Memo

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Date	Number of pages	Project
21 March 2013	6	1204697
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Subject
Grain size distribution

Grain size distribution analysis of bottom samples

Knowledge about grain size distribution is essential for a large variety of purposes. This note focuses on turbidity related studies especially caused by bottom material that may come into suspension by energy supplied by tidal currents, waves or dredging. To get insight in the sediments that potentially may cause turbidity, when suspended, bottom samples are analysed for, amongst others, grain size distribution. One of the parameters of interest is the content of fines in the bottom material. Although occasionally the content of fines may exceed 10%, usually it ranges from less than 1% to a few percent. The accuracy of determination should be better than 25% of the content.

A multitude of protocols is in use for determination of particle size distribution of bottom samples obtained from the sea floor. Dutch institutes like Imares, Vrije Universiteit Amsterdam, NIOZ (NIOO), Alterra, Deltares chose the protocol which meets best the requirements as imposed by the purpose of the specific analysis, hence, the choice of protocol is application based. In general, routine analyses are executed in compliance with standards like (ref 1): CEN ISO/TS 17892-4, NEN 5753. ISO 11277:2009. The samples may be pre-treated in accordance with the NEN 5751 (ISO 11464, DIN 19683) norm, after which organic materials, carbonates, and, possibly, iron oxides are removed. Some general standards on grain size analyses are ISO 13320 and ISO 11277, both of 2009. ISO 13320:2009 provides guidance on instrument qualification and size distribution measurement of particles in suspensions in liquids through the analysis of their light-scattering properties. Particle sizes may range from approximately 0,1 µm to 3 mm. ISO 11277:2009 specifies a basic method of determining the particle size distribution applicable to a wide range of mineral soil materials.

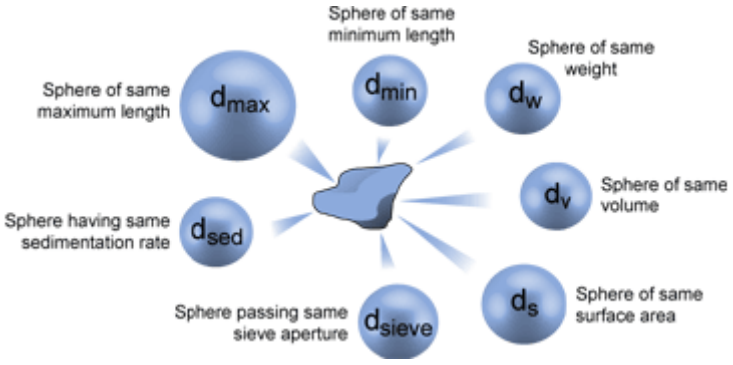
The above mentioned science institutes often apply special analysis protocols dedicated to meet application specific requirements. These protocols may partly or entirely comply to a number of national / international standards. In most cases the protocols are applied routinely according to well defined steps. Removal of carbonates and organic materials is usually part of the protocols.

Several parties in the Netherlands and Flanders have developed a protocol (Afstemming deeltjesgrootte bepaling t.b.v. MONEOS, ref 2) for the analysis of bottom samples obtained from the Scheldt estuary. One of the main goals of Moneos is to set a practical standard in support of the exchange of grain size distribution data as obtained by laser diffraction instruments. The preference for laser diffraction as analysis technique originates

from the wide particle size range together with the large dynamic reach that can be determined in a single measurement.
U.S. Geological Survey has published a standard procedure in open-file report 00-358. Chapter 1: Grain-size analysis of marine sediments: methodology and data processing, L.J. Poppe et.al. (2000, ref 3)

1. definitions

In nature samples rarely have a spherical shape, hence characterising particles by a single size, like a diameter is not a trivial matter. A definition of characteristic size is to be formulated. The figure on the right shows a number of definitions in use.



Other definitions are:
Drag diameter (dd): Diameter of a sphere that has the same resistance to motion in the same fluid (having same viscosity), moving at the same velocity as the particle
Free-falling diameter (df): Diameter of a sphere having the same density and the same free-falling speed as the particle in the same fluid
Surface volume diameter (dsv): Diameter of a sphere having the same surface to volume ratio as the particle
Stokes' diameter (dSt): Diameter of a free falling sphere (i.e., particle having attained the Stokes velocity) in the laminar flow region
The above definitions were obtained from: Measuring particle size using modern laser diffraction techniques, Dr P. Kippax (2007, ref 4).

For turbidity applications the cross sectional area would be a proper size parameter because turbidity is primarily caused by blocking the passage of light. At higher concentrations also (forward) scattering affects the perceived turbidity. The time constant of a turbidity event (duration) is affected by the fall velocity of the particles, the latter depends upon the cross sectional area, the particle volume, the difference in specific gravity of the particle and the fluid and the roughness/shape of the particle.

Gravity based analysis methods determine the equivalent radius based on Stokes Law on stationary settling. The equivalent radius is linked to a theoretical sphere having the same settling velocity as observed from the particle.

The pipette withdrawal method and the Sedigraph are typical representatives of analysis methods based on particle separation by size dependant fall velocity. The laser diffraction based instruments determine particle size based on radius dependant properties.

2. Sampling

The method used for collecting the bed material is chosen to obtain sample in an undisturbed condition. A concern is that while landing the sampling device on the seabed fluf and fluid mud is washed away by the displacement current around the sampler. Another concern is that should the sampling device not be completely closed, e.g. due to some object like wood, stone, shell, in particular the top section of the sample (bottom -

water interface, possibly covered by a fluf layer) is easily lost. To some extend also fines residing in the pores of the sand package can be washed out. This washing may occur during manipulation of the sampler, especially above water, and while opening it.
Frequently used sampling devices are: Box corer, Drop corer, Shipek, Van Veen Grab, Seaboss. Seaboss is a sensor and camera system that also comprises a Van Veen grab. Shipek makes use of two shells, much like the Van Veen grab, however, the shipek shells are spring loaded whereas the Van Veen shells are gravity loaded (by added weights). Box corers exist in a variety of sizes (and weights). The sample is collected in a fairly undisturbed condition. The same applies to the drop corer. In the latter case the sample depth may exceed that of the other mentioned samples.

In all of the samplers there is a significant risk of sediment loss due to washing out of pore water and water from above the sample. The box corer, if well designed, properly maintained and carefully operated, may collect bed material up to several decimetres deep without significant loss of water.

3. Conservation

Conservation, handling and storage of the sample will unavoidably affect the existence of aggregates like flocculated silt. By accidentally breaking up the conglomerates, the grain size distribution and the fall velocity distribution will shift. Is turbidity one of the aspects under investigation, then also the organic matter and calcium carbonates are of importance and should be preserved. A general policy is to store samples completely without light and cool them to just above freezing point in case analysis will be executed within about 24 hours. For storage over longer periods samples are kept in frozen condition, depending on the purpose down to temperatures in the range of -80°C. Frozen samples may also be freeze dried. Experimental results under the Moneos project suggest that the grain size distribution is not significantly altered by storing the samples in frozen condition for a short duration (couple of weeks). However, it should be noted that the manipulation during sampling, handling and analysis may destroy virtually all fragile conglomerates.

4. Preparation

As mentioned above, in particular conglomerates of fines but also fines ‘glued’ to coarse particles may be altered due to the manipulations (mechanical; chemical). The coarse section of the grain size distribution will be hardly affected. Turbidity is caused by mineral matter like fines (and sand), by calcium carbonate and by matter of organic origin (e.g. algae, bacteria but also detritus); this should be taken into account when deciding the method of sample preparation.

Only a few grams are needed for particle size analysis by laser diffraction; hence, the sub sample has to be split form the main sample after carefully mixing the entire main sample. Potentially this may result in the loss of some fines due to adhesion of the fines to any object it comes in contact with. For the splitting a special splitter tool would be helpful.

As part of the sample preparation procedure usually organic matter is removed (to a great extend) by oxidation under application of hydrogen peroxide. Calcium carbonate is dissolved by hydrochloride acid. Both affect the turbidity properties of the sample. In addition, washing out of salt changes the electrical charge distribution of the particles and as a result the flocculation properties, further, the washing may remove (partly) the colloidal-clay fraction.

During grain size distribution analysis the formation of particle aggregates is to be avoided. Many instruments are equipped with an ultrasound stirrer device. However, if too much energy is applied the particles may loose slivers and as such increase the amount of fines. Such fragments can be visualised by microscope. This fragmenting also may occur when ultrasound is applied to break bonds between samples (Blok & Arentz, 2012, ref 5).

A more gentle method to separate particles is by shaking. To some extend, the shaking action mimics the wave induced currents near the seabed. Shaking breaks aggregates of fine particles into individual particles and helps separation of loosely glued fines from sand particles. The assumption is that shaking makes the fines available that can be washed out off the bottom sediment by wave action without damage to the sand particles. Tightly glued fines will not be separated; hence, those particles will not come available to be washed out off the seabed.

5. Analysis

Samples containing a small fines fraction only may be split into a fine fraction and a coarse fraction by sieving. This to avoid bias to the smaller fines fraction or even overwhelming of that fraction by the large fraction of coarse particles. Often 63 µm sieves are applied, but also 90 µm. The latter to ascertain that (virtually) all fines will reach the fines fraction. The fine and coarse fractions are analysed separately. A sieving standard is ISO 3310-1/-2.

Some methods to determine grain size distribution are:
Gravity and Stokes law based:
Pipette withdrawal: samples are taken from a settling suspension and analysed for sediment content;
x-ray sedigraph: the instrument monitors the decreasing concentration is a settling suspension;
sedimentation balance: the settled sediment accumulates, hence, into increasing weight on a weighing device.

Laser based:
Particle counter (Coulter counter): particles pass through a detector counting the particles while measuring the cross sectional area
Laser diffraction (Malvern Master Sizer 2000): suspended particles pass trough a measuring volume illuminated by one or several laser beams.

The gravity based methods provide information about the fall velocity distribution of the suspension. The fall velocity of a particle not only depends upon its size but also on the specific gravity of the particle and of the fluid. For each fall velocity while applying Stoke's law the equivalent particle diameter pertaining to the fall velocity can be calculated. The shape of the particle and also offset from the assumed specific gravity will result in a deviation of the observed particle size. Hence, the estimated turbidity of the suspension will also deviate. Fall velocity data is also useful to assess the required flow velocity / turbulence to bring the particle into suspension and keep it in suspension.

Laser based instruments may produce a representative grain size distribution but the fall velocity distribution could deviate from reality because of lack of specific gravity data. For particles of the same size sand particles drop at a different rate than calcium carbonate or algae.

An instrument based on Stoke's law in combination with a turbidity measurement in the lower section of a settling column would yield data about the turbidity decrement over time in still water. None of the above mentioned instruments alone delivers all the parameters required for assessment of turbidity over time. To complicate matters turbidity varies also due to change in grain size distribution, e.g. by formation of aggregates or destruction thereof, production or annihilation of particles.

References 6 to 8 give technical details of laser diffraction technology and recommended procedures, whereas references 9 and 8 compare several grain size analysis methods with each other. Reference 11 is an example of application specific analysis.

Some publications of interest are listed below.

1. The ISO standards as mentioned in this Note contain information about specific details related to sediment preparation and analysis methods.

2. Afstemming deeltjesgrootte bepaling tbv MONEOS, VNSC - werkgroep O&M - projectgroep Monitoring en Data.
   Bakker, G. Spronk, (2012)
   Rijkswaterstaat Waterdienst

3. U.S. Geological Survey open-file report 00-358
   Chapter 1: Grain-size analysis of marineSediments: methodology and data processing
   Poppe, L.J.[1], Eliason, A.H.[2], Fredericks, J.J.[3], Rendigs, R.R.[1],Blackwood D.[1], and Polloni, C.F.[1] (2000)
   1) Coastal and Marine Geology Program, USGS, Woods Hole, MA 02543
   2)Eliason Data Services, 230 Meetinghouse Road, Mashpee, MA 02649
   3) Woods Hole Oceanographic Institution, Woods Hole, MA 02543

4. Measuring particle size using modern laser diffraction techniques
   Dr Paul Kippax (2007),
   Malvern Instruments Ltd, UK

5. Bodemmonster Analyse, Egmond
   B. Blok en L. Arentz (2012),
   Deltares, Delft.
   Project 1205620-000

6. A Primer on Particle Sizing by Static Laser Light Scattering
   Paul A. Webb (2000)
   Micromeritics.

7. NIST particle size SP960-1
   Ajit Jillavenkatesa, Stanley J. Dapkunas, Lin-Sien H. Lum (2001)
   NIST Materials Science and Engineering Laboratory

8. U.S. Geological Survey Open-File Report 2005-1001
   Edited by: L.J. Poppe, S.J. Williams, and V.F. Paskevich (2005)
   USGS East-Coast Sediment Analysis: Procedures, Database, and GIS Data

9. Laser-diffraction and pipette-method grain sizing of Dutch sediments: correlations for fine fractions of marine, fluvial, and loess samples
P. Buurman, Th. Pape, J. A. Reijneveld, F. de Jong & E. van Gelder (2001)
Wageningen University, Geologie en Mijnbouw Netherlands Journal of Geosciences 80 (2): p49-57. - ISSN 0016-7746

10. Comparison of laser grain size analysis with pipette and sieve analysis: a solution for the underestimation of the clay fraction
Martin Konertand, Jef van den Berghe (1997)
Vrije Universiteit Amsterdam, Faculty of Earth Sciences

11. A grain size analysis approach to variations in Iceland-Scotland overflow water during ice rafted debris events.
Bryan Lougheed (2007)
Vrije Universiteit Amsterdam