic near-bed exchange processes and therefore it is most logic to study the settling behaviour of these sediments. This can be done most easily in a laboratory settling column with native (sea) water and wet subsamples without pre-treatment. Only wet subsamples should be used (no drying and loosening). Wet sieving using a 63 μm-sieve may be applied to remove the sand fraction to study the effect of fine sand particles on the settling of the fine fraction (with and without tests).

Examples of these types of tests are shown in Figure 2.5.

Examples of the settling velocity as function of the mud concentration are shown in Figure 2.6.

Various subsamples should be taken from the original bed sample and diluted/mixed using native water to obtain concentration suspensions, as follows (Figure 2.5):

- flocculating range: 0.3, 0.5, 1, 3, 5 g/l using the TAP-method;
- hindered settling range: 10, 30, 50, 100, 200 and 300 g/l using the hydrometer-method and/or the consolidation method (sinking of sediment interface in short settling column).

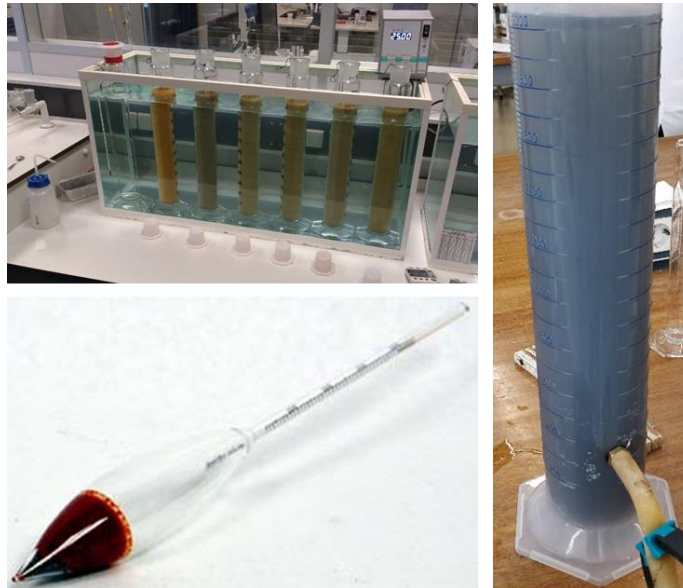


Figure 2.5: Sedimentation methods. (Left) Hydrometer-method with settling columns and float. (Right) TAP-method with settling column.

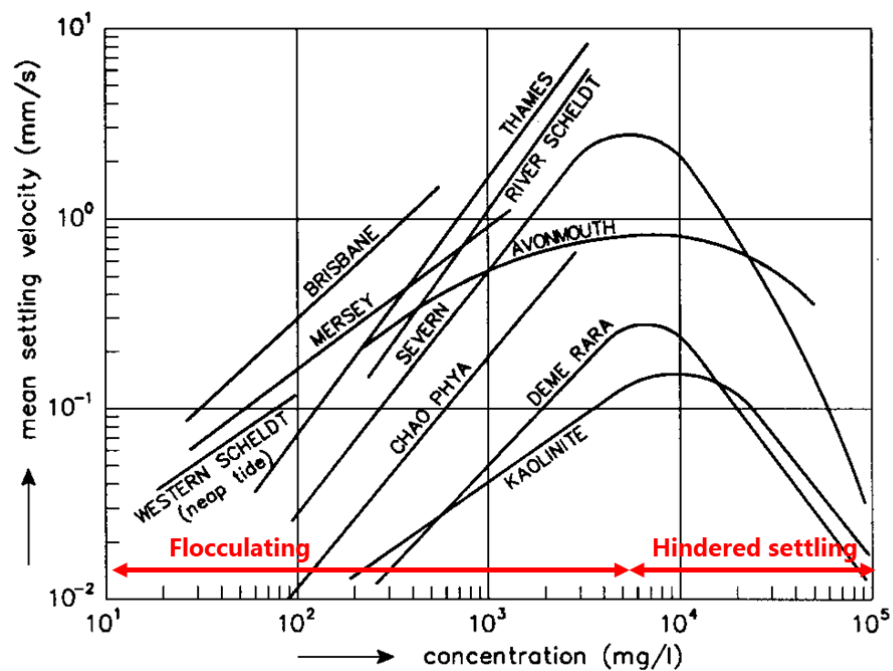


Figure 2.6: Settling velocity as function of concentration (Van Rijn 1993).

3 Methods and procedures of sedimentation methods

3.1 Introduction

This chapter describes the methods and procedures for the two sedimentation methods: Hydrometer-method and TAP-method. Information from other studies (literature) is also included. Sedimentation methods are most appropriate for the fine particles < 63 μm; both the particle size and the settling velocity can be determined using this type of method, see Figure 3.1.

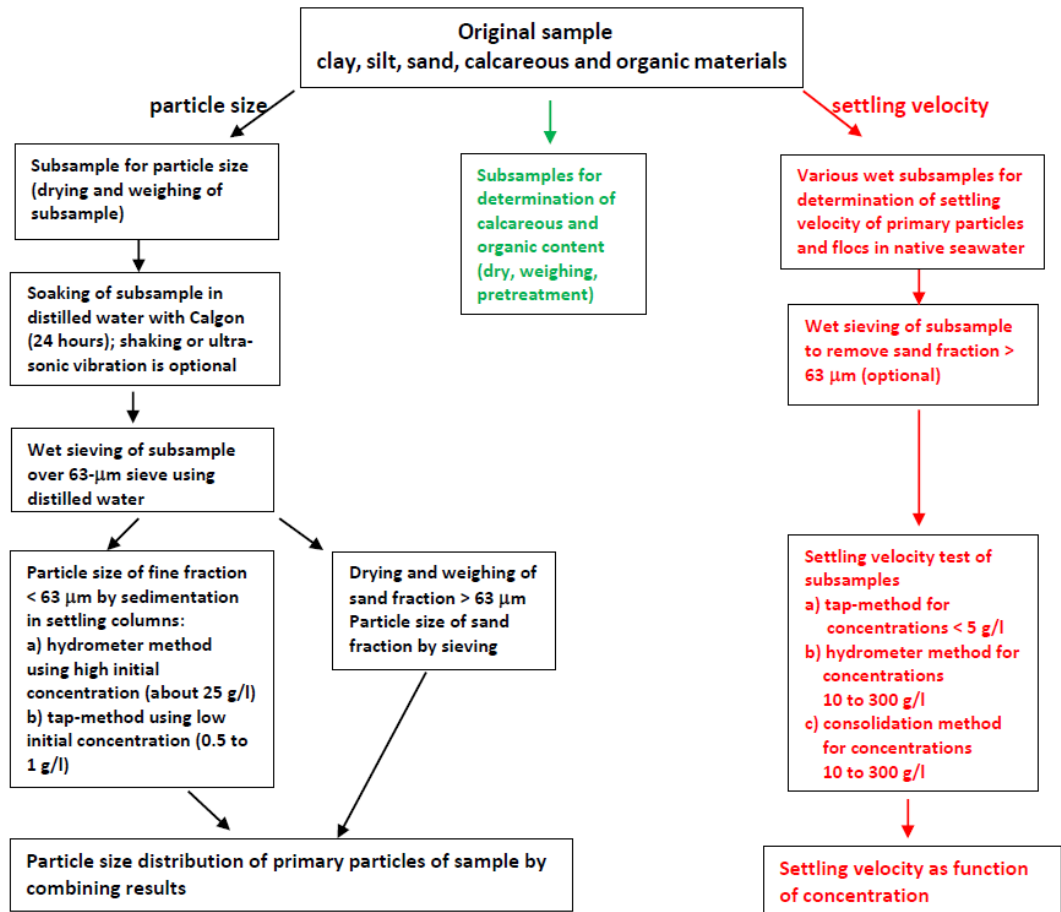


Figure 3.1: Schematic procedure for determination of size and settling velocity of fine particles using sedimentation methods

3.2 Sample pretreatment and preparation

3.2.1 Sample pretreatment

The starting point of the size determination of the primary particles using sedimentation methods is a dry sediment sample with known mass (M_{total}). Therefore, each subsample from the original base sample has to be dried, weighed and loosened again by rewetting with dispersing-solution (known as Calgon).

Kaur and Fanourakis (2016) have studied the effects of various types of dispersing agents on the percentage of clay ($< 2 \mu\text{m}$) in soil samples using the Hydrometer-method. Two types of floats were used: 152h and ASTM152h:E100, see Figure 3.2.



Figure 3.2: (Left) Float 152h and (Right) ASTM152H:E100

The dispersing agent (DA) is a mixture of sodiumhexameta-phosphate (S) and Natriumcarbonate (N), known as CALGON.

The following CALGON-solutions were used:

- 35 g S+ 0 g N in 1 liter distilled water (3.5% solution; 35 gram in 1000 gram water);
- 35 g S+ 20 g N in 1 liter distilled water (5.5% solution; 55 gram in 1000 gram water);
- 35 g S+ 20 g N in 1 liter distilled water (6.5% solution; 65 gram in 1000 gram water);
- 35 g S+ 7 g N in 1 liter distilled water (4.2% solution); reference standard solution.

Sample preparation of Kaur and Fanourakis 2018 was, as follows:

- 1 make suspension of 50 gram soil, 400 ml distilled water and volume of DA (volume of DA was varied in the range of 25 to 200 ml);
- 2 suspension was allowed to stand overnight (> 16 hours);
- 3 suspension was dispersed/mixed for 15 minutes using a paddle (paddle was rinsed clean with distilled water; wash water goes into suspension);
- 4 pour suspension in glass cylinder and add distilled water to 1000 ml;
- 5 use stirring rod to make a homogeneous suspension;
- 6 start stopwatch; insert float; take reading at 18, 40 s, 2, 5, 1 5, 30 min, 1, 4, 24 hours (hydrometer is removed after each reading for times > 2 min and placed in glass with distilled water
- 7 similar readings were done at the same times in cylinder with a blank solution of 1 liter of distilled water and the same DA solution
- 8 correct readings by subtracting the blank readings.

The best results (highest clay content) are obtained for a volume of 125 ml of 4.2%-solution DA, see Figure 3.3 and Figure 3.4. Using this solution, the mass of DA added to 1 liter of suspension is 5.25 gram to 1000 gram water, which is equal to 0.525%

Table 3.1 shows the effect of dispersing agents (DA) on the percentage of fines of Black soil (based on the data of Figure 3.4) with respect to the total mass of the sample. All samples were washed through a sieve mesh of $75 \mu\text{m}$ (wet sieving), which implies that the percentage $< 75 \mu\text{m}$ should be constant. However, the test results show a varying percentage $< 75 \mu\text{m}$. Calgon of 125 ml 4.2%-solution leads to the highest percentage $< 75 \mu\text{m}$ and $< 4 \mu\text{m}$. The reason for this is unknown. Test results without DA shows a strong decrease of the fraction of fine materials.

Table 3.1: Effect of various types of dispersing agent on percentage of fines of Black soil.

Type of dispersing agent	Percentage $< 75 \mu\text{m}$	Percentage $< 30 \mu\text{m}$	Percentage $< 10 \mu\text{m}$	Percentage $< 4 \mu\text{m}$
None	44%	24%	3%	$< 1\%$
125 ml Calgon	52%	44%	38%	34%

20 ml Sodium-tetra-Pyrophosphate	49%	38%	25%	20%
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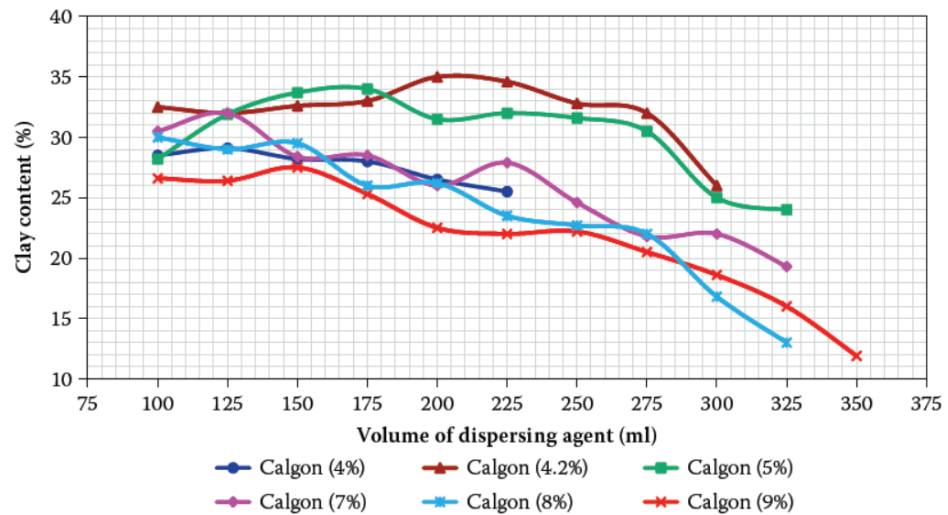


Figure 3.3: Effect of various types of Calgon on the clay content of Black soil (Kaur and Fanourakis, 2016).

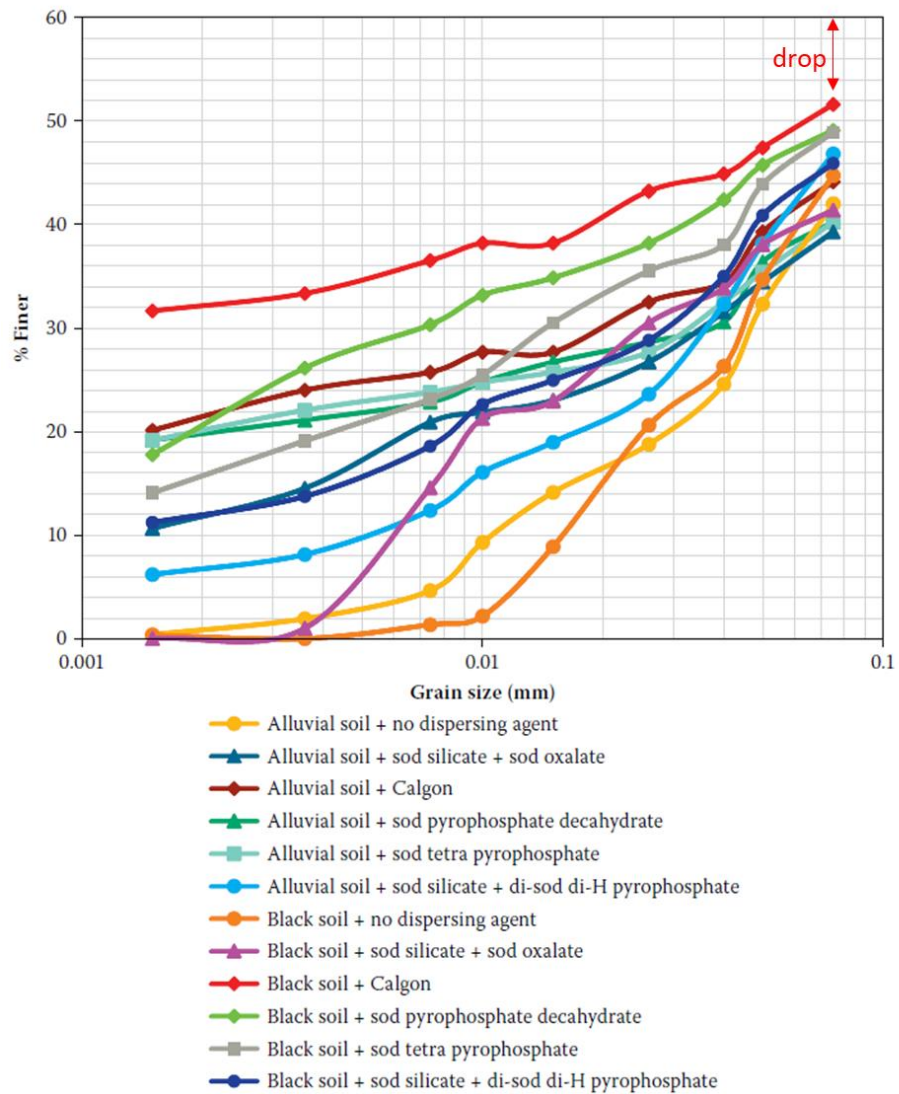


Figure 3.4: Effect of various types of Calgon on the particle size distribution of Alluvial soil and Black soil. Adapted from (Kaur and Fanourakis, 2016).

Finally, some remarks are given with respect to various characteristics of Calgon.

- Solid density Natrium-Carbonate= 2540 kg/m³;
- Solid density Sodiumhexameta-phosphate = 2430 kg/m³;
- Solid density mixture Calgon consisting of N + S (35/7) = 2520 kg/m³;
- Density water at 21.4 °C = 997.9 kg/m³.

Using these characteristics and the density equation, the density of the deflocculant mixture and the deflocculant-sediment mixture can be determined:

$$\rho_{\text{mixture}} = \rho_{\text{water}} + [(\rho_{\text{solids}} - \rho_{\text{water}}) / \rho_{\text{solids}}] C_{\text{solid}} \quad (3.1)$$

with:

- ρ_{mixture} = density of mixture (kg/m³);
- ρ_{water} = density of water (kg/m³);
- ρ_{solids} = solid density (kg/m³);
- C_{solid} = dry solid concentration (kg/m³)

For the deflocculant mixture, 125 ml Calgon is used in 1 liter of water. This equals to (35+7)/8= 5.25 gram solids in 1 liter, or 5.25 g/l (5.25 kg/m³). The density of the water and Calgon mixture at 21.4 °C then becomes: 997.9+[(2520-997.9)/2520]x5.25=997.9+3.2=1001.1 kg/m³.

This latter value is in good agreement with the measured values of the hydrometer in a blank solution of water and Calgon at the Waterproof Laboratory.

Using the density of the mixing fluid, the density of 25 gram sediment in 1 liter water including 125 ml Calgon at 21.4 °C is calculated: 1001.1+ (2650-1001.1)/2650x25= 1016.6 kg/m³.

The first reading of the float after 18 s in a glass with the water Calgon and sediment mixture should be smaller than 1016.6 kg/m³.

3.2.2 Sample preparation

The sample preparation for the Hydrometer tests requires the following steps:

- 1 dry sediment sample of about 100-300 gr on tray in oven (temperature of 150 °C); use larger sample mass for very sandy samples;
- 2 weigh dry sample of:
 - 30 g (mass M_{total}) when % sand is 0-30%;
 - 50 g (mass M_{total}) when % sand is 30-70%;
 - 150 g (mass M_{total}) when % sand is 70-100%;
- 3 put dry sample in measuring glass with 125 ml-deflocculant (Calgon) + 400 ml distilled water; wait minimal 16 hours for complete deflocculation (use ultra-sonic vibration of 3 minutes in water bath if flocs are still present in suspension); We use the reference standard solution of 4.2% (35 g S+ 7 g N in 1 liter of distilled water).
- 4 wash sample over 63- μm sieve using distilled water (with minimum distilled water, maximum 400 ml);
- 5 wash sand fraction in glass; dry and weigh sample (M_{sand});
- 6 put mud-residue into measuring glass and add distilled water until volume of 1 liter;
- 7 mass mud fraction is $M_{\text{total}} - M_{\text{sand}}$; initial mud concentration is $c = (M_{\text{total}} - M_{\text{sand}}) / \text{liter}$ (range of 10 to 25 g/l).

Detailed information on procedures of sedimentation methods can be found in various ISO and NEN-standards and U.S. Geological Survey open-file report 00-358/2005-1001.

3.3 Hydrometer method

3.3.1 General test procedure for particle size

This procedure refers to the left part of Figure 3.1 and is based on ASTM D4221-18, D7928.

- 1 Prepare the sample as in section 3.2.2.
- 2 prepare the basin with water, place the water pump; this brings the basin to a constant temperature.
- 3 make a blank suspension of 1 liter of distilled water, place it in the basin and perform a reading when the temperature is constant; This measurement is for the hydrometer calibration.
- 4 make a blank suspension of 1 liter of distilled water with 4.2% solution of Calgon, place it in the basin and perform vertical level reading when the temperature is constant; This measurement is for the hydrometer calibration.
- 5 shake/stir mud fraction in glass cylinder for 15 minutes;
- 6 perform hydrometer test with vertical level readings at 18, 40 s, 2, 5, 15, 30 min, 1, 4, 24 hours;
 - a insert the hydrometer directly after the shaking and start the timer
 - b leave the hydrometer in the tube for the first 3 readings (max 2 min)
 - c carefully take out the hydrometer (minimal disturbance), rinse with distilled water and place in the blank suspension to keep it at the desired temperature
 - d insert the hydrometer 30 s before each reading moment (with minimal disturbance)
 - e repeat step 6.c and 6.d until all readings have been done;
- 7 compute settling velocities $w_{s,i} = h_i/t_i$ with $h_i =$ distance between fluid surface and middle of float at time t_i (based on calibration curve). Use the calibration sheet of the hydrometers to work out the results;
- 8 Determine the cumulative mass distribution:
 - a. the density reading at time t is converted into a mass of sediment M_s suspension using the equation:
$$\rho_{\text{suspension}} + C_{\text{hydro}} + C_{\text{men}} - C_{\text{calgon}} = (\rho_{\text{water}} + C_{\text{hydro}} + C_{\text{men}}) + [(\rho_s - (\rho_{\text{water}} + C_{\text{hydro}} + C_{\text{men}})) / \rho_s] M_s / V \quad (3.2)$$

with
 ρ_s = density of sediment = 2650 kg/m³;
 ρ_{water} = density of water at temperature during the test (water without Calgon);
 C_{calgon} = density difference due to Calgon (≈ 3 kg/m³),
 C_{men} = density difference related to inaccuracy of meniscus (small pos. value),
 C_{hydro} = density difference related to manufacturing error of float (small pos. or neg. value),
 M_s = mass of fines in suspension, $V =$ volume of settling column (= 1 Liter),
 - b. The mass percentage of the fines is equal to 100% $\times M_s / M_{s,0}$;
 - c. Compute the mass of the fines for all reading, and use this in the combined grain size distribution curve (including the sieve results of the sandy part of the sample)

Remark 1: Various types of commercial floats are available with measuring grades in density (kg/m³=g/L) or directly in gram with a range of 0 to 50 gram for a settling tube of 1 L (Type ASTM152H:E100).

Remark 2: The Hydrometer-procedure has 4 corrections/calibrations:

- 1 reading of water surface (meniscus): C_{men} ;
- 2 effect of Calgon (so that Stokes-diameter can be computed with density of water): C_{calgon} ;
- 3 deviation of float (reading in distilled water at 20 °C should be 998.2 kg/m³): C_{hydro} ;
- 4 effective settling height of the float center to water surface.

Correction 2 is most accurate if a control measurement is done in distilled water and Calgon mixture without sediment and subtracted from the reading in distilled water without sediment.

Correction 4 refers to the effective settling height between the water surface and the gravity center of the float. This calibration depends on the type of float used and should be checked regularly (see Figure 3.5).

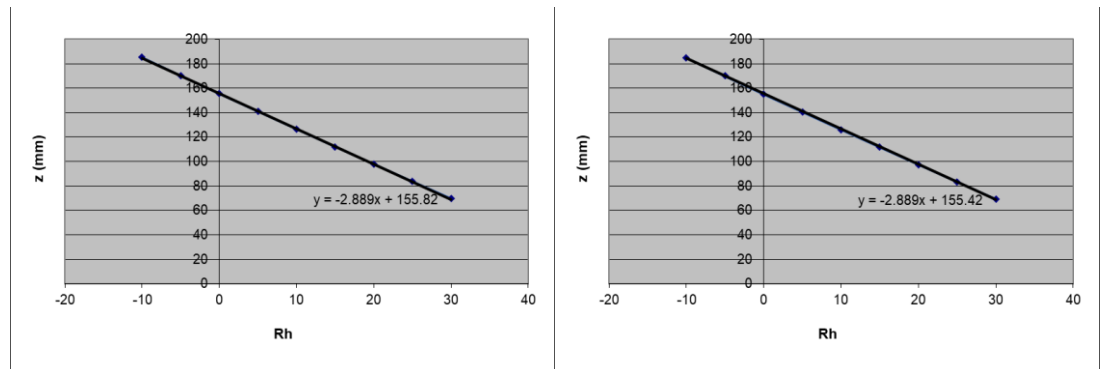


Figure 3.5: Calibration curves for the effective settling heights for each hydrometer relative density markings (Rh) for (left) hydrometer 1 (serial nr. 1535) and (right) hydrometer 2 (serial nr. 1625).

3.3.1.1 Stokes diameter

The diameters are computed from the equation of Stokes for spherical particles, which reads as:

$$\text{Settling velocity: } w_s = (s-1) g D_{\text{stokes}}^2 / (18\nu) \quad (3.3)$$

$$\text{Sediment diameter: } D_{\text{stokes}}^2 = (18\nu w_s) / \{(s-1)g\} \\ D_{\text{stokes}} = [(18\nu) / \{(s-1)g\}]^{0.5} \{w_s\}^{0.5} = [(18\nu) / \{(s-1)g\}]^{0.5} \{H_e/t\}^{0.5} \quad (3.4)$$

with:

D_{stokes} = Stokes settling diameter (m);

w_s = settling velocity (m/s) = H_e/t ;

H_e = effective settling height (m);

t = time (s);

g = 9.81 m/s²;

s = relative density = ρ_s/ρ_m ;

ρ_s = sediment density = 2650 kg/m³;

ρ_m = fluid mixture density (1000 to 1020 kg/m³); use 1010 kg/m³ and $s=2650/1010=2.623$;

ν = kinematic fluid density depending on temperature (m²/s).

3.3.2 General procedure for hindered settling

This procedure refers to the right part of Figure 3.1.

- 1 Take 5 wet subsamples (including sand fraction) with mass of 20, 60, 100, 200 and 400 gram (desired concentrations should be about 10, 30, 50, 100 and 200 g/L).
- 2 Wash each sample in a settling column and add native (sea) water to obtain a volume of 1 liter.
- 3 Take a subsample to check the initial concentration (drying, filtering and weighing).
- 4 Perform hydrometer-test (see above).

These tests can be used to determine the hindered settling velocity as function of concentration. The test with 200 and 400 g/l can also be done without the float by just reading the interface of the sinking sediment surface (as a function of time) in the settling column with respect to the original surface at the start of the test. From these results the hindered settling velocity and the ceiling concentration can be determined.

3.4 TAP-method

3.4.1 General test procedure for particle size

This procedure refers to the left part of Figure 3.1. The initial concentration of the suspension should be relatively low in the range of 500 to 1000 mg/l to prevent rapid blocking of the filter papers.

- 1 Make a dry sample as given in section 3.2.2; weigh the sample; mix the dry sample and take a representative subsample of 2 gram;
- 2 put the dry sample of 2 gram in measuring glass with 125 ml-deflocculant (Calgon) + 400 ml distilled water; wait minimal 16 hours for complete deflocculation (use ultra-sonic vibration of 3 minutes in water bath if flocs are still present in suspension); We use the reference standard solution of 4.2% (35 g S+ 7 g N in 1 liter of distilled water).
- 3 wash sample over 63- μ m sieve using distilled water (with minimum distilled water, maximum 400 ml);
- 4 put mud-residue into measuring glass and add distilled water until volume of 2 liter;
- 5 mass mud fraction is $M_{\text{total}} - M_{\text{sand}}$; initial mud concentration is $c = (M_{\text{total}} - M_{\text{sand}}) / 2$;
- 6 Record the initial height of the water limit in the pipe on the corresponding measurement form "Measurement form settling speed". Prepare 100 ml graduated cylinders. These measuring cylinders must be numbered from 1 to 9. Also prepare aluminum containers with filter papers in them. Number both and weigh the filter papers 5 times. Record the weight on the measurement form.
- 7 Mix everything in the tube well with the stir bar (stick with pat flat at the end with holes) by moving up and down. Do that for exactly 1 minute. And make a note of the start time (after stirring is stopped) of the measurement once mixing is complete.
- 8 After mixing, once the mixing rod is out of the tube, sample 1 of approximately 30-50 ml can be taken in the small numbered graduated cylinders (100 ml) by opening the stopcock at the bottom of the tube. At the same time, the stopwatch must be turned on. (Hold the tube firmly, this requires two people). Record the new height of the water limit and the water volume of the measuring cylinders on the measurement form.
- 9 10 seconds before taking a new sample, shortly rinse the TAP by taking a sample of max 5 ml. This sample goes into the residue bucket.
- 10 Take all samples as explained, and make sure to rinse before each sample. Follow the times on the measurement form ($t = 0, 2, 5, 15, 30, 60, 240, 1440$ minutes; no more than 8 samples to minimize eddy generations in the settling column) at which samples must be taken and keep the time in the correct holes. The times can be extended if the final concentration is still relatively high.
- 11 After taking the samples they can be filtered (see filtration procedure). Make sure that the correct samples in the measuring cylinders end up with the correct filter papers!

Remark: If the both the Hydrometer-method and the TAP-method are used for the same sample; the initial concentration for the TAP-method can be made by taking a subsample from the high-concentration suspension prepared for the hydrometer-test; this subsample must be diluted to obtain a concentration of the order of 500 to 1000 mg/L;

3.4.2 General test procedure for flocculated settling velocity in native (sea) water

This procedure refers to the right part of Figure 3.1.

- 1 Take 5 wet subsamples (including sand fraction) with mass of 1, 2, 5, 10 and 20 gram (desired concentrations should be about 300, 500, 1000, 2000 and 5000 mg/l).
- 2 Wash each sample in a settling column and add native (sea) water to obtain a volume of 2 litres.
- 3 Take a subsample to check the initial concentration (drying, filtering and weighing).
- 4 Record the initial height of the water limit in the pipe on the corresponding measurement form "Measurement form settling speed". Prepare 100 ml graduated cylinders. These measuring

cylinders must be numbered from 1 to 9. Also prepare aluminium-containers with filter papers in them. Number both and weigh the filter papers 5 times. Record the weight on the measurement form.

- 5 Mix everything in the tube well with the stir bar (stick with pat flat at the end with holes) by moving up and down. Do that for exactly 1 minute. And make a note of the start time of the measurement once mixing is complete.
- 6 After mixing, once the rod is out of the tube, sample 1 of approximately 30-50 ml can be taken in the small numbered graduated cylinders (100 ml) by opening the stopcock at the bottom of the tube. At the same time, the stopwatch must be turned on. (Hold the tube firmly, this requires two people). Record the new height of the water limit and the water volume of the measuring cylinders on the measurement form.
- 7 10 seconds before taking a new sample, shortly rinse the TAP by taking a sample of max 5 ml. This sample goes into the residue bucket.
- 8 Take all samples as explained, and make sure to rinse before each sample. Follow the times on the measurement form (t = 0, 2, 5, 15, 30, 60, 240, 1440 minutes; no more than 8 samples to minimize eddy generations in the settling column) at which samples must be taken and keep the time in the correct holes. The times can be extended if the final concentration is still relatively high.
- 9 After taking the samples they can be filtered (see filtration procedure). Make sure that the correct samples in the measuring cylinders end up with the correct filter papers!

Remark: These tests should also be done on a sample without the sand fraction (removal of the sand fraction by using wet sieving over 63- μ m sieve) before washing the sample into the settling column. Using on this approach, the effect of the sand fraction on the flocculation process can be studied.

The TAP-method involves the preparation of an initial concentration from a wet subsample. The inaccuracy of the initial mud concentration has been tested by a series of laboratory tests, as follows:

- 1 preparation of various concentration samples using the same wet sediment mass in a volume of 1 L; subsamples were taken by pipet of 15 ml and 10 times repeated resulting in concentrations in the range of the range of 590 to 710 mg/l; mean concentration= 650 ± 60 mg/l (inaccuracy of about 10%);
- 2 preparation of concentration samples using the same wet sediment mass in a volume of 1 L; each sample was poured in a settling column and concentration was determined from TAP subsamples (about 20 mL) and 10 times repeated resulting in concentrations in the range of 500 to 700 mg/l; mean concentration= 610 ± 100 mg/l (inaccuracy of about 15%).
- 3 The preparation of initial concentrations from wet subsamples is fairly inaccurate with values up to 15% due to the many handlings involved.

3.4.3 Filtration and analysis procedure

- 1 Number nylon filters (with ballpoint pen) and weigh empty oven-dried filters just before the test
 - Weigh 3 times to minimize weighing error
- 2 Place pre-numbered filter paper in filter setup
- 3 Carefully pour the sample from the graduated cylinder into the filtering unit. Rinse the cylinder and filter unit with spray bottle (if necessary)
- 4 Switch on the vacuum pump
- 5 When the filter is dry, put the filter paper with sludge in the oven to dry (at 50 ° C) for at least 20 min (longer if needed)
- 6 Weigh filter paper immediately after removal from oven
 - Weigh 3 times to minimize weighing error
- 7 Work out the results in a table, for determination of sand and silt concentration.

An example computation of the settling velocity is given in Table 3.2.

Table 3.2: Example data of settling test of TAP-method.

Sample	Sample time after start settling process (seconds)	Settling height from surface to TAPping point (mm)	Settling velocity (mm/s)	Mud concentration (mg/liter)	Mass percentage smaller than (%)
1	5	261	$\cong 100$	2634 (initial concentration)	100
2	60	250	4.17	2212	$2212/2634 \times 100 = 84.0$
3	180	236	1.31	1180	$1180/2634 \times 100 = 44.8$
4	300	225	0.75	754	$754/2634 \times 100 = 28.6$
5	600	211	0.35	481	$481/2634 \times 100 = 18.3$
6	1800	200	0.11	262	$262/2634 \times 100 = 10.0$
7	3600	186	0.05	174	$174/2634 \times 100 = 6.6$
8	7200	170	0.024	107	$107/2634 \times 100 = 4.1$

4 Particle size measured by Laser Diffraction and Sedimentation method

4.1 Introduction

Various instruments have been used to determine the particle size distribution of mud-sand samples, as follows:

- Laser-Diffraction method (Malvern 2000 operated by University of Utrecht);
- Sedimentation method-Sedigraph for fine fraction < 63 μm ; sand fraction separated by washing over sieve of 63 μm , dried and sieved to determine size distribution of sand fraction (operated by Soil Mechanics Laboratory of Wiertsema, Tolbert, NL);
- Sedimentation method-Hydrometer for fine fraction < 63 μm ; sand fraction separated by washing over sieve of 63 μm , dried and sieved to determine size distribution of sand fraction (operated by Laboratory of WaterProof, Lelystad, NL);
- Sedimentation method-TAP for fine fraction < 63 μm ; sand fraction separated by washing over sieve of 63 μm , dried and sieved to determine size distribution of sand fraction (operated by Laboratory of WaterProof, Lelystad, NL).

Mud-sand samples were taken from tidal flats, banks and channels in The Netherlands, Belgium, United Kingdom and Bangladesh and stored in (base) containers at a low temperature in the Laboratory of WaterProof BV in Lelystad (NL).

Small subsamples (100 to 200 g) were taken from the base containers after thorough mixing of the material in the base containers. The subsamples were stored in closed bottles and transferred to the laboratories of University of Utrecht and Soil mechanics Wiertsema in Tolbert (Groningen) for analysis of particle size, mineralogy, plasticity (Atterberg limits) and yield stress.

4.2 Description of samples

4.2.1 Physical characteristics

MUSA-samples

WS-Bath-APP: Sandy sample from the intertidal bank site Appelzak about 5 to 10 km south of Bath along the northern side of the Western Scheldt Estuary (NL), Figure 4.2

WS-Bath-BAPU: Muddy sample from the intertidal deposits between two short groins of the pumping station outlet near Bath along the northern side of the Western Scheldt Estuary (NL), Figure 4.2

WS-PA1: Muddy sample from the intertidal deposits in the small harbour basin near Paal along the southern side of the Western Scheldt Estuary (NL), Figure 4.2

WS-SO3: Sandy sample from the intertidal bank near Oosterweel (Antwerp, Belgium) along the left side of the Scheldt river ending in the Western Scheldt Estuary (BE), Figure 4.2

NPZ-B5: Muddy sample from the intertidal deposits in the small harbour basin of Noorpolderzijl at the end of a long tidal channel to the Wadden SAea (NL), Figure 4.1

- NPZ-H2:** Muddy sample from the intertidal deposits in the small harbour basin of Noorpolderzijl, Figure 4.1
- PLUK1, 4:** Muddy-Silty samples from the intertidal banks of the Plymouth Estuary (UK), Figure 4.3
- PLUK2:** Sandy sample with gravel and shells from the intertidal banks of the Plymouth Estuary (UK), Figure 4.3
- BB2:** Silty sample from the seabed in Bengal Bay near the coast of Bangladesh, Figure 4.4
- BB3:** Silty sample from the loadbox of the hopper dredger (JDN) near navigation channel in Bengal Bay near the coast of Bangladesh, Figure 4.4

Additional JDN-bed samples

BBB1-21 Additional set of 20 bed material samples from Bengal Bay (Bangladesh) supplied by dredging contractor JDN for analysis of particle size composition

Sample characteristics are shown in Table 4.1.

Table 4.1: Characteristics of samples.

Location	Sample	% sand > 63 μm	% silt 10-63 μm	% fines < 63 μm	% Cal.
Noordpolderzijl channel (NL)	B5	33	43	67	22
	H2A	13 ($d_{50}=85 \mu\text{m}$)		87	<5
	H2B	14 ($d_{50}=85 \mu\text{m}$)		86	<5
Westerschelde and Zeeschelde (NL, BE)	PA1	9	47	91	30
	BAPU	29	42	71	23
	APP	86	9	14	10
	SO3	61	26	39	20
Plymouth Estuary (UK)	PLUK1	11	64	89	7
	PLUK4	8	68	92	10
Bengal Bay, Bangladesh	BB2	8	64	92	10
	BB3	9	64	91	11
Bengal Bay, Bangladesh	BBB1-21	5 to 25	25 to 35	75 to 95	< 5

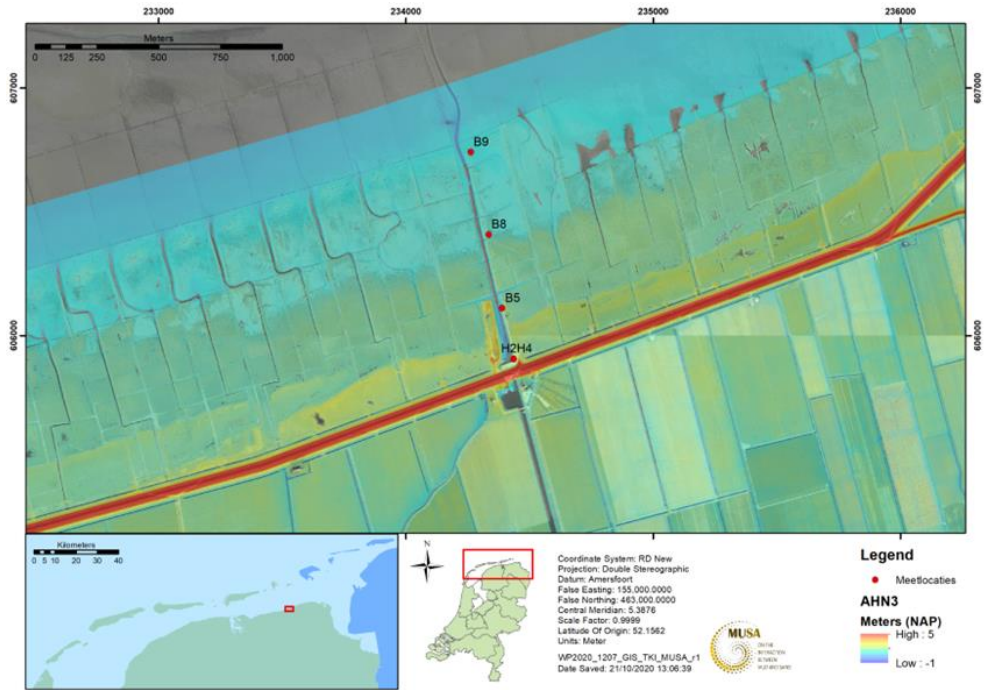


Figure 4.1: Tidal field site Noordpolderzijl (The Netherlands) along the Wadden-Sea

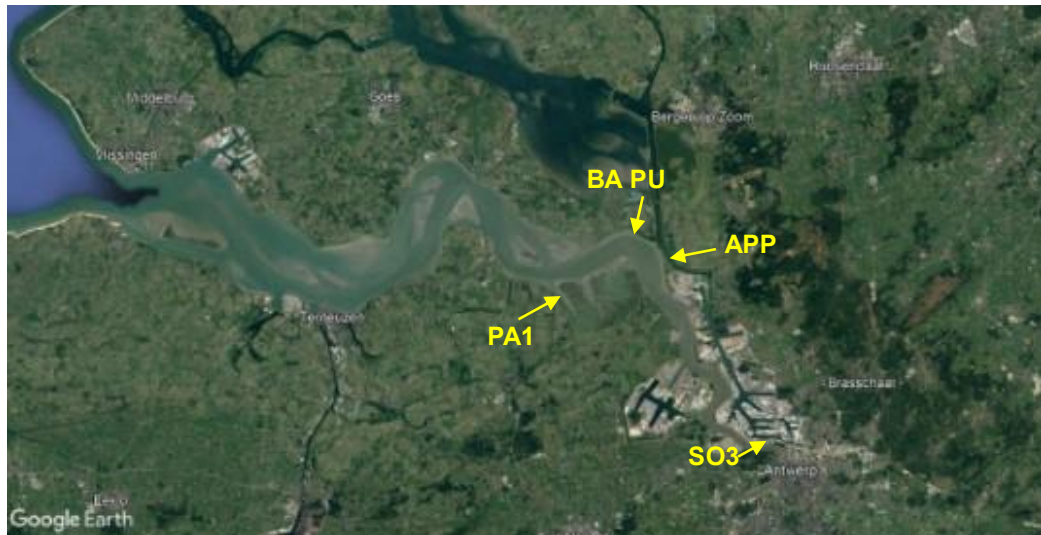


Figure 4.2: Tidal field site Westerschelde (The Netherlands) and Zeeschelde (Belgium)

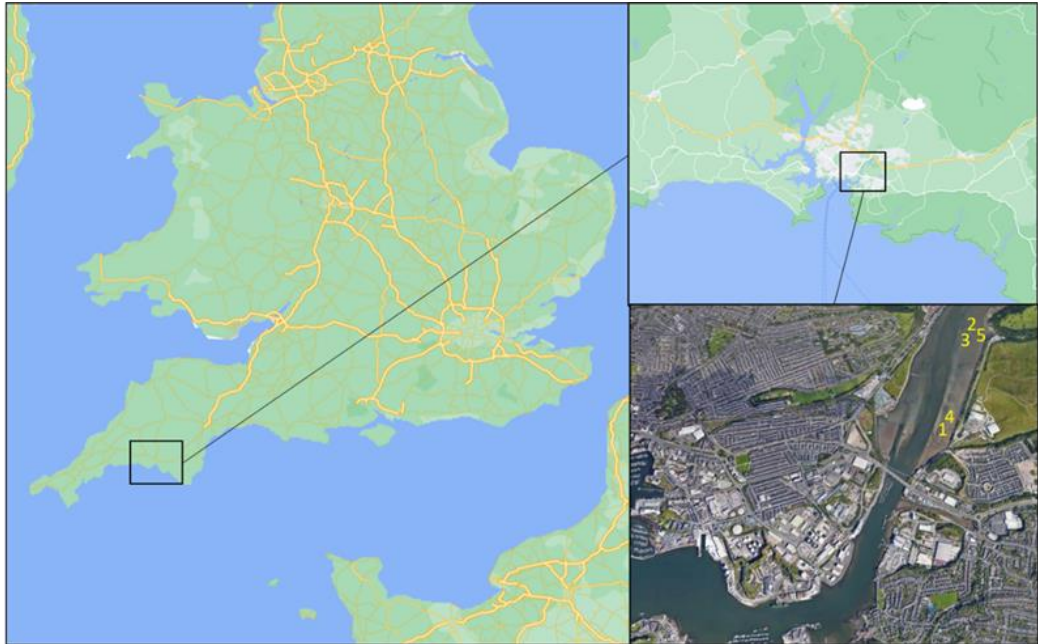


Figure 4.3: Tidal field site (PLUK 1,2,3,4,5) Plymouth Estuary (UK)

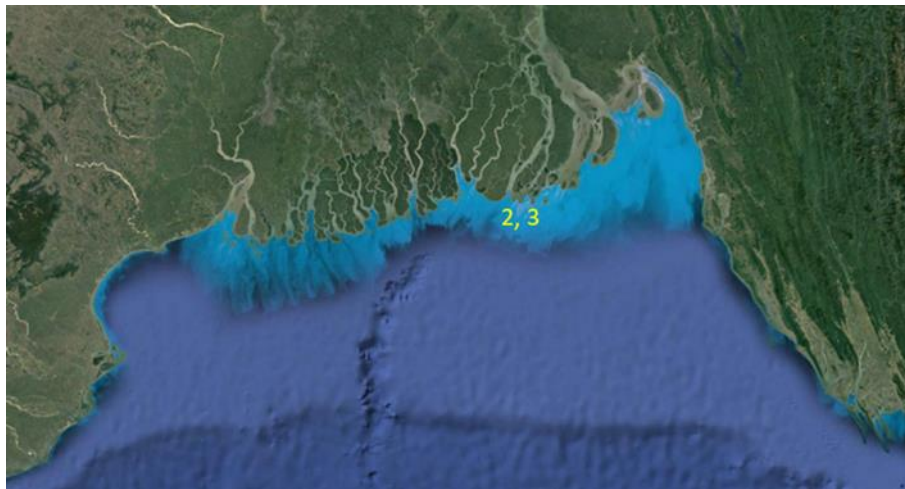


Figure 4.4: Tidal field site (BB 2,3; BBB1-21) Bengal Bay (Bangladesh)

4.2.2 Mineral composition

4.2.2.1 General

The properties of clays are (to some extent) related to the physico-chemical characteristics of the clay minerals and the relative proportions of the minerals in the soil (Skempton, 1953). Clay can be defined as a naturally occurring material, composed mainly of fine-grained minerals ($< 8 \mu\text{m}$), it becomes plastic in presence of water and becomes hard when dried or fired. Clay can be distinguished from other fine-grained soils on the basis of size difference and mineralogy. Clay mixtures may contain organic materials (peat, muck) and calcareous materials (aragonites). The plasticity of clays is due to its particle size, geometry as well as content of water and become hard, stiff, coherent and non: plastic upon drying or firing. Plasticity and hardness are greatly affected by the chemical composition of the material present in the clay. Clays can be molded in any form when they retain water.

Clay particles are formed due to the long-term weathering and erosion of rocks containing soil, ceramic clays, clay shales, glacial clays and the mineral group feldspar. During weathering, the content of feldspar is distorted by hydrolysis process results in formation of clay minerals such as kaolinites and smectites (Kumari and Mohan, 2021). Clay can incorporate one or more clay

minerals even in presence of minute quantities of quartz (SiO₂), metal oxides (Al₂O₃, MgO etc.) and organic matter.

Clay consists primarily of clay minerals, but other minerals like quartz and feldspar may also be present, see Table 4.2. Clay minerals basically are hydrated aluminium phyllosilicates with a layered (phyllo) structure in which aluminium can be replaced by magnesium or iron other alkali-metals (kalium, lithium, cesium) resulting in varying chemical compositions. Clay minerals have the capacity to hold water and can be identified by using the method of Röntgen-diffraction. Diffraction occurs because the wave lengths of Röntgen-beams (some Å) are of the same order of magnitude as the distances between the atoms in the crystalline structures. The Röntgen-beams are reflected via the parallel atomic layers in a mineral under different diffraction-angles resulting in peaks in the diffractogram and each mineral has its own set of specific diffraction peaks. Common clay minerals belonging to the group of layered silicate minerals are Kaolinite ($\cong 7\text{Å}$) and Illite ($\cong 10\text{Å}$) and a group of 14Å-clay minerals consisting of Montmorillonite, Vermiculite and Chlorite. These latter minerals can be identified by heating which has a different effect on each mineral or by using glycerol leading to swelling (from 14 to 18 Å) of only Montmorillonite. Kaolinite, illite and montmorillonite often occur in nature as intermixed minerals in the soil. Kaolinite is a soft, white mineral produced by chemical weathering of silicate minerals like feldspar. It has a low shrink-swell capacity in contact with water. Similarly, Illite is a non-expanding (non-swelling) layered silicate mineral. It occurs as aggregates of small to grey crystals produced by chemical weathering of other silicate minerals like feldspar. Illite is the most common clay mineral. Montmorillonite is a subclass of the smectite minerals consisting of plate-shaped particles. The individual crystals of Montmorillonite and water can easily intervene causing this clay to swell (increase of volume due to absorption of water). Chemically, it is a sodium (Na)-Calcium (Ca) based clay. Bentonite is a typical example of Montmorillonite clay, which is used as a viscous mud slurry in soil drilling industry (for cooling of drilling equipment and removing of drilling solids). Quartz mineral is a pure silicon dioxide, which is an oxide of silicon and also known as silica (chemically inert; non-reactive), most commonly found in nature as sand, gravel, boulders and granite. Silica flowers with spherical type of particles with sizes between 5 and 50 µm and are produced by grinding of quartz particles. Feldspar, mica (glimmer), muscovite are quartz-type minerals consisting of aluminium silicates including calcium, sodium and potassium accounting for about 60% of exposed rocks (granite). Feldspar is the most abundant mineral group. Micas are a group of minerals that can be easily split into extremely thin elastic plates. Muscovite is the most common form of mica.

Table 4.2: Summary of clay minerals.

Clay mineral group	Layer thickness and swell	Composition
Kaolinite	$\cong 7\text{ Å}$ no swell	soft white mineral produced by weathering of aluminium silicate like feldspar; strong hydrogen bonding between layers hindering intrusion of water molecules accounting for non-swelling character; when moistened, the plates adhere causing cohesiveness; bonds are weak enough to allow plates to slip when molded
Illite	$\cong 10\text{ Å}$ no swell	non-expanding mica-type (very platy; dioctahedral micaceous) mineral produced by weathering of feldspar and muscovite, dominant in argillaceous rocks. Named after the state of Illinois, USA. Widely distributed in marine sediments. It can alter to Montmorillonite in humid conditions with higher swelling capacity
Vermiculite	$\cong 14\text{ Å}$ swell	group of phyllosilicates which can undergo substantial expansion when heated; produced by weathering of rocks
Smectite	14 Å swell	group of minerals of which Montmorillonite is the most important one; large chemically active surface area; Montmorillonite is very fine

		(1 μm) and has high swelling capacity because the individual crystals are not tightly bound and water can intervene causing swell; plates adhere strongly when wet and improves plasticity of mixtures; Bentonite is a swelling clay consisting mostly of Montmorillonite; Bentonite is produced by weathering of volcanic ash in seawater which converts the volcanic glass in the ash to clay mineral; ability to absorb large volume of water (factor 8)
Chlorite	≅14 Å no swell	group of phyllosilicates with insertion of Mg, Fe, Ni, Mn, Zinc, Lithium, Calcium; the plates are flexible, but not as elastic as the plates of mica

MUSA samples

Table 4.3 shows the mineralogical properties of MUSA samples found by University of Utrecht (UU).

UU used the Biscaye method (1965) on magnesium saturated oriented mounts (based on weighted peak heights) to determine the dominant minerals in the sample fraction <10 μm. It is assumed that Smectite (Montmorillonite), Illite, Kaolinite, and Chlorite comprise 100% of this fraction.

Remarks are (see also X-ray diffractograms in Annex A):

- samples (fraction < 10 μm) are nicely grouped by geographic region; samples B5 (Noordpolderzijl), PA1, BAPU, BATH-APP, SO3 (Western Scheldt, Zeeschelde) are very similar; samples PLUK1 and PLUK4 (Plymouth UK) are very similar; samples BB2 and BB3 from Bengal Bay (Bangladesh) are very similar.
- samples (fraction < 10 μm) from the Western Scheldt and Noordpolderzijl (NL, BE) are dominated by Illite (55%); Smectite is very low < 10%; Kaolinite and Chlorite are of same order (about 20%);
- samples (fraction < 10 μm) from Plymouth (UK) are dominated by Kaolinite (50-55%) with a provenance from Cornish granites and Illite (45-50%); no Smectite and Chlorite;
- samples (fraction < 10 μm) from Bengal Bay (BB) are dominated by Illite (80%); Chlorite is much lower (20%) with a provenance from the Himalaya; no Smectite and Kaolinite.

Table 4.3: Mineralogical properties of samples (University of Utrecht). **Yellow**=sandy sample; **Green**=clayey sample; **black**=silty sample.

Location	Sample	% sand > 63 µm	% silt 10-63 µm	% fines < 63 µm	% Ca	Minerology*	
						Minerals detected in full sample (all fractions)	Dominant mineralogy of fraction <10 µm (clay-dominated fraction)
Noordpolderzijl channel (NL)	B5	33	43	67	22	calcite, aragonite, quartz, illite/mica, K-feldspar, 14Å clay, halite	S I K C 8% 55% 9% 18%
Western Scheldt and Zeeschelde (NL, BE)	PA1	9	47	91	30	calcite, aragonite, quartz, illite/mica, K-feldspar, 14Å clay, halite	S I K C 4% 56% 18% 22%
	BAPU	29	42	71	23	calcite, aragonite, quartz, (illite/mica), K-feldspar, 14Å clay, halite	S I K C 8% 55% 20% 17%
	APP	86	9	14	10	calcite, aragonite, quartz, (illite/mica), K-feldspar, 14Å clay	S I K C 8% 55% 21% 16%
	SO3	61	26	39	20	calcite, aragonite, quartz, illite/mica, K-feldspar, 14Å clay	S I K C 10% 55% 21% 14%
Plymouth Estuary (UK)	PLUK1	11	64	89	7	quartz, illite/mica, K-feldspar, kaolinite, 14Å clay, halite	S I K C 0% 45% 55% 0%
	PLUK4	8	68	92	10	quartz, illite/mica, K-feldspar, kaolinite, 14Å clay, halite	S I K C 0% 49% 51% 0%
Bengal Bay, Bangladesh	BB2	8	64	92	10	quartz, illite/mica, plagioclase feldspar, amphibole, 14Å clay (chlorite), halite	S I K C 0% 80% 0% 20%
	BB3	9	64	91	11	quartz, illite/mica, plagioclase feldspar, amphibole, 14Å clay (chlorite), halite	S I K C 0% 81% 0% 19%

*S=Smectite (Montmorillonite); I=Illite; K=Kaolinite; C=Chlorite

*14Å mud is (Smectite, Chlorite)

*Halite is marine salt from the water derived from the bulk sample (dried and crushed).

%Ca= percentage Carbonate materials (in bulk samples); %fines+%sand=100% (OCR samples)

4.2.3 Plasticity (Atterberg limits)

The cohesivity of samples can be expressed in terms of the Plasticity Index based on the Atterberg limits (Atterberg tests).

The Atterberg tests consists of:

- roll test; moisture content (plastic limit) at which a thin thread-type roll can be made without crumbling;
- fall (cone-penetration) test: moisture content (liquid limit) at which penetration of standard cone is 20 mm.

The plasticity index of a soil is the numerical difference between the liquid limit and the plastic limit, which are known as the Atterberg limits. Both limits basically are moisture contents (in %). These limits define ranges in moisture content at which a soil will behave as solid, plastic or liquid materials. Typical PI-ranges are:

- $PI < 30$: low plasticity (sandy soils);
- $30 < PI < 50$: intermediate plasticity (silty/loamy soils);
- $PI > 50$: high plasticity (clayey soils).

Clays with relatively large proportions of Montmorillonite minerals with significant swelling capacity generally have relatively high PI-values, in particular the sodium based Montmorillonite clays (see Figure 4.5). Thus, a very high PI-value (>70) is an indication for the presence of a relatively proportion of Montmorillonite minerals. Clays with high proportions of kaolinite minerals without much swelling capacity generally have low PI-values (see Figure 4.5).

According to Skempton (1953), the type of clay minerals and the relative proportions of the clay mineral in a soil have a major influence on the degree of plasticity of a soil as determined by routine Atterberg tests.

The measured values of the MUSA-samples are given in Table 4.4. Figure 4.5 and Figure 4.6 show the PI-results of MUSA-samples (Table 4.4) as function of the percentage of clay $< 2 \mu\text{m}$ and clayey materials $< 8 \mu\text{m}$. For comparison, the PI-values of Kaolinite, Illite and Montmorillonite-dominated clays and three geological clays found in the UK (Skempton 1953) are also shown in Figure 4.5.

The results are:

- The sandy samples (Bath-APP, SO3 and PLUK2) have relatively low PI-values between 12 and 22.
- The silty samples (PLUK1, PLUK4, BB1, BB3) have PI-values between 15 and 30.
- The clayey samples (Bath-BAPU, PA1, B%) have relatively high PI-values between 45 and 60 in the range of the Montmorillonite (Ca) clays.

Figure 4.6 shows that 8 samples (except the very silty samples BB2, BB3 from Bengal Bay) can be represented by linear trendline: $PI = p_{<8 \mu\text{m}}$ with $p_{<8 \mu\text{m}}$ = percentage of sediment $< 8 \mu\text{m}$.

Gyamera et al. (2014) reported many PI-values in the range of 25 to 35 for very sandy/silty/loamy samples with $p_{\text{clay}<2\mu\text{m}}$ in the range of 10 to 30 from the Ghana coast in Africa (see Figure 4.5).

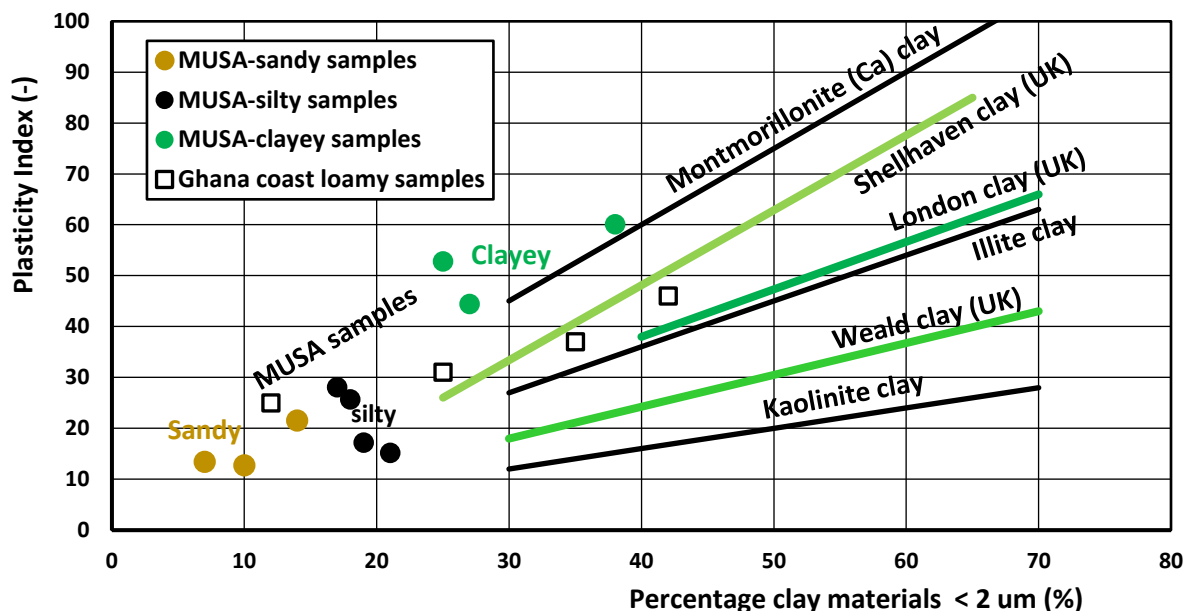


Figure 4.5: Plasticity Index as function of percentage clay ($< 2 \mu\text{m}$)

Table 4.4: Liquid and plasticity limits (Atterberg Limits) and Plasticity Index

Sample	Percentage < 2, 8, 63 μm (%)	Natural water content (%)	Liquid Limit W_L (%)	Plasticity Limit W_P (%)	Plasticity Index $PI = W_L - W_P$ (%)	Classification
WS-Bath-APP dd=1100 \pm 100	7, 12, 24	63	30.6	17.2	13.4	clay; low plasticity
WS-Bath-BAPU dd=670 \pm 30	27, 40, 70	107	68.9	24.4	44.4	clay; high plasticity
WS-PA1 dd=600 \pm 30	38, 60, 95	136	94.3	34.2	60.1	clay; very high plasticity
WS-SO3 dd=850 \pm 30	14, 22, 43	80	48.5	27.0	21.5	clay; medium plasticity
NPZ-B5 dd=600 \pm 30	25, 40, 72	117	83.1	30.3	52.8	clay; very high plasticity
PLUK1 dd=670 \pm 30	17, 29, 87	108	65.6	37.5	28.1	silt; high plasticity
PLUK2 dd=1050 \pm 30	10, 15, 37	58	33.9	21.2	12.7	clay; low plasticity
PLUK4 dd=630 \pm 30	18, 28, 80	119	66.1	40.4	25.7	silt; high plasticity
BB2 dd=1500 \pm 50	21, 42, 88	28	40.9	25.7	15.2	silt; medium plasticity
BB3 dd=1200 \pm 50	19, 35, 94	39	41.3	24.1	17.2	clay-silt; medium plasticity

Natural water content=[(wet density-dry density)/dry density] x 100%

Percentage < 2 μm and Percentage < 8 μm based on SE/HY-methods

Yellow=sandy sample; Green=clayey sample; black=silty sample

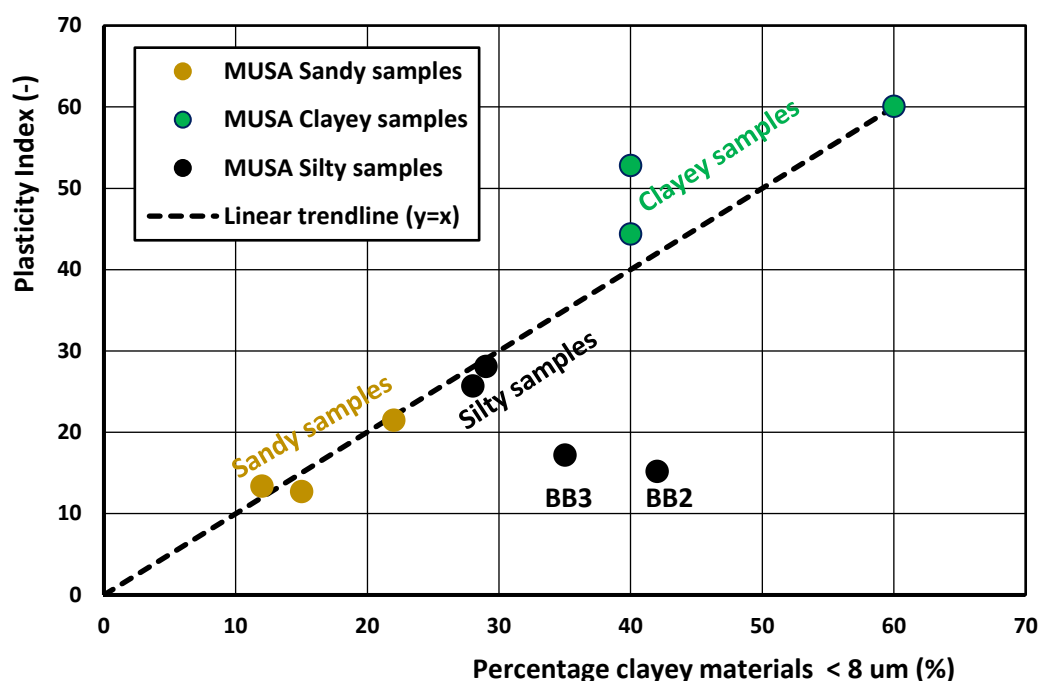


Figure 4.6: Plasticity Index as function of percentage clayey materials (< 8 μm)

4.2.4 Yield stress

A parameter related to the internal resistance of mud-sand samples is the yield stress (undrained shear strength) which can be determined by a vane test in-situ. This test can also be carried out in the laboratory using a small soil sample. The vane is pushed into the soil and the vane is rotated at slow rate (5° to 10° degree per minute). The torque is measured at regular time intervals, see Figure 4.7. If the soil fails, the rotation rate will suddenly increase and the torque will decrease to a lower constant value which is used as an estimate of the undrained remoulded shear strength.

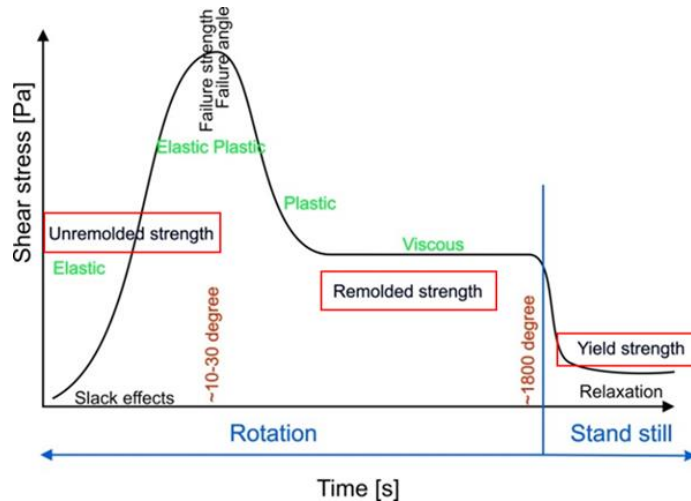


Figure 4.7: Shear stress results of vane experiment

Mud-sand samples under the action of shear (shear rate due to velocity gradients) show three distinct ranges, see Figure 4.8):

- 1 **linear visco-elastic range**; shear stress is linearly related to shear rate ($\tau < \tau_y$ with τ_y =yield stress); viscosity is fully determined by the sediment skeleton (network of interacting particles); below this stress the network remains intact with an elastic-type of behaviour; viscosity is slope of the stress-shear rate curve;
- 2 **yield range**; transition range ($\tau_y < \tau < \tau_{fp}$ with τ_{fp} =flow point stress); sediment skeleton-structure gradually collapses;
- 3 **flow range** ($\tau > \tau_{fp}$); shear is linearly related to shear rate ($\tau = v_{mud} du/dz$ with $v_{mud} = \mu_{mud} / \rho_{mud} =$ kinematic viscosity coefficient of fluid mud, $\rho_{mud} =$ wet bulk density of mud and $\mu_{mud} =$ dynamic viscosity coefficient); viscosity is fully determined by the fluid-sediment mixture.

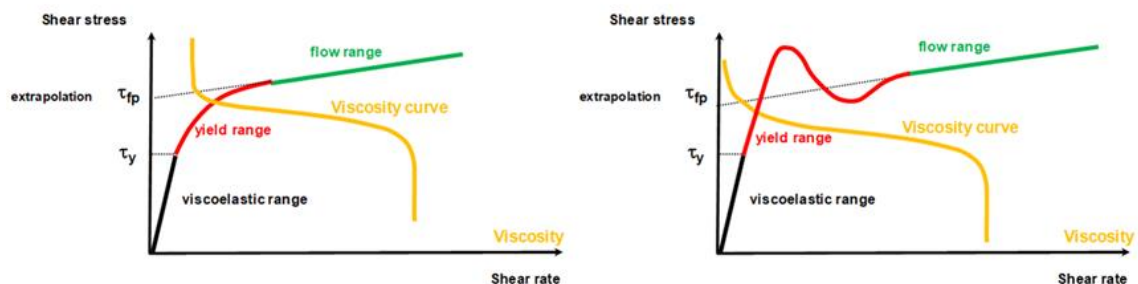


Figure 4.8: Shear stress as function of shear rate (du/dz)

The yield stress is the stress just before initiation of viscous deformation (creep). The yield stress is somewhat smaller (factor 1.5 to 2) than the remoulded shear strength, as found by Locat and Demers (1988) for about 70 types of soils.

The flow point stress is the stress just before initiation of flow and can be determined from a plot of shear stress against shear rates as the intersect of the higher shear stresses (values for shear rates > 10) with the stress axis (y-axis). It is the stress above which the sample behaves as a fluid. The flow point stress is somewhat larger than the undrained remoulded shear strength. The yield stress, flow point stress and elastic properties of the fluid mud are dependent on its stress history.

The yield stress of the MUSA-samples was determined using the Brookfield Rheometer DV3T with a vane-type spindle at the Soil Mechanics Laboratory of Wiertsema (Tolbert, Groningen). The basic test data are given in Table 4.5.

Figure 4.9 shows three typical examples of the measured shear stress versus the measured strain of the sample (output data of Brookfield Rheometer). The yield stress is derived from stress-strain curves.

Figure 4.10 shows the yield stress results of all tests and the envelope curves of the data from the Literature for marine and estuarine muds (Deltares 1991; Wurpts, 2005; Wurpts and Greiser, 2005; McAnally, 2007; Seifert and Kopf, 2010). The yield stress values show an increasing trend for increasing dry density of the mud-sand mixture in agreement with the data envelope curves. The yield stress values of the clayey MUSA-samples with low sand content (<30%) are fairly close together and mostly within the envelope curves. These results are most reliable as the sand particles are well buried within the mud matrix. The yield stress values of the silty samples are in the low range of the envelope curves. Two samples with a relatively high sand content >60% to 70% are significantly above the envelope curves, which may be the effect of high internal friction between the sand particles.

It is noted that the yield stress values for a mixture with dry density of 100 kg/m³ are less reliable as segregation of mud, silt and sand particles may easily occur during the test procedure.

Table 4.5: Yield stress values of MUSA-samples

Type of mud	Wet/Dry density (kg/m ³)	Percentage < 2, 8, 63 μm (%)	Water content= (ρ _{wet-dry} /ρ _{dry} ×100%	Yield stress (N/m ²) at 100, 200, 300 kg/m ³
WS-Bath-APP	1100±100	7, 12, 24	63	2.7; 9.3; 49.2
WS-Bath-BAPU	670±30	27, 40, 70	107	0.9; 3.4; 47.4
WS-PA1	600±30	38, 60, 95	117	0.08; 2.2; 3.1
WS-SO3	850±30	14, 22, 43	80	0.08; 0.16; 2.6
NPZ-B5	600±30	25, 40, 72	136	0,24; 3.1; 21.2
PLUK1	670±30	17, 29, 87	108	0.11; 1; 3.6
PLUK2	1050±30	10, 15, 37	58	0.08; 28.7; 59.8
PLUK4	630±30	18, 28, 80	119	0.1; 0.2; 3
BB2	1500±50	21, 42, 88	28	0.08; 0.24; 0.8
BB3	1200±50	19, 35, 94	39	0.08; 2.3; 9.8

Yellow=sandy sample; Green=clayey sample; black=silty sample

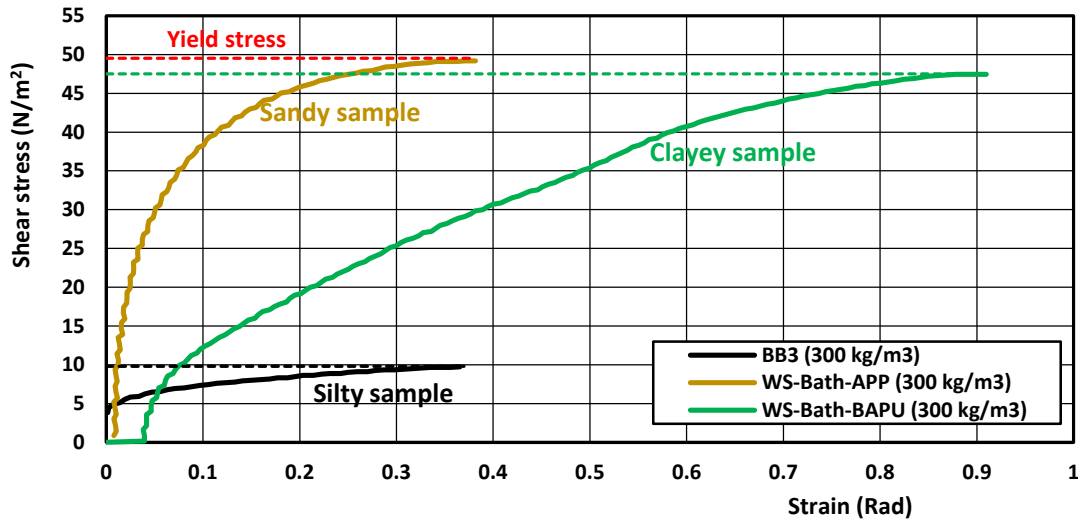


Figure 4.9: Shear stress against Strain for three mud samples (MUSA samples) at dry density of 300 kg/m³.

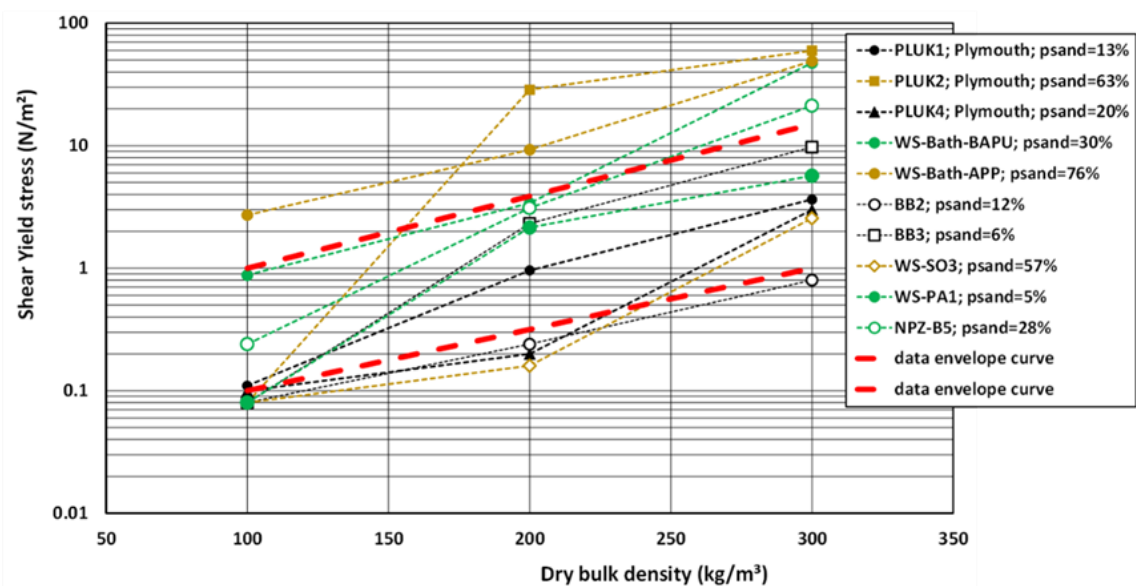


Figure 4.10: Yield stress as function of dry density for MUSA-samples.

4.3 Test results of particle size; MUSA samples

4.3.1 Comparison of Hydrometer-method and TAP-method

Subsamples (H2B) were taken from the base mud sample H2 for performing tests with the Hydrometer-method and the TAP-method. Sample preparation was conform the procedures in Section 3.2.2.

The subsamples used in the TAP-method were taken from the high-concentration suspension made for the Hydrometer test H2B and diluted to obtain the desired initial concentration as given in Table 4.6. The difference between the TAP-concentrations made by dilution and taken immediately after the start of the settling test is maximum 7%, which can be seen as the experimental inaccuracy related to errors caused by dilution, filtering and weighing, etc..

The calculation of the settling velocity and particle size distribution from the TAP-test data have been done by using the initial concentration based on dilution (430 and 388 mg/l), see Table 4.6.

Table 4.6: Initial density and concentrations of Hydrometer-method and TAP-method. Remark: sand was removed by wet sieving over 63 μm .

Sample	Hydrometer		TAP-method	
	Initial suspension density and dry mass fines (< 63 μm) in 1 L	Suspension density after 18 s (first reading)	Initial concentration after dilution	Initial concentration immediately after start of test
H2B	1017.55 g/L (25.795 gram fines)	1014.9 g/L	430 mg/L 388 mg/L	433 \pm 3 mg/L 360 \pm 12 mg/L

The particle size distribution (psd) of muddy/clayey sample H2B of the Hydrometer-method (H-method) and the TAP method are shown in Figure 4.11 for the complete sample (sand fraction included).

Based on Figure 4.11, the following percentages of the clay-dominated fractions are given:

- Hydrometer-method: $p < 2 \mu\text{m} = 36\text{-}38\%$, $p < 4 \mu\text{m} = 40\text{-}46\%$, $p < 8 \mu\text{m} = 50\text{-}54\%$;
- Pipet/TAP-method: $p < 2 \mu\text{m} = 32\text{-}34\%$, $p < 4 \mu\text{m} = 40\text{-}44\%$, $p < 8 \mu\text{m} = 50\text{-}58\%$.

These values are fairly close together indicating that both methods can be used with confidence for the determination of the percentage of clay and very fines.

Figure 4.12 shows the psd for the fine fraction only (< 63 μm ; sand fraction excluded). The results of the Hydrometer-method on the right part of the curve are all below the values of the TAP-method, which is caused by the discrepancy between the first density-reading of the H-method and the known initial density-value, see Table 4.5. This gives a drop of about 10% in the particle size distribution related to the first reading after 18 s, see Figure 4.11 and also Figure 4.12. The size distributions of the H-method in Figure 4.12 indicate that still about 10% of the sample is larger than 63 μm (sand fraction), which is not realistic as the sand fraction was removed by wet sieving over a mesh of 63 μm . This type of drop is not observed in the psd of the TAP-method. A similar drop can be seen in the psd of Black soil (Figure 3.4) of Kaur and Fanourakis (2016) and in the psd of an earlier sample of Noordpolderzijk (Figure 4.13) analyzed in the laboratory of Wiertsema soil mechanics (Tolbert, The Netherlands). The Hydrometer test results of Ibanez Sanz (2018) also show a similar drop (see Figure 2.14 of Doctoral Thesis).

Various causes can be mentioned for the drop related to the initial reading after 18 s, as follows:

- presence of sand particles, which is however very unlikely as the sand fraction was removed by wet sieving over mesh of 63 μm ;
- generation of aggregates (coagulation/flocculation) with relatively large settling velocities due to the intensive mixing to produce a homogeneous high-concentration suspension;
- presence of non-homogeneous suspension despite mixing efforts;
- initial float readings are not (fully) representative for the density of the suspension of water with discrete sediment particles.

It is recommended to neglect float readings within the first 1 to 2 minutes after the start of the test, see dashed line in Figure 4.11. This procedure is also followed by soil mechanics laboratory of Wiertsema (Dijkstra 2020).

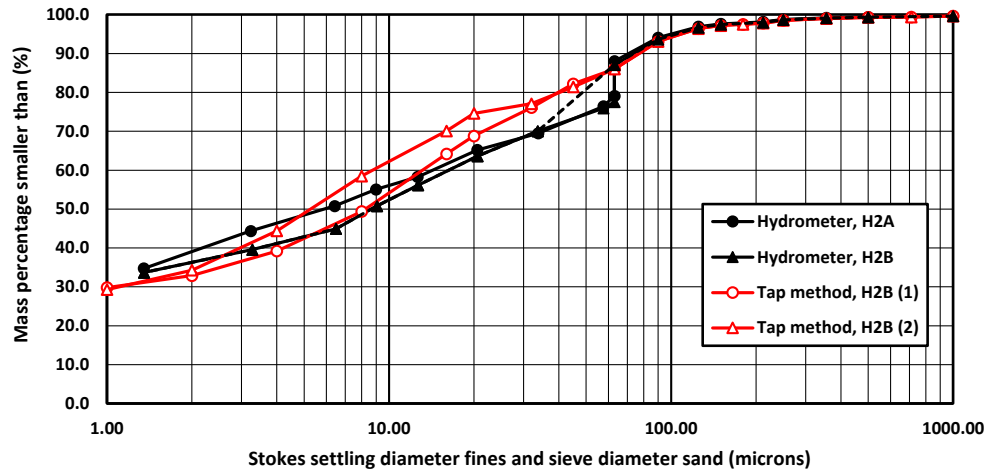


Figure 4.11: Particle size distribution of sample H2 from harbour Noordpolderzijl (2020) based on Hydrometer-method and TAP-method for fine fraction (sieve results of sand fraction included).

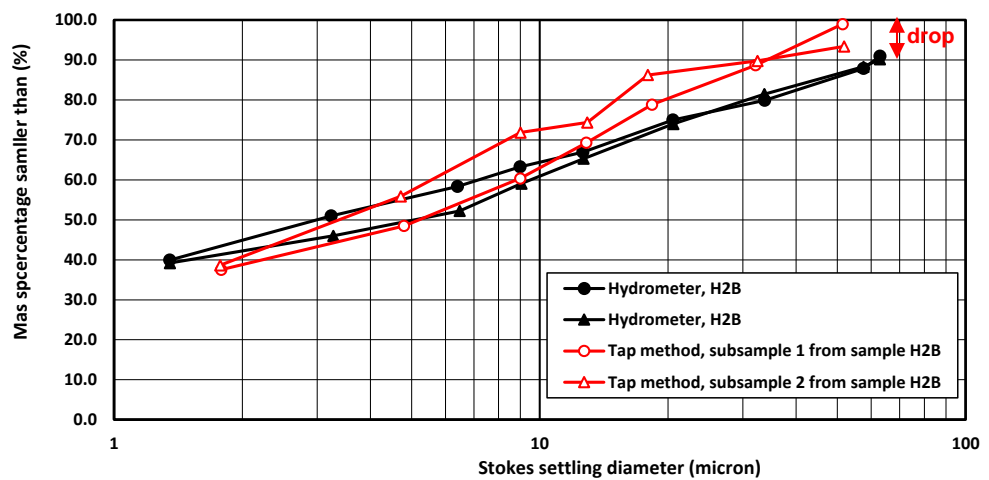


Figure 4.12: Particle size distribution of sample H2 from harbour Noordpolderzijl (2020) based on Hydrometer-method and TAP-method for fine fraction (sand fraction excluded).

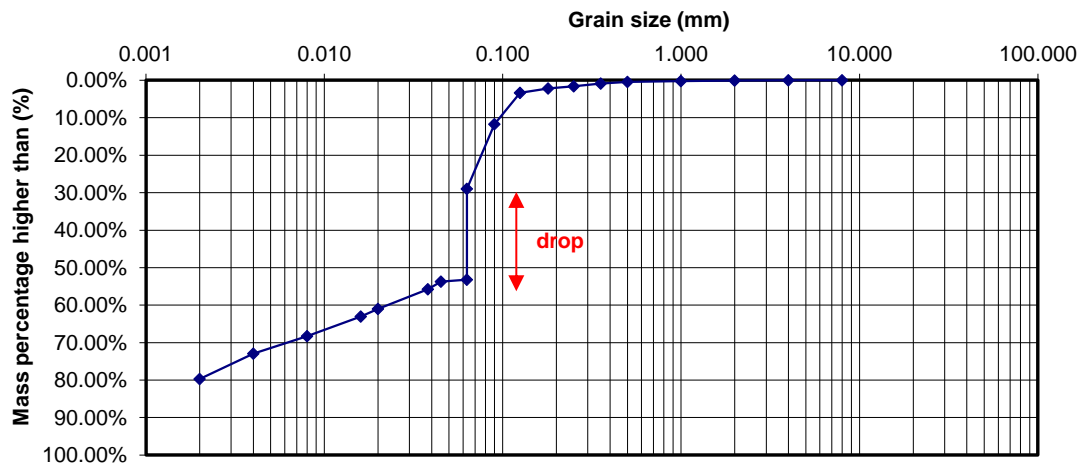


Figure 4.13: Particle size distribution of sample from harbour Noordpolderzijl (2017) based on Hydrometer-method for fine fraction and sieve method for sand fraction (percentage sand > 63 μm = 30%; percentage clay < 2 μm = 20%).

4.3.2 Comparison of LD, Sedigraph and Hydrometer-methods

Sample treatment Laser-Diffraction method (Malvern 2000; University of Utrecht UU)

Two types of samples are tested: bulk samples and OCR-samples (OCR=Organic and Calcareous materials Removed; insoluble residues). Sample treatment is as follows:

- OCR-samples are obtained by treating the sample overnight with 1N HCl and for a few days with hydrogen peroxide until a significant colour change is visible; carbonate and organic matter are removed using this procedure.
- Bulk and OCR samples are suspended in de-ionized water to the sample presentation unit containing 1 litre of deionized water with 25 mL of dispersion agent (44.6 g of Na₄P₂O₇·10H₂O + 4.24 g Na₂CO₃ / 1 L H₂O).
- UU (University of Utrecht) adds as much sample as to obtain an obscuration of the red laser of about 20%. Before each measurement, UU applies 60 s of ultrasounds with a maximum tip displacement of 4 µm.
- grain size distribution is calculated using a refractive index of 1.544 and an absorption index of 0.9; Malvern 2000 uses a blue laser to measure the fine fraction; algorithm then calculates the full grain size distribution

UU has 20 years of experience in carrying out grain size analysis with excellent reproducibility.

Sample treatment Sedigraph and Hydrometer-methods

The test method consists of (NEN-EN-ISO 17892-4 and NEN-ISO 13317- 2001):

- no removal of organic and calcareous materials;
- separation of sand fraction by washing over a sieve of 63 µm; determination of particle sizes of sand fraction by using a standard set of sieves;
- preparation of a high-concentration suspension of the fine fraction (< 63 µm) using distilled water; addition of chemical agent (peptiser) for complete deflocculation (period of 24 hours);
- settling test under controlled conditions in Sedigraph and in Hydrometer;
- combining results of fine fraction and sand fraction for particle size distribution(pdf).

Table 4.7 presents a summary of all results.

Table 4.7: Particle size properties of mud-sand samples.

Sample	Percentage lutum < 2 µm (%)			Percentage clay mat <8 µm (%)			Percentage fines <63 µm (%)			d ₁₀ (µm)			d ₅₀ (µm)			d ₉₀ (µm)		
	LD	SE	HY	LD	SE	HY	LD	SE	HY	LD	SE	HY	LD	SE	HY	LD	SE	HY
Bath-APP dd=1100±100 PI=13	2	10	5	3 (4)	13	10	10 (14)	25	23	15 (30)	2	8	190 (165)	14 4	150	300 (290)	232	200
Bath-BAPU dd=670±30 PI=44	7	30	24	22 (26)	43	37	55 (70)	71	70	3 (2)	<2	<2	40 (27)	15	20	200 (150)	158	125
PA1 dd=600±30 PI=53	8	40	37	32 (38)	64	56	76 (91)	94	93	2.5 (1.4)	<2	<2	20 (13)	3	6	200 (60)	37	50
SO3 dd=850±30 PI=22	4	17	12	8 (12)	24	21	30 (39)	44	42	10 (5.5)	<2	<2	90 (80)	70	85	370 (190)	163	400
NPZ-B5 dd=600±30 PI=60	7	29	22	25 (21)	45	35	65 (67)	74	70	3.0 (2.7)	<2	<2	32 (38)	12	20	160 (120)	92	100
PLUK1 dd=670±30 PI=28	3	19	15	13 (20)	31	27	64 (90)	87	88	5.5 (3.5)	<2	<2	45 (25)	19	40	150 (65)	77	65
PLUK2 dd=1050±30 PI=15	nm	10	nm	nm	15	nm	nm	37	35	nm	2	nm	nm	22 0	230	nm	1570	1800
PLUK4 dd=630±30 PI=26	3	19	17	12 (20)	30	27	65 (90)	84	75	6 (3.5)	<2	<2	50 (25)	21	25	150 (60)	88	90
BB2 dd=1500±50 PI=13	6	21	15	20 (22)	42	33	80 (92)	88	88	3.6 (3.1)	<2	<2	23 (19)	10	13	115 (55)	92	73

Sample	Percentage lutum < 2 μm (%)			Percentage clay mat < 8 μm (%)			Percentage fines < 63 μm (%)			d ₁₀ (μm)			d ₅₀ (μm)			d ₉₀ (μm)		
	LD	SE	HY	LD	SE	HY	LD	SE	HY	LD	SE	HY	LD	SE	HY	LD	SE	HY
BB3 dd=1200±50 PI=17	6	20	17	20 (22)	39	30	82 (90)	95	94	3.5 (3.0)	<2	<2	25 (20)	11	14	95 (60)	36	50

Green=clayey samples; Yellow=sandy samples; Black=silty samples; nm=not measured

Sample treatment: all samples deflocculated; Samples tested by LD: with and without Organic and Calcareous materials Removed (OCR).

LD= Laser Diffraction method of University of Utrecht; bulk samples and OCR-samples (between brackets)

SE= Sedigraph method of soil mechanics laboratory Wiertsema; only bulk samples

HY= Hydrometer method of WaterProof BV; only bulk samples

dd=dry density (kg/m³)= value of the natural sample (top layer of bed) at sampling site

PI= Plasticity Index; d₁₀,d₅₀,d₉₀=diameters of mud-sand sample

Figure 4.14 shows the particle size distribution (pdf) of all samples after removal of organic and calcareous materials and treated with deflocculant agents based on the Laser-Diffraction method. The most important characteristics are:

- samples Bath-APP, SO3 and PLUK2 are fine sandy samples with d₅₀ > 63 μm and percentage of sand > 50%; low PI-values < 30, relatively high dry bulk density >800 kg/m³;
- samples PLUK1, PLUK4, BB2 and BB3 are silty samples with 70% between 8 and 63 μm ; low PI-values < 30, relatively high dry bulk density >800 kg/m³; dry density values > 800 kg/m³;
- samples Bath-BAPU, B5 and PA1 are clayey/muddy sample with percentage of clayey materials (<8 μm) > 40% and relatively high PI-values > 40; dry density values < 800 kg/m³.

Figure 4.15 to Figure 4.18 show the particle size distribution (pdf) of all samples based on the three methods used: Laser-Diffraction (University of Utrecht); Sedigraph (Wiertsema Soils Mechanics Laboratory) and Hydrometer method (WaterProof BV Laboratory).

The most important characteristics are:

- the Sedigraph and the Hydrometer-methods produce significantly smaller d₁₀, d₅₀ and d₉₀-values than the LD-method; the Sedigraph-method produces somewhat smaller values than the Hydrometer-method;
- removal (OCR) of organics and calcareous materials from the samples yields a shift to the left of the pdf-curve with smaller values d₅₀-values; percentage of fines (<63 μm) increases
- LD-method shows relatively large variability between similar samples (bulk1, bulk2, bulk3).

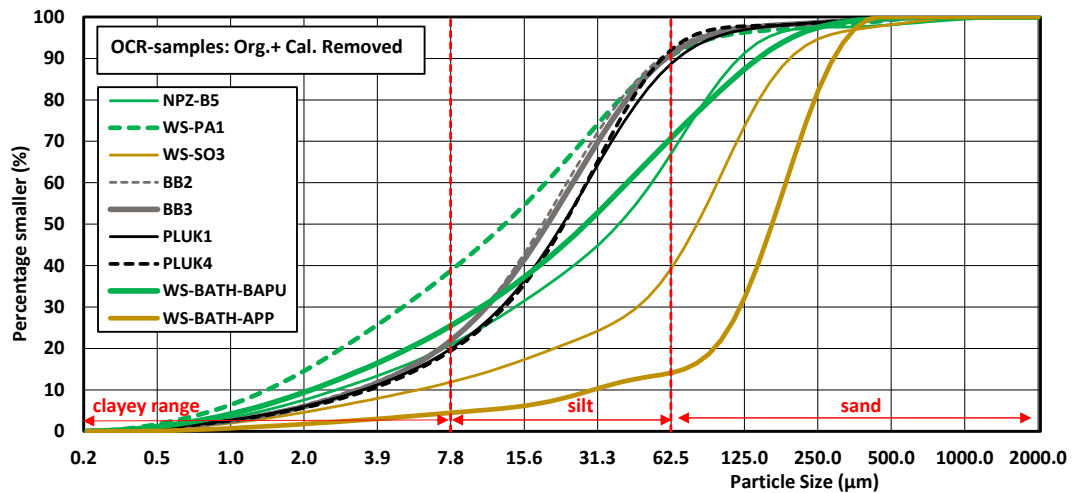


Figure 4.14: Particle size distribution based on LD (deflocculated; organic and calcareous materials removed);

Yellow=sandy; Green=clayey; Black/Grey=silty

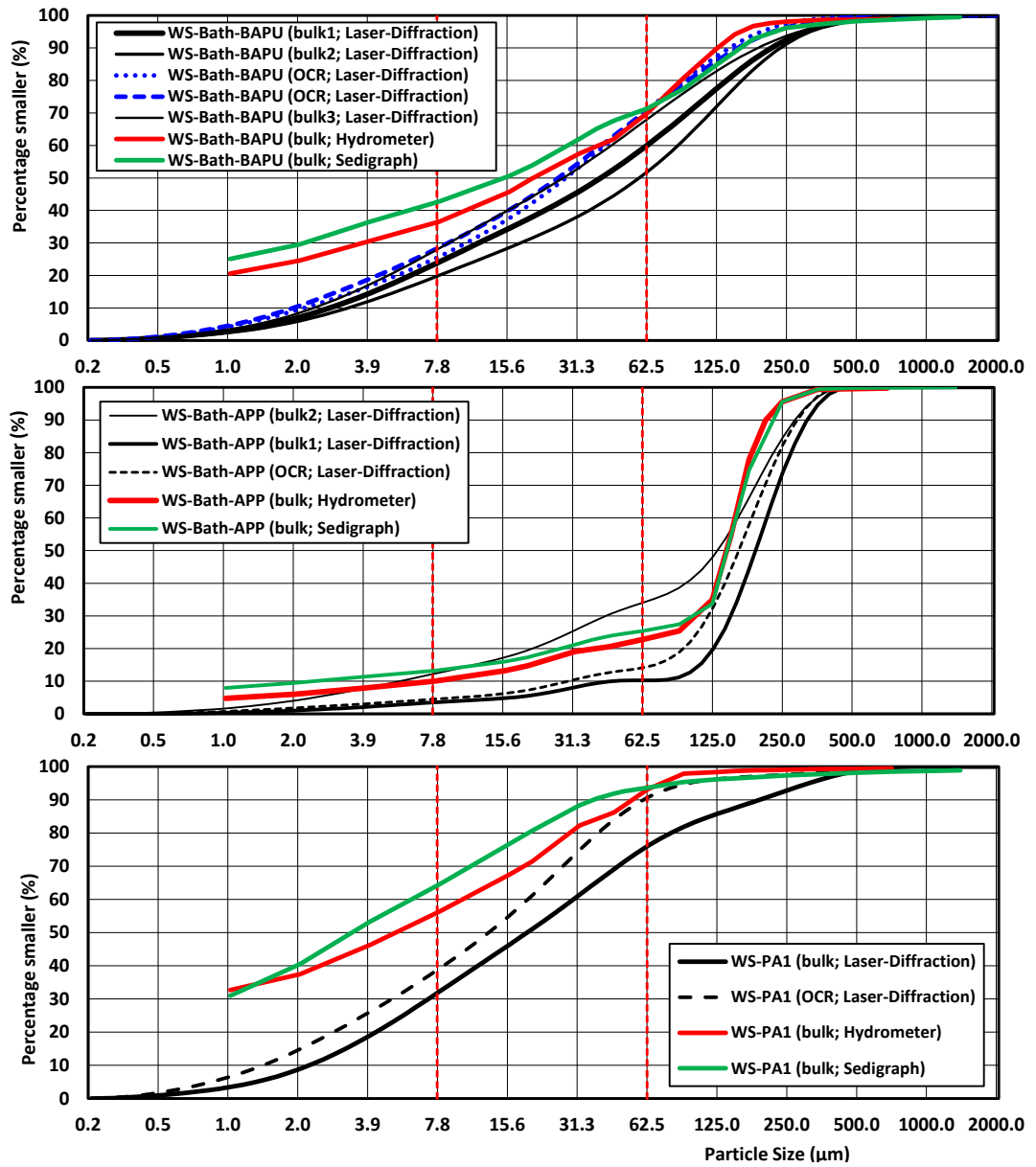


Figure 4.15: Particle size distributions of samples (top) BaPU, (middle) Bath-APP and (bottom) PA1 (Westerschelde, NL).

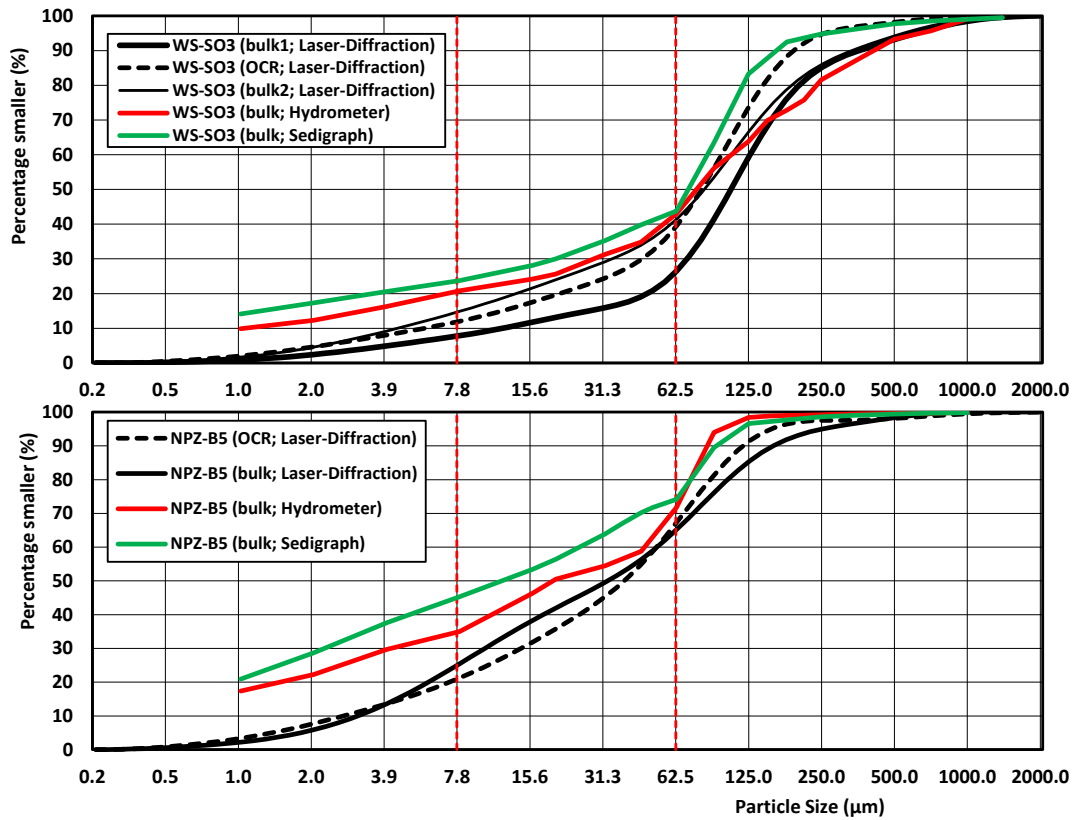


Figure 4.16: Particle size distributions of samples (top) SO3 (Western Scheldt) and (bottom) B5 (Noordpolderzijl).

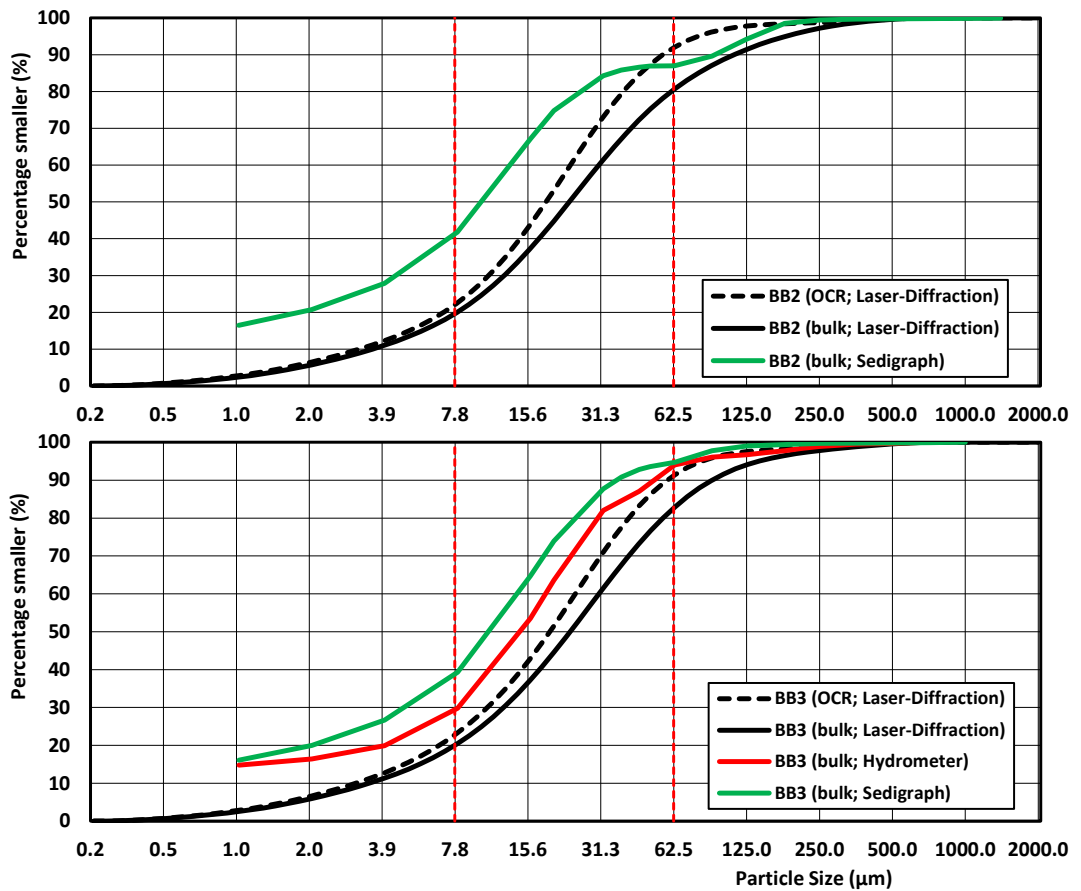


Figure 4.17: Particle size distributions of samples (top) BB2 and (bottom) BB3 (Bengal Bay, Bangladesh).

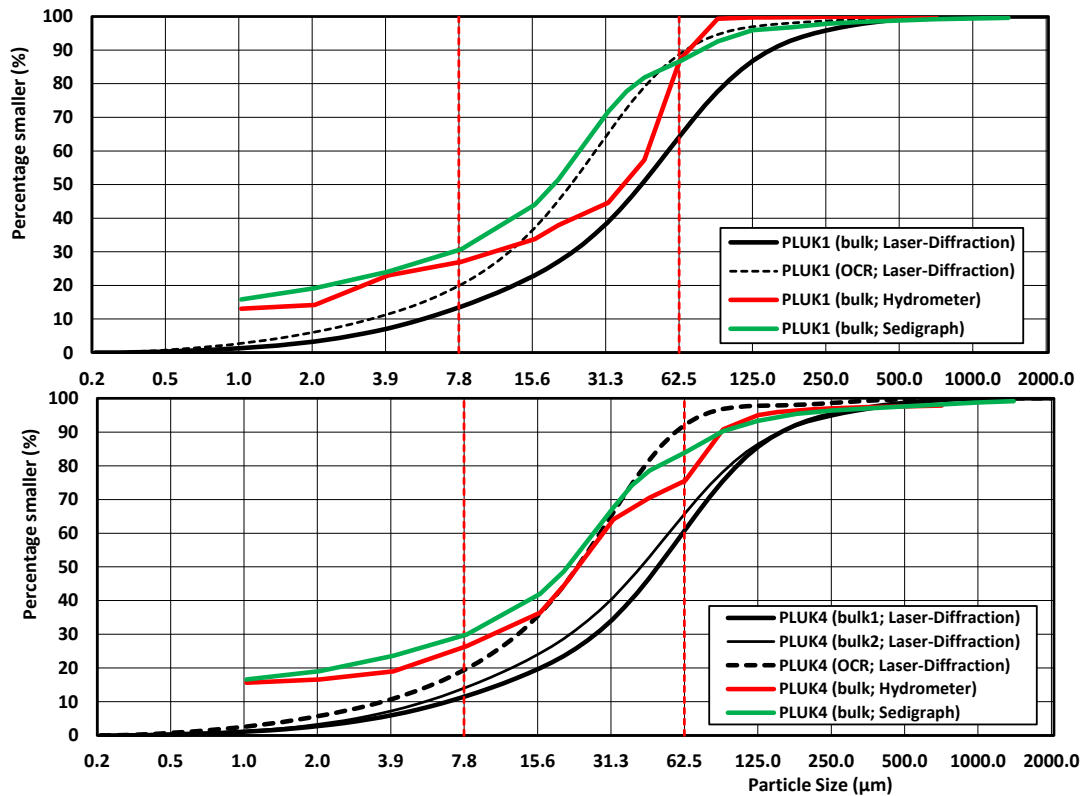


Figure 4.18: Particle size distributions of samples (top) PLUK1 and (bottom) PLUK4 (Plymouth Estuary, UK).

Detailed analysis and comparison of the particle size data shows that (Figure 4.19):

- the percentage of fines < 63 μm based on the Sedigraph and Hydrometer-methods are close together within 15%; the percentage of clayey materials (< 8 μm) is within 25%; the Sedigraph produces the highest percentage of fines;
- the median particle (d_{50}) of the Sedigraph and Hydrometer-methods are reasonably close together within 25% for samples Bath-BAPU, SO3, PLUK4, BB3; the differences are up to a factor of 2 for samples PA1, B5 and PLUK1; the Sedigraph produces the smallest d_{50} -values;
- the median particle (d_{50}) of the LD-method is significantly higher than that of the Sedigraph and Hydrometer for samples with a relatively high percentage of fines (>50%);
- the median particle size (d_{50}) decreases exponentially for increasing percentage of fines < 63 μm (Figure 4.20);
- the ratio of percentage of clayey materials (< 8 μm) and percentage of fines (< 63 μm) is in the range of 0.2 and 0.4 for the LD-method and in the range of 0.4 to 0.6 for the SE/HY-methods (Figure 4.21); very silty and sandy samples have a relatively low percentage of clayey materials;
- the ratio of percentage of Lutum (< 2 μm) and percentage of fines (< 63 μm) is in the range of 0.05 and 0.2 for the LD-method and in the range of 0.2 to 0.4 for the SE/HY-methods (Figure 4.22); very silty and samples have a relatively low percentage of lutum.

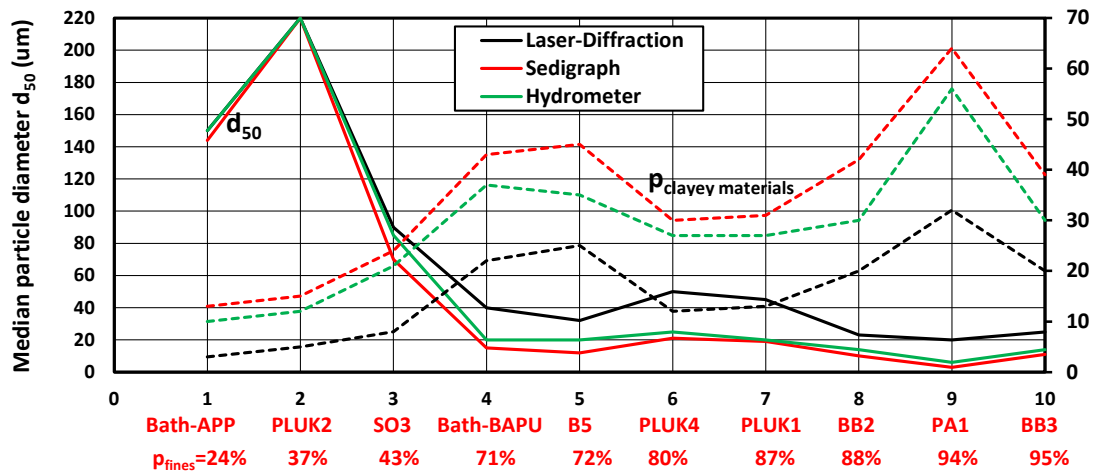


Figure 4.19: Median particle (d_{50}) and percentage of clayey materials ($< 8 \mu\text{m}$) of samples based on three methods: Laser-Diffraction (LD), Sedigraph (SE) and Hydrometer (HY). (p_{fines} =percentage of fines $< 63 \mu\text{m}$ based on average values of SE and HY-methods).

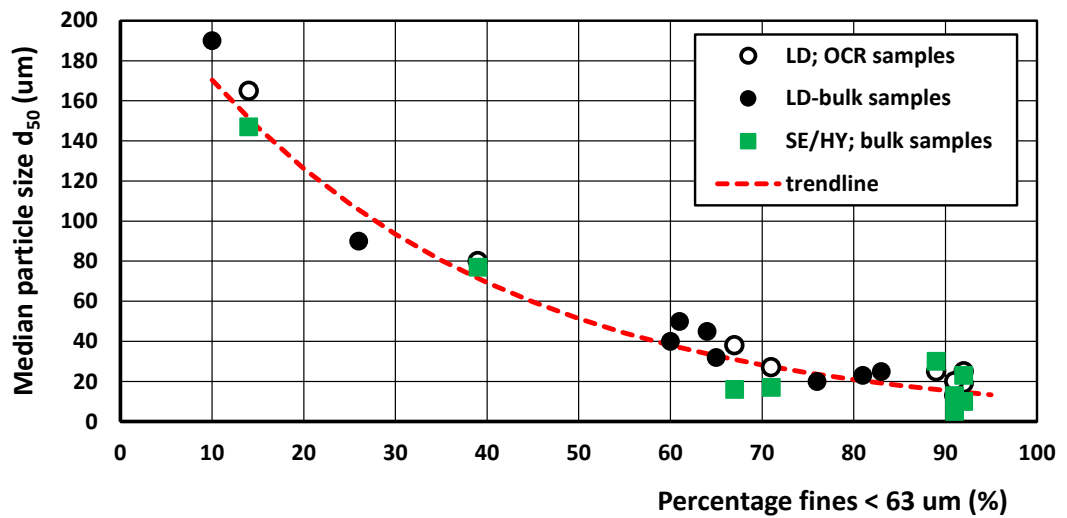


Figure 4.20: Median particle size (d_{50}) as function of percentage of fines $< 63 \mu\text{m}$.

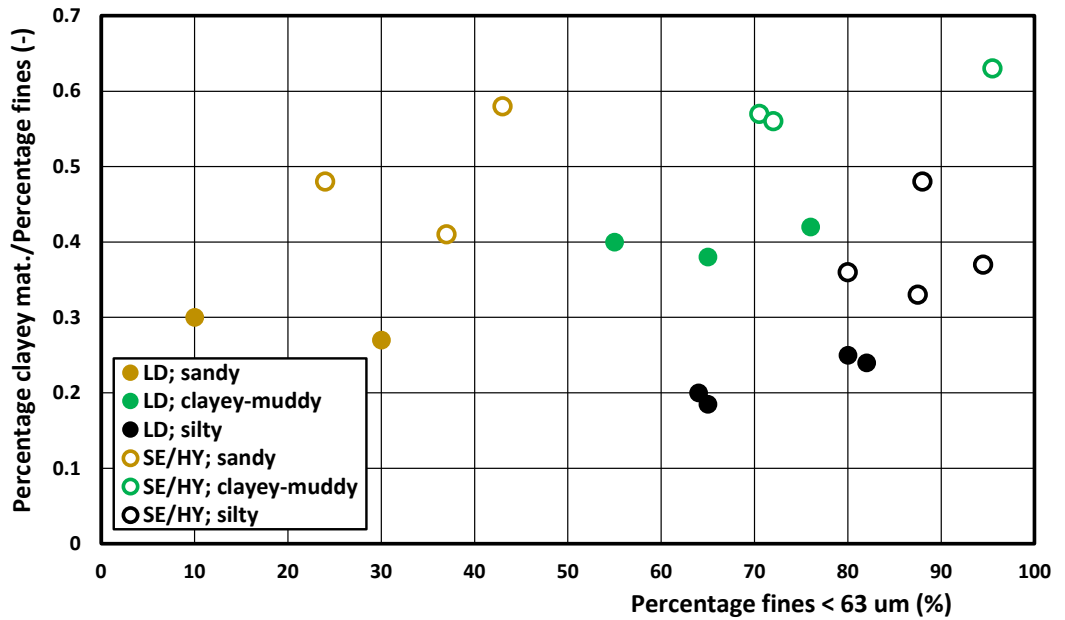


Figure 4.21: Ratio of percentage of clayey materials (< 8 μm) and percentage of fines (< 63 μm) as function percentage of fines.

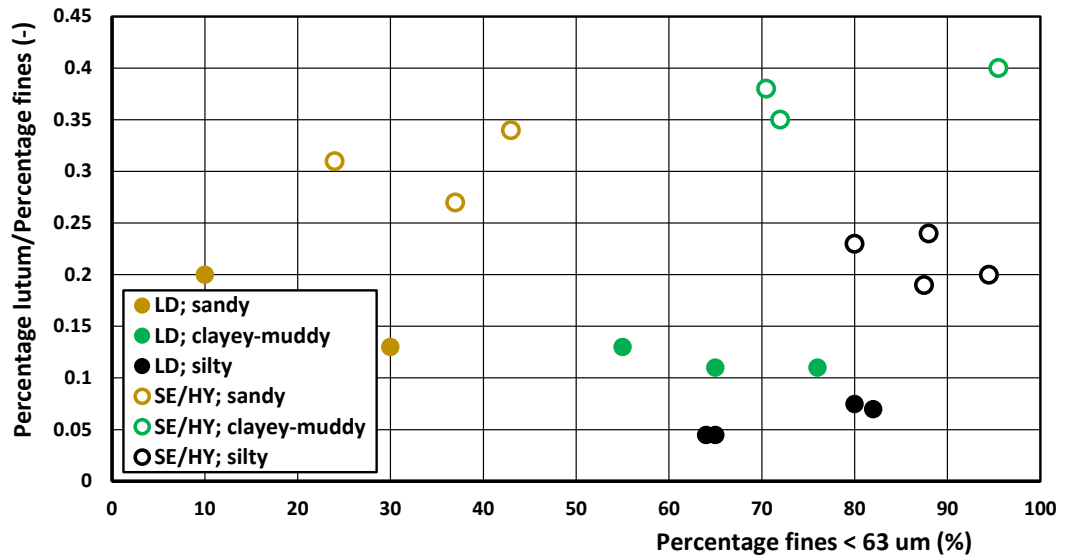


Figure 4.22: Ratio of percentage of Lutum (< 2 μm) and percentage of fines (< 63 μm) as function of percentage of fines.

4.4 4.4 Test results of particle size; additional JDN-bed samples

4.4.1 4.4.1 Methods

Dredging contractor JDN (Belgium) has taken 20 bed material samples (Van Veen grab) along a navigation channel in Bengal Bay near Bangladesh.

Each sample was split into 3 subsamples. Each subsample was analyzed by a different method, as follows:

1. LD-method; Laser Diffraction method (JDN in Belgium)); with and without pretreatment with anti-flocculant;
2. Wet sieving and Andreasen-settling method; sand fraction by wet sieving and fine fraction by settling in Andreasen settling column with subsamples taken by pipette (laboratory IWM in Bangladesh); no pretreatment with anti-flocculant
3. Wet sieving and Hydrometer-settling method; sand fraction by wet sieving and fine fraction by settling in hydrometer column (laboratory GVV in Belgium); pretreatment with anti-flocculant.

4.4.2 4.4.2 Comparison of methods

Percentage of sand

The percentage of sand $> 63 \mu\text{m}$ determined by the laboratories of GVV and IWM is based on the wet sieving procedure in which the samples are washed over a sieve of $63 \mu\text{m}$. Hence, the results of both laboratories should be similar. However, the results are quite scattered; compare data of GVV and IWM in **Figure 4.23**. The overall mean value of the 20 samples of both laboratories is almost similar: $p_{\text{sand,average of GVV}}=13.5 \%$ and $P_{\text{sand,,average of IWM}}=15 \%$. The percentage of sand of 14 of the 20 samples tested by both laboratories is within 30% of each other, which is approximately the natural variation of the percentage of sand in the three subsamples (error related to sample splitting). Hence, substantially deviating values may be related to the sampling splitting method and the number of subsamples involved. Many subsamples should be tested for accurate mean values.

The percentages of sand of the LD-method (of JDN) are substantially higher than the values of GVV. The overall mean value of the 20 samples is 43% (without anti-flocculant) and 32% (with anti-flocculant). Thus, the use of anti-flocculant leads to a smaller percentage of sand.

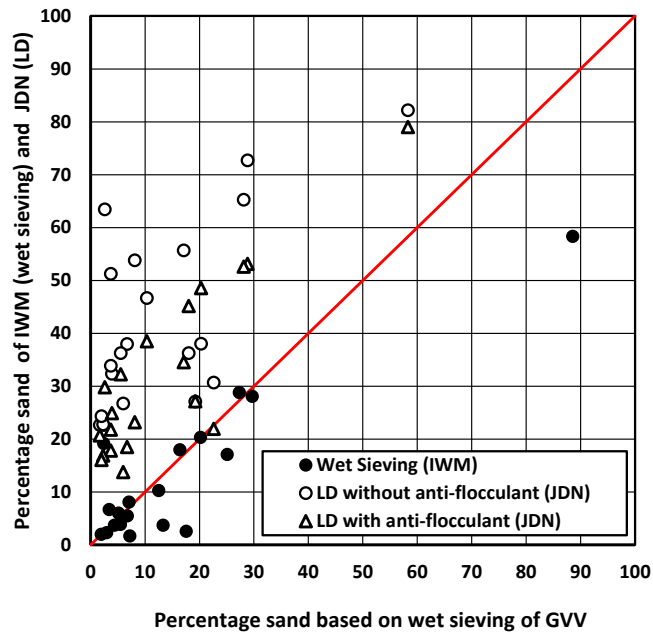


Figure 4.23 Comparison of percentage of sand (p_{sand})

Median particle size, d_{50}

Figure 4.24 shows the median particle size as function of the percentage of sand (method of GVV). The highest percentage of sand is about 60% and the lowest value is about 2%.

The median particle size increases for increasing percentage of sand (p_{sand}), as follows:

- from about 10 to 80 μm for the method of GVV; the scatter is relatively low and the results of this method are most likely the most realistic of the three methods used;
- from about 40 to 90 μm for the method of IWM; d_{50} -values < 40 μm are not found, most likely because anti-flocculant was not used;
- from 20 to 120 μm for the LD-method of JDN; the scatter is relatively high; the d_{50} of most samples treated with anti-flocculant is smaller; most likely, the LD overestimates the particle sizes of the fine fractions.

The particle size distribution of a typical bed sample is shown in Figure 4.25.

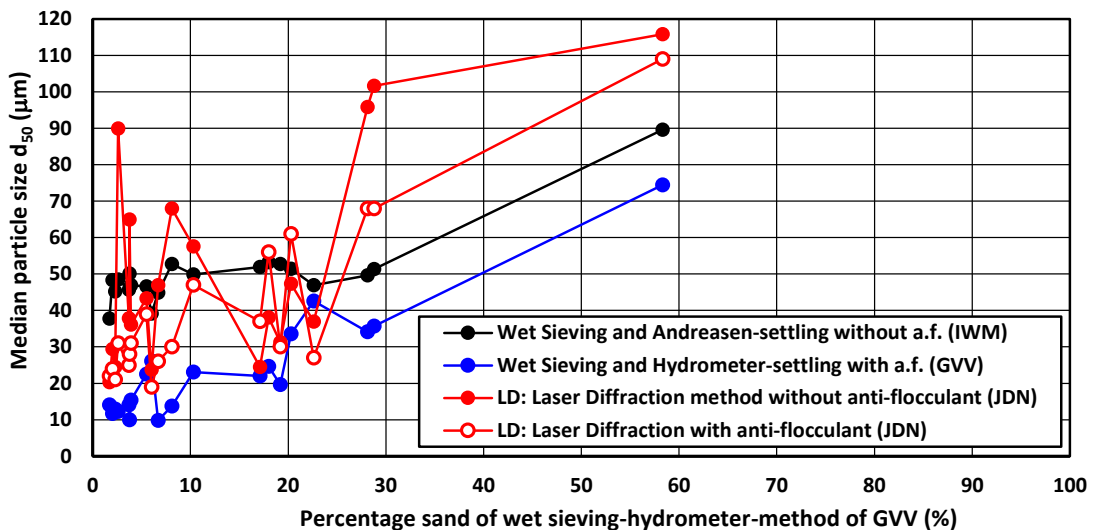


Figure 4.24 Median particle size d_{50} as function of the percentage of sand (method of GVV)

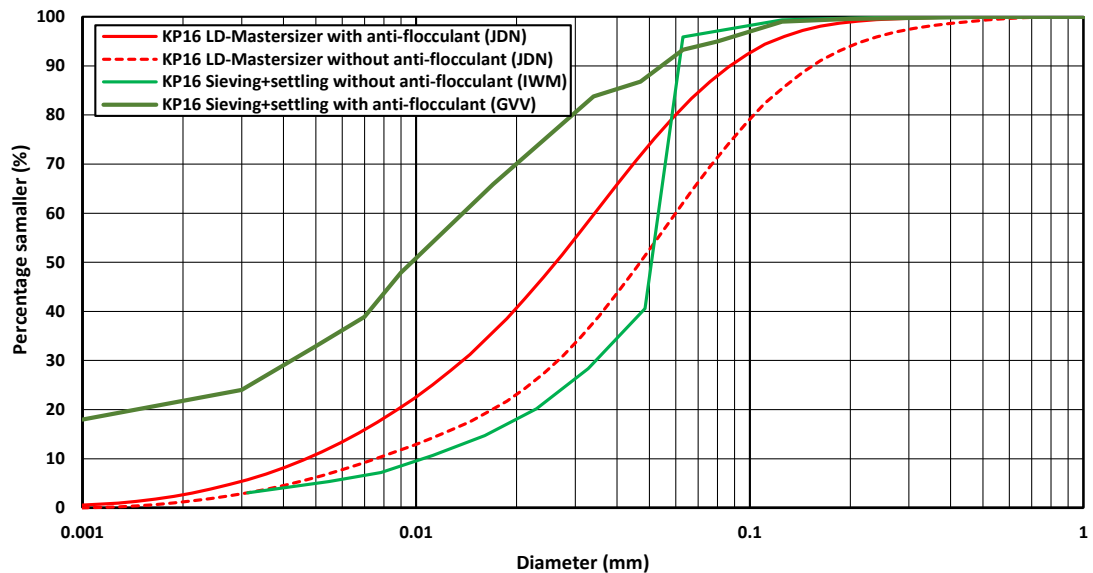


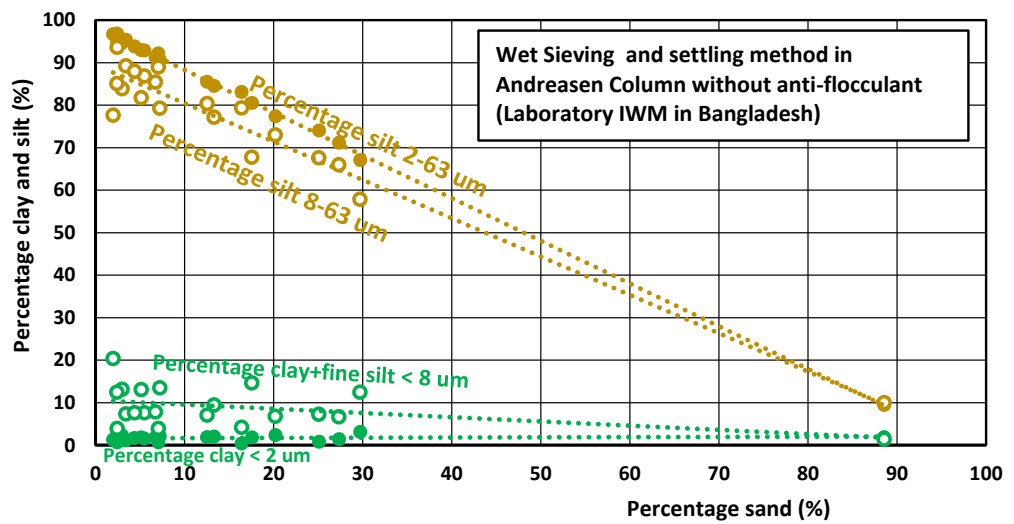
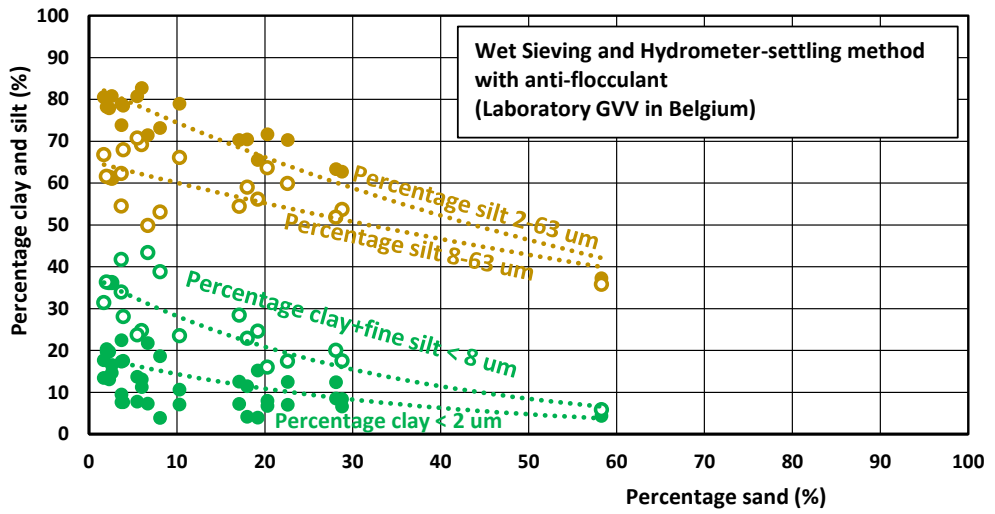
Figure 4.25 Particle size distribution of a typical bed sample

Percentage of clay and silt

Figure 4.26 shows the percentage of clay and silt as function of the percentage of sand for the three methods.

The most important results are:

- wet sieving and hydrometer-settling method of GVV (upper plot):
 - percentage clay < 2 μm decreases from 25% to 5% for p_{sand} increasing from 2% to 60%;
 - percentage clay+fine silt < 8 μm decreases from about 40% to 7%;
 - percentage silt 2-63 μm decreases from 80% to 40%;
 - percentage silt 8-63 μm decreases from 70% to 40%;
 - percentage silt 2-8 μm decreases from 20% to 2% for p_{sand} increasing from 2% to 60%;
 - ratio $P_{\text{silt } 2-63 \mu\text{m}}/p_{\text{clay} < 2 \mu\text{m}}$ is in the range of 5 to 10;
 - ratio $P_{\text{silt } 8-63 \mu\text{m}}/p_{\text{clay} < 8 \mu\text{m}}$ is in the range of 2 to 5;
- wet sieving and TAP-settling method of IWM (middle plot):
 - percentage clay < 2 μm is very low at about 2%;
 - percentage clay+fine silt < 8 μm is lower than 10% to 15%;
 - percentage silt 2-63 μm decreases from 95% to 10%;
 - percentage silt 8-63 μm decreases from 90% to 10%;
 - percentage silt 2-8 μm decreases from 10% to 2% for p_{sand} increasing from 2% to 90%;
 - ratio $P_{\text{silt } 2-63 \mu\text{m}}/p_{\text{clay} < 2 \mu\text{m}}$ is in the range of 20 to 50;
 - ratio $P_{\text{silt } 8-63 \mu\text{m}}/p_{\text{clay} < 8 \mu\text{m}}$ is in the range of 5 to 10;
 - psd-curve often shows a jump between 63 μm and 48 μm (Figure 4.25);
- Laser-Diffraction method of JDN (lower plot):
 - percentage clay < 2 μm is very low at about 1% to 4%;
 - percentage clay+fine silt < 8 μm is lower than about 20% to 25%;
 - percentage silt 2-63 μm decreases from 80% to 20%;
 - percentage silt 8-63 μm decreases from 65% to 20%;
 - percentage silt 2-8 μm decreases from 20% to 2% for p_{sand} increasing from 15% to 80%;
 - ratio $P_{\text{silt } 2-63 \mu\text{m}}/p_{\text{clay} < 2 \mu\text{m}}$ is in the range of 20 to 40;
 - ratio $P_{\text{silt } 8-63 \mu\text{m}}/p_{\text{clay} < 8 \mu\text{m}}$ is in the range of 3 to 5.



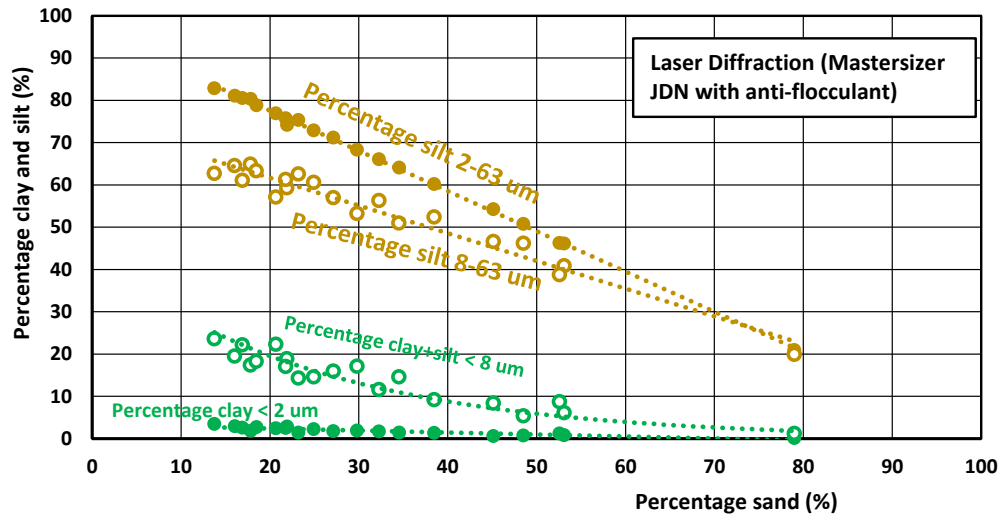


Figure 4.26 Percentage of clay and silt as function of percentage of sand
Upper: method of GVV; Middle: method of IWM; Lower: LD of JDN

4.4.3 Conclusions

The wet-sieving and hydrometer-settling method of laboratory GVV (with pretreatment of anti-flocculant) is found to produce the most realistic results with d_{50} -values over the range of 10 to 100 μm and limited data scatter.

The percentage of clay < 2 μm is in the realistic range of 5% to 25%.

The wet-sieving and Andreasen-settling method of laboratory IWM produces relatively high d_{50} -values between 40 and 100 μm . The percentage of clay < 2 μm is very low (<2%), most likely because pretreatment with anti-flocculant was not used. The psd-curves of IWM show a suspicious jump between the coarser sand fraction > 63 μm and the finer silt fractions < 48 μm , similar to that in Figure 4.13.

The Laser-Diffraction method of dredging contractor JDN (with and without pretreatment of anti-flocculant) produces rather scattered results with d_{50} -values in the range of 20 to 100 μm . The percentage of clay < 2 μm is very low (<3%). The percentage of sand and the median particle size decrease substantially when anti-flocculant is used.

The use of anti-flocculant has a substantial effect on the particle size composition based on the results of the LD-method. Generally, it leads to smaller d_{50} -values because the cohesive and adhesive forces between individual particles are reduced (more individual particles). However, the volume of the subsample also plays a role. The LD-method uses rather small subsamples. So, it is possible that a new sample may have a slightly finer or coarser composition than the old sample. This effect of subsample representativeness can only be overcome by analyzing many subsamples (at least ten) of the same population.

5 Discussion, conclusions and recommendations

5.1 Discussion and conclusions

Methods

The optical Laser-Diffraction method (LD-method) and three sedimentation methods (Hydrometer-method; Pipet/TAP-method; Sedigraph) for measuring the particle sizes of fine silt and clay particles have been studied.

Sample characteristics

Various mud-sand samples were taken from tidal flats, banks and channels in The Netherlands, Belgium, United Kingdom and Bangladesh and stored in (base) containers at a low temperature in the Laboratory of WaterProof BV in Lelystad (NL).

Small subsamples (100 to 200 g) were taken from the base containers after thorough mixing of the material in the base containers. The subsamples were stored in closed bottles and transferred to the laboratories of University of Utrecht and Soil mechanics Wiertsema in Tolbert (Groningen) for analysis of particle size and mineralogy.

The particle size distribution of the samples was determined by using three different methods: Laser-Diffraction method at the University of Utrecht (NL), Sedigraph-method at the soil mechanics laboratory of Wiertsema (Tolbert, NL) and the Hydrometer-method at the laboratory of WaterProof BV (Lelystad, NL).

In addition, the Plasticity Index of the samples were determined and the mineral composition of the very fine fraction ($< 10 \mu\text{m}$) of the samples.

The most important characteristics (physical, mineralogical, plasticity, internal resistance) of the samples are:

- samples Bath-APP (NL), SO3 (BE) and PLUK2 (UK) are fine sandy samples with $d_{50} > 63 \mu\text{m}$ and percentage of sand $> 50\%$; low PI-values < 30 , relatively high dry bulk density $> 800 \text{ kg/m}^3$;
- samples PLUK1 and PLUK4 from Plymouth estuary are silty samples with 70% between 8 and $63 \mu\text{m}$; low PI-values < 30 , relatively high dry bulk density $> 800 \text{ kg/m}^3$; dry density values $> 800 \text{ kg/m}^3$;
- samples BB2 and BB3 from Bengal Bay (Bangladesh) are silty samples with 60% between 8 and $63 \mu\text{m}$; PI-values between 15 and 20 are relatively low, relatively high dry bulk density $> 800 \text{ kg/m}^3$; dry density values $> 800 \text{ kg/m}^3$;
- samples Bath-BAPU, B5 and PA1 (NL) are clayey/muddy sample with percentage of clayey materials ($< 8 \mu\text{m}$) $> 40\%$ and relatively high PI-values > 40 ; dry density values $< 800 \text{ kg/m}^3$;
 - yield stress values show an increasing trend for increasing dry density of the mud-sand mixture;
 - yield stress values of the clayey samples with low sand content ($< 30\%$) are fairly close together and mostly within the envelope curves of literature data;
 - yield stress values of the silty samples are in the low range of the envelope curves;
 - yield stress values of two samples with a relatively high sand content $> 60\%$ to 70% are significantly above the envelope curves, which may be the effect of high internal friction between the sand particles.
- mineral composition shows distinct grouping by geographic region:
 - samples B5 (Noordpolderzijl) and BAPU, PA1, APP and SO3 (Western Scheldt, NL and Zeeschelde, BE) are very similar; Illite is dominant;
 - samples PLUK1 and PLUK4 (UK) are very similar; Kaolinite is dominant; Illite is also substantial
 - samples BB2 and BB3 are very similar; Illite and Chlorite are dominant.

Particle size distribution

The mud-sand samples consist of sand, silt and clay particles. The shape of most silt particles is nearly spherical, but the shape of clay-type particles is more platy/flaky. The plate sizes (diameter) of clay particles can only be measured accurately by using Scanning Electron Microscopy (SEM-method), but this method is very laborious (labour-intensive) and requiring many subsamples for representative analysis results.

The particle size measured by sedimentation methods (Hydrometer-method; Pipet/TAP-method; Sedigraph-method) is an equivalent spherical diameter based on the Stokes settling equation. The spherical Stokes diameter is much smaller (factor 2 to 5) than the plate diameter of clay particles, which are found to be as large as 4 to 5 μm for clayey samples in China (Lu et al., 2000).

The particle size method measured by the Laser-Diffraction method is an equivalent spherical diameter derived from cross-sectional projected areas of the particles and represents the major diameter of the particles and is therefore closer to the platy diameter of disc-type (platy/flaky) clay particles. The LD-method may produce less accuracy for mixtures of mud and sand, because the fine mud particles in the test sample are partly shaded by the larger sand particles resulting in an erroneous particle size distribution (Ibanez Sanz, 2018). A solution may be to separate the mud and sand fractions and measure both fractions individually by the LD-method and combine both size distributions into the psd of the total sample. However, this may also introduce biased results due to loss of fines in the separation procedure. It also reduces the advantage of the LD-method that it can measure the full sample in one go.

The problem of measuring the clay fraction $< 2 \mu\text{m}$ may be overcome by defining a clay-dominated fraction $< 8 \mu\text{m}$ as an indication of the presence of cohesive clay-type particles in a soil sample. The clay-dominated fraction $< 8 \mu\text{m}$ can be more accurately determined by both the Sedimentation methods and the LD-method. For example, the percentage of clay-type particles $< 8 \mu\text{m}$ is about 50% for sample H2 of Noordpolderzijk and the percentage $< 2 \mu\text{m}$ is about 35%. The former value (50%) is more reliable than the latter value (35%), as the (systematic) inaccuracy of the Hydrometer-method increases for particles smaller than $8 \mu\text{m}$.

It may be confusing to describe the fraction $< 8 \mu\text{m}$ as the clay-dominated fraction. Instead we can use the $8 \mu\text{m}$ fraction to determine which samples are cohesive based on Atterberg-limits. Soils are cohesive for plasticity index $PI > 10\%$. For a limited number of samples we can determine the relation between the $8 \mu\text{m}$ content and the PI-value, and from this determine the critical amount of the $8 \mu\text{m}$ fraction for which the sediment is cohesive.

Based on the research results from this study and the literature, it is found that:

- the disc-type diameter of clay type particles ($< 8 \mu\text{m}$) is up to 5 times larger than the spherical Stokes diameter ($d_{\text{plate,clay}} \cong 5 d_{\text{sphere,stokes}}$) and up to 1.5 times larger for silty particles in the practical range of 8 to $32 \mu\text{m}$;
- the equivalent diameter (d_{LD}) of the LD-method is much larger than the spherical Stokes' diameter for platy clay-type particles ($d_{\text{LD}} \cong 5 d_{\text{sphere,stokes}}$); the LD-diameter (d_{LD}) is slightly smaller than the plate diameter of clay-type particles ($d_{\text{LD}} \cong 0.7$ to $0.8 d_{\text{plate}}$); the LD-diameter may be somewhat larger than the sieve diameter for non-spherical (ellipsoid) sand particles ($d_{\text{LD}} \cong 1.2$ to $1.3 d_{\text{sieve}}$), because ellipsoid sand particles may pass through a sieve mesh with their smallest diameter;
- the percentage of clayey sediments ($< 8 \mu\text{m}$) in terms of the spherical Stokes diameter measured by the Pipet/TAP-method and the Hydrometer-method are within 20% difference for muddy samples; the differences of the percentage of clayey sediments measured by both methods may go up to 50% to 100% for silty samples with a small amount of clay;
- the sedimentation methods overestimate the percentage of clay $< 2 \mu\text{m}$, as many clay-type particles have plate diameters in the range of 2 to $4 \mu\text{m}$, but which are seen as spherical diameters $< 2 \mu\text{m}$ by the sedimentation methods.

Differences between the Hydrometer sedimentation method and the Pipet/TAP sedimentation method are most clearly visible for the coarser silt range. The first readings of the Hydrometer show a sudden drop in the suspension density (and therefore mass percentages). Various causes can be mentioned for the drop related to the initial readings:

- presence of sand particles, which is however very unlikely as the sand fraction was removed by wet sieving over mesh of 63 μm ;
- generation of aggregates (coagulation/flocculation) with relatively large settling velocities due to the intensive mixing to produce a homogeneous high-concentration suspension;
- presence of non-homogeneous suspension despite mixing efforts;
- initial float readings are representative of the density of the suspension of water with discrete sediment particles.

The most important results of the comparison of the LD-method, Sedigraph-method and Hydrometer-method for analysis of particle size analysis of the mud-sand samples of the MUSA project are:

- the samples B5 (Noordpolderzijl) and BAPU (Western Scheldt) are similar; sample B5 exhibits a sharper peak in the very fine sand fraction, whereas the distribution of BAPU is more platykurtic; PLUK1 and PLUK 4 from Plymouth estuary are very similar samples; BB2 and BB3 from Bengal Bay are also very similar samples;
- the Sedigraph and the Hydrometer-methods produce significantly smaller d_{10} , d_{50} and d_{90} -values than the LD-method; the Sedigraph-method produces somewhat smaller values than the Hydrometer-method;
- the LD-method produces a particle size distribution which is shifted to the right (coarser sediment) compared to that of the Sedigraph-method and the Hydrometer-method, which is in good agreement with earlier findings in the literature (Yang et al., 2019);
- removal (OCR) of organics and calcareous materials from the samples yields a shift to the left of the pdf-curve with smaller values d_{50} -values; percentage of fines ($<63 \mu\text{m}$) increases;
- LD-method shows relatively large variability between similar samples (bulk1, bulk2, bulk3) requiring many measurements to obtain accurate average values.

Finally, it is noted that the size of platy sediments is better represented by the Laser-Diffraction-method. The Sedigraph and Hydrometer sedimentation methods are preferred in the case that the particle size results are used for determination of settling velocities. The Hydrometer method is more laborious, but the variability of the results is low due to the relatively large sample mass involved (30 gram).

The LD-method offers an attractive solution for a rapid indication of particle sizes involved, but many (small) samples are required to reduce variability.

The use of anti-flocculant (pretreatment) has a substantial effect on the particle size composition based on the results of the LD-method with and without pretreatment. Generally, it leads to smaller d_{50} -values because the cohesive and adhesive forces between individual particles are reduced by anti-flocculant (more individual particles). However, the volume of the subsample also plays a role. The LD-method uses rather small subsamples. So, it is possible that a new sample may have a slightly finer or coarser composition than the old sample. This effect of subsample representativeness can only be overcome by analyzing many subsamples (at least ten) of the same population.

5.2 Recommendations

The following recommendations are made:

- The first two readings of the hydrometer method show a drop in the suspension density (and therefore mass percentages). It is recommended to neglect float readings within 1 to 2 minutes (systematic error) after the start of the test, see dashed line in **Error! Reference source not found.**
- The TAP-method shows some variability per individual TAP sample, due to the slight disturbance within the settling suspension generated by each TAP. The overall random measuring error is larger than that of the Hydrometer-method due to the many handlings involved (preparation of relatively small concentration; filtration, drying). It may be wise to further reduce the number of TAPs taken, to reduce the disturbance on the sample and the handlings involved to a minimum.

More comparative research should be done on mud-sand samples using sedimentation methods and the LD-method. Determination of the Atterberg-limits of the samples should be included.

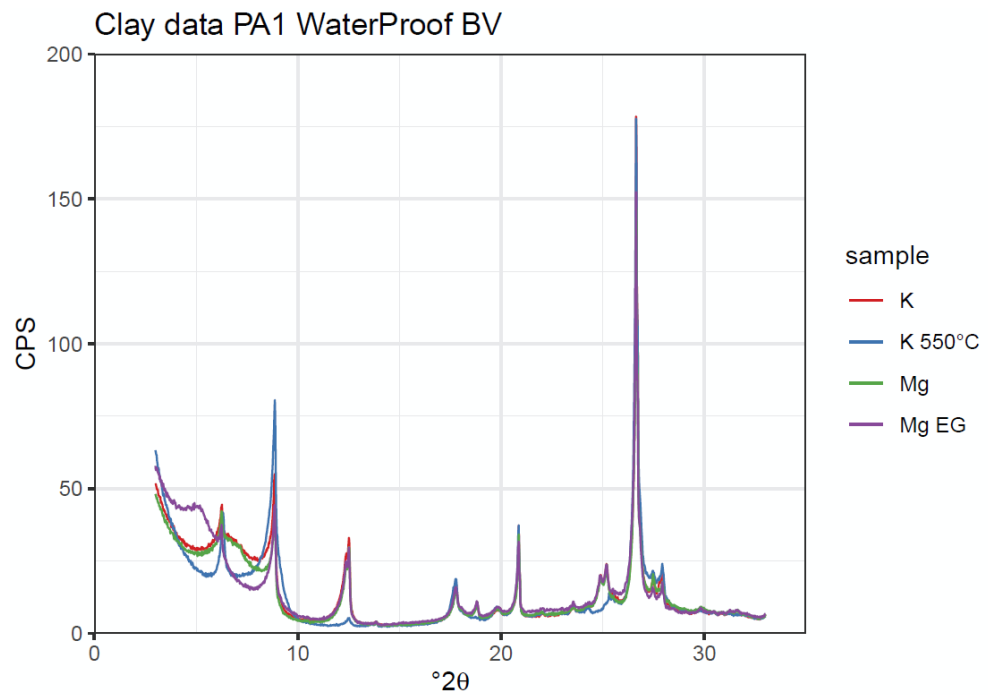
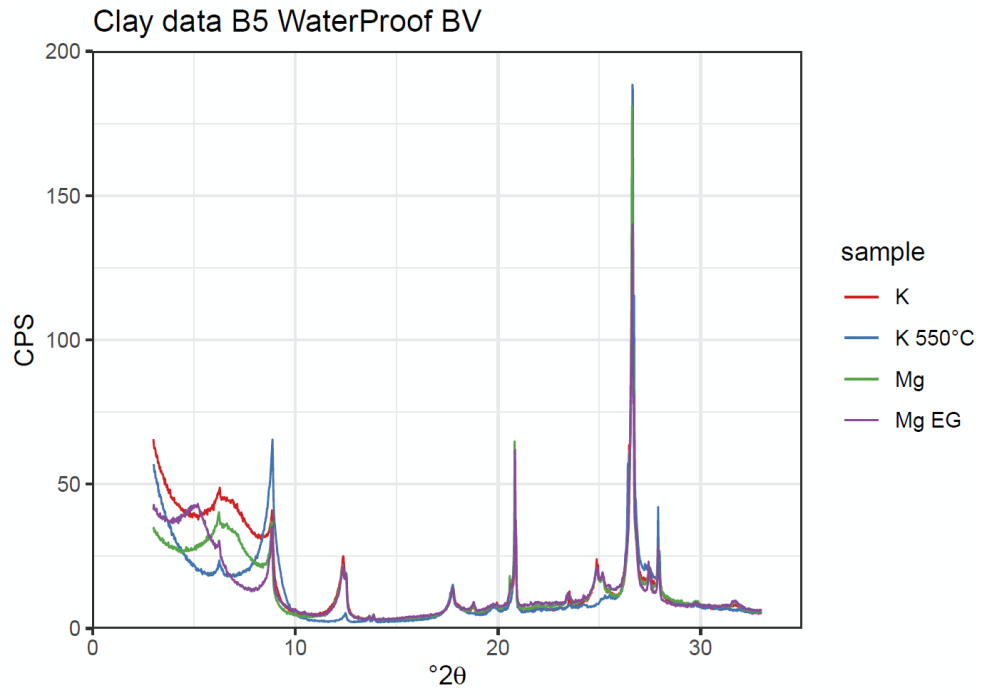
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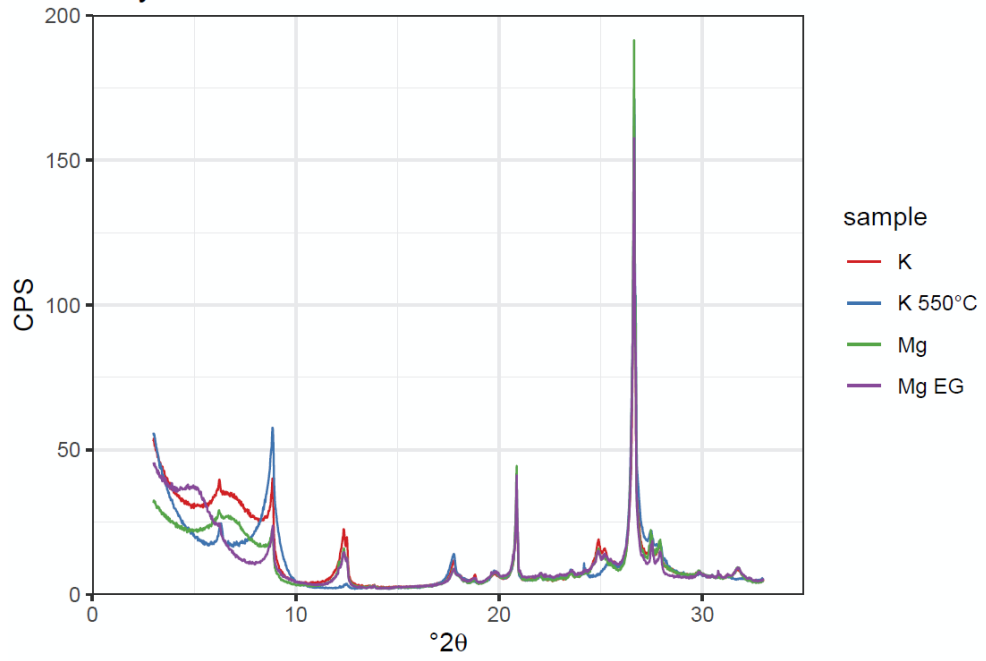
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A Diffractograms of MUSA samples

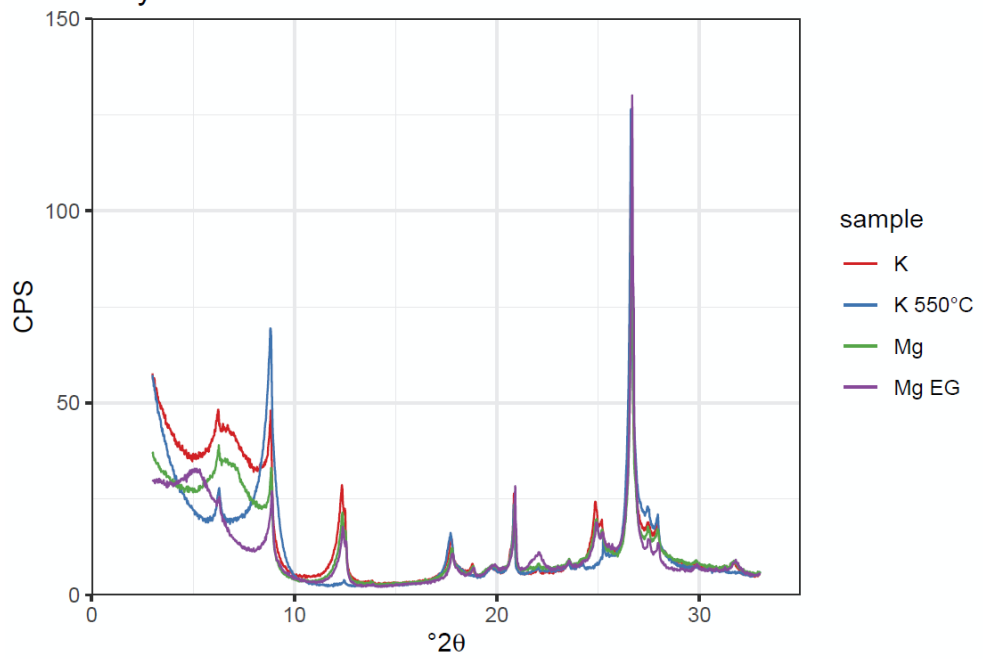
Diffractograms of MUSA-samples B5, PA1, BAPU, APP, SO3, PLUK1, PLUK4, BB2, BB3



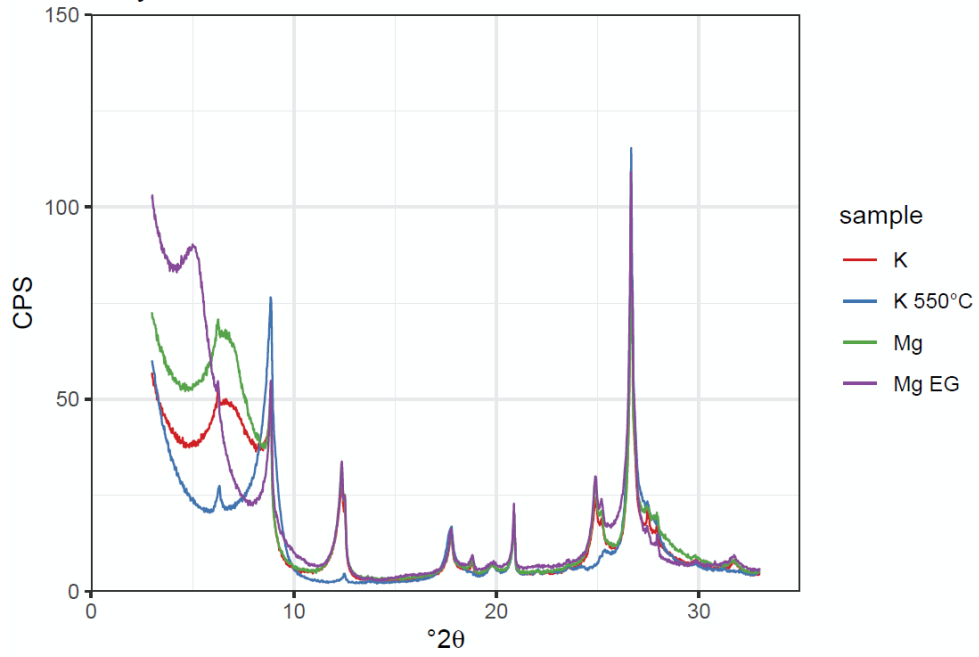
Clay data BATH WaterProof BV



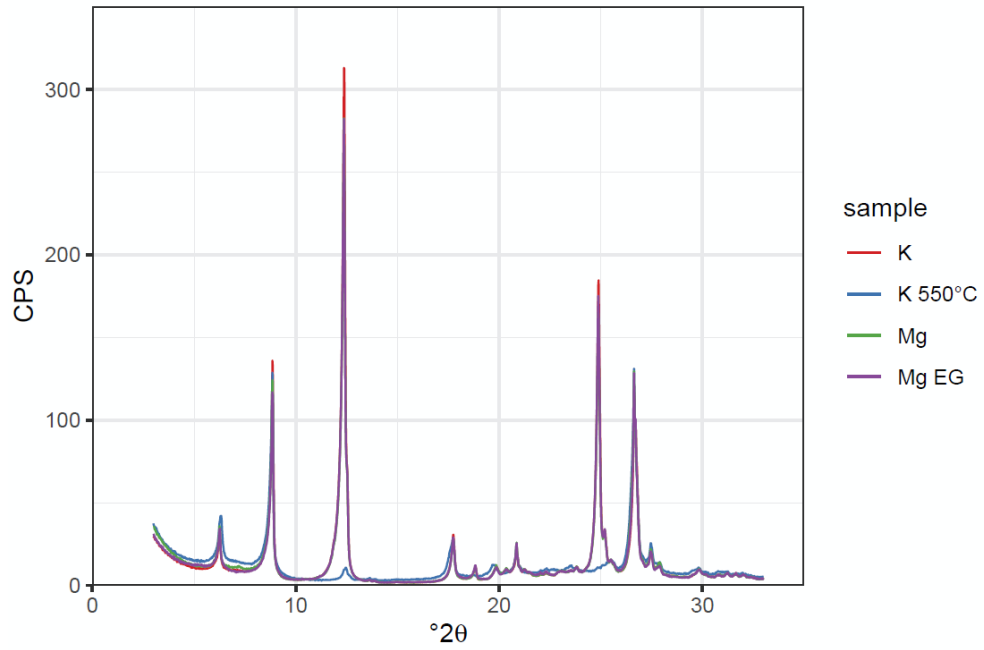
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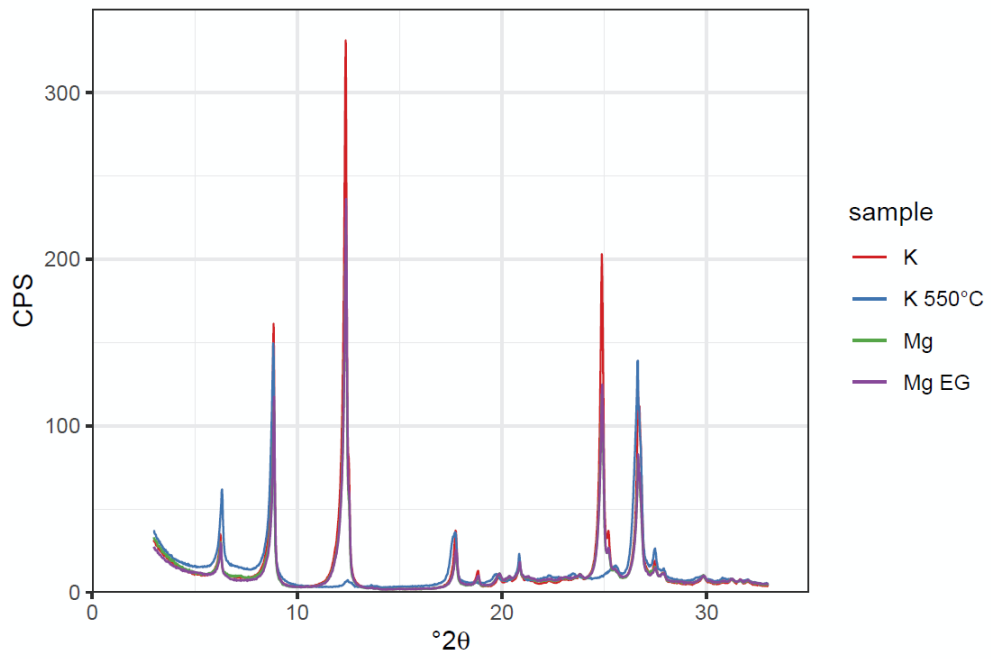
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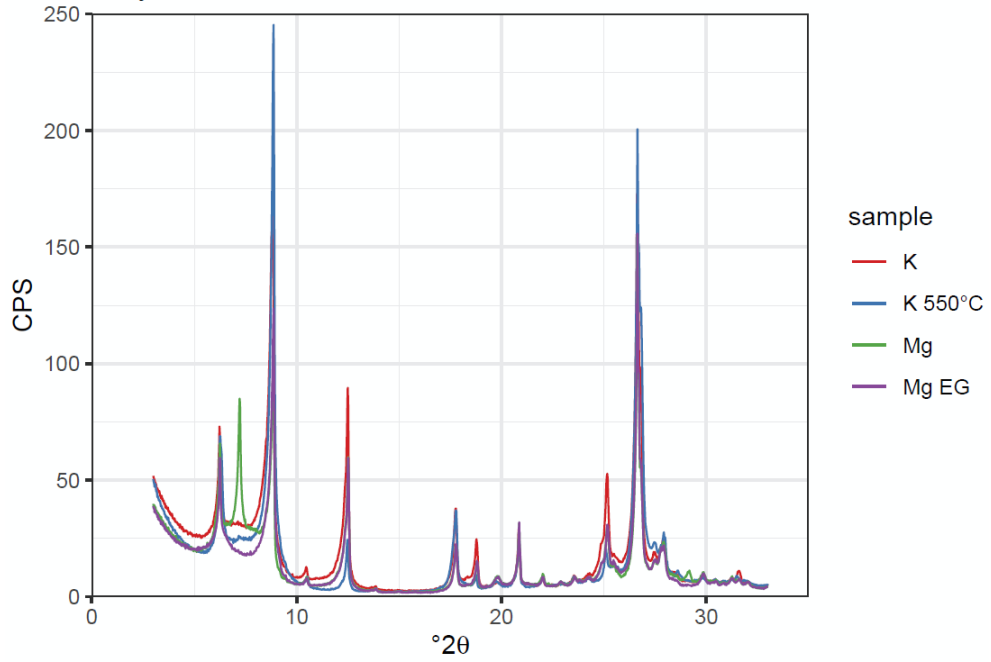
Clay data PLUK1 WaterProof BV



Clay data PLUK4 WaterProof BV



Clay data BB2 WaterProof BV



Clay data BB3 WaterProof BV

