



Particle Characterisation in Geological Applications

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1 State of the art

Measurements of the particle size distribution and characterisation of shape is one of the necessary tasks in order to describe sediments and soil samples in detail (SCHEFFER & SCHACHTSCHABEL 2002). After pretreatment of unconsolidated rocks like drying, gentle comminution of lumps and removal of biological material as well as removal of soluble salts (ISO 11464 2006), the classic method for measurements of the particle size distribution is regulated by several standards (DIN 19683 Blatt 2 1973, DIN 18123 1971, DIN 66115 1983, DIN ISO 1299 2000). Most of them are related to the German nomenclature for grain size fractions of soils and sediments (SCHEFFER & SCHACHTSCHABEL 2002). Classic methods for measurements of broadly distributed samples always require a separation into at least one fine and one coarse fraction.

The common procedure can be described as follows:

Weighting of the pretreated sample-sieve analysis (manually or sieve shaker) at 2 mm-recording the mass of the coarse amount-wet-sieving of the fraction < 2 mm at 63 microns-drying and recording the mass of the fraction between 63 microns and 2 mm-measurements of the fraction < 63 microns using a sedimentation method (e.g. Andreasen Pipette, Köhn Pipette or Areometer application)-synthesis of sieve- and sedimentation results.

One essential disadvantage of sedimentation methods is the long measuring time, sometimes lasting longer than one day. In addition it is questionable, how the density of a sample containing different minerals can be determined precisely. Another disadvantage is the missing information on the shape of the particles: Especially in humid climate zones the process of siallitic alteration is responsible for an enrichment of clay minerals in the soil and the finer fractions often reveal essential amounts of kao-

linite, montmorillonite, illite, etc. (RÖLLIG 1971). These minerals belong to the so-called phyllosilicates (or layer silicates), which are composed of $[\text{SiO}_4]$ tetrahedrons combined with Al-octahedrons. A determination of shape varies frequently from particle to particle. The reason for the "random" shape is, that the fit between the network of tetrahedral and octahedral sheets is based on weak Van der Waals forces, thus the fits are not exact, the different distortions of these sheets have an influence on the dimensions of the particles, and the sheet structure causes a flaky habit of the minerals (HATHAWAY 1979). Unfortunately, for the calculation of the particle size distribution with help of sedimentation technique, which uses settling velocity of the particles, the precise knowledge of the shape is needed, especially with regard to the flaky habit of the phyllosilicates, which can orientate themselves in a laminar flow in that way that their settling velocity is much slower than it should be for their volume. Hence, this effect results in a too fine calculation of the particle size distribution with respect to the reality. Laser diffraction calculates volume information based on the equivalent spherical diameter of the particles passing a laser beam in a laminar flow, so that all possible orientations are measured (DIN 13220-1 1999). Published results of particle size analyses of kaolinite, illite as well as montmorillonite revealed - as expected - much finer distributions for the results based on sedimentation technique than for results based on laser diffraction, the determination of a correction factor was not feasible and for the measurement of the particle size distribution a laser diffraction system was recommended (BERTHOLD, LÜHMANN, KLEIN & NICKEL 2000). Missing correlation factors are also known from a comparison between sieve-pipette measurements and analyses performed with a laser diffraction system (BEUSELINCK, GOVERS, POESEN, DEGRAER & FROYEN 1998).

2 Introduction of a new measuring method

Thus, in order to overcome the problems appearing with the classic methods, time is ripe to analyse soil samples using modern measuring techniques.

As a proposal of a modern technique, a step by step – procedure with a presentation of an accompanying sample (field Name: “sand”, fig. 1) is



Fig. 1: Broadly distributed sample with the field name “sand”

given below:

1. Weighing the entire amount of the dry or dried sample:
2. Dry sieving at 6.3 mm and recording the mass of the residue on the sieve (9.55 % > 6.3 mm)
3. Wet – sieving of the fraction < 6.3 mm at 63 microns, weighing and recording the mass of the dried residue (60.06 % > 63 microns)
4. Measurement of the fine fraction performed with a laser diffraction instrument using a measuring range from e.g. 0.5 – 175 microns:
5. Measurement of the fraction between 63 microns and 6.3 mm performed with an image analysis instrument using a measuring range from e.g. 20 – 6820 microns:(fig. 2,3).
As an outstanding innovation of this measuring technique the characterisation of the shape (as one essential criterion to identify the composition of the soil, the grade of transportation or its origin) during the analysis itself should be highlighted (fig.4).
7. Merge of the results from the image analyser with those from the laser diffraction instruments under consideration of the recorded mass measured with the wet–sieving procedure (Fig.. 5)
8. Visual determination of the maximum size (estimated maximum size: Approx. 3 cm)
9. Link of the merged curve with the recorded mass of the fraction > 6.3 mm using a commercial evaluation software package (fig. 6-8).

MEASUREMENT SPECIFICATION

Product name	: Sand
Sample preparation	: Separation of the coarse fraction with a 6.3 mm sieve; wet sieving of the finer fraction (90.45 % < 6.3 mm) at 63 µm (39.94 % < 63 µm); sampling of the fine fraction with a pipette
Sensor	: HELOS
Dispersing system	: QUIXEL
Measuring range HELOS	: R3: 0.5/0.9...175 µm (f = 100.00 mm)
Mass requirement	: 200 ml of the sieved fine fraction
Start condition of measurement	: button
Stop condition of measurement	: 10 s meas.time or 20 s real time
Time base	: 100.00 ms
Measuring time	: 10.00 s (10.00 s - 10.00 s)
Optical concentration	: 19.99 % (19.81 % - 20.21 %)
Suspension liquid	: H ₂ O
Additive	: Na ₄ P ₂ O ₇
Window spacing of the cuvette	: 2.00 mm
Dispersing time	: 180.00 s internal ultrasonication
Pause time after ultrasonication	: 360.00 s
Pump speed	: 30.00 %
Number of measured partial samples	: 2
Measurements per partial sample	: 3
HELOS evaluation mode	: HRLD

Statistical evaluation of analytical data

HELOS (H0157) & QUIXEL. R3: 0.5/0.9...175µm (f = 100.00 mm)					
Product: Sand	Evaluation: HRLD (5.5.0.0)				
density: 1.00 g/cm ³	evaluation mode: HRLD (5.5.0.0)				
shape factor: 1.00	contamination: 0.00 %				
sample number: 90506.005					
Measuring condition: ref0, startbutton, mdu10s...	Dispersing method: 2mm.H2O.US180/360.pump30				
start condition: button	liquid: H2O				
stop condition: 10s meas.time or 20s real time	additive: Na4P2O7				
time base: 100.00 ms	ultrasonic: 180.00 s				
measuring duration: 10.00 (10.00 - 10.00) s	pause: 360.00 s				
opt. concentration: 19.99 (19.81 - 20.21) %	pump speed: 30.00 %				
	temperature: 30.00 °C				
	window spacing of the cuvette: 2.00 mm				
Preparation: Customer: First Institute of Oceanography					
sample splitting: no	operator: Hff				
amount of sample: 200 ml	database: HELOS 2009				
additional info: -					
Measurements:	Identifiers:				
2009-05-13 13:30:22.8230 0157 H	Sand_c005				
2009-05-13 13:30:34.3060 0157 H	Sand_c006				
2009-05-13 13:30:45.7120 0157 H	Sand_c007				
2009-05-13 13:51:34.4790 0157 H	Sand_d005				
2009-05-13 13:51:45.8540 0157 H	Sand_d006				
2009-05-13 13:51:57.2760 0157 H	Sand_d007				
upper band limit	pct. under cumulative	standard deviation	in band different	mean size for band (log)	distribution density (log)
x_{i+1} [µm]	Q_i [%]	abs. [%]	ΔQ_i [%]	x_{i+1} [µm]	q_i
0.90	1.79	0.1212	1.79	0.67	0.07
1.10	2.56	0.0272	0.77	0.99	0.09
1.30	3.28	0.0217	0.72	1.20	0.10
1.50	3.94	0.0287	0.67	1.40	0.11
1.80	4.67	0.0310	0.93	1.64	0.12
2.00	5.99	0.0371	1.12	1.99	0.13
2.50	6.99	0.0422	1.00	2.39	0.14
3.10	8.11	0.0476	1.12	2.84	0.15
3.70	9.30	0.0529	1.19	3.39	0.15
4.30	10.35	0.0573	1.05	3.99	0.16
5.00	11.45	0.0617	1.09	4.64	0.17
6.00	12.82	0.0668	1.37	5.48	0.17
7.50	14.59	0.0732	1.77	6.71	0.18
9.00	16.12	0.0786	1.53	8.22	0.19
10.50	17.52	0.0836	1.40	9.72	0.21
12.50	19.34	0.0901	1.82	11.46	0.24
15.00	21.66	0.0986	2.32	13.69	0.29
19.00	24.63	0.1096	2.97	16.43	0.37
21.00	27.79	0.1194	3.16	19.44	0.47
25.00	32.19	0.1270	4.40	22.91	0.58
30.00	37.77	0.1364	5.58	27.39	0.70
36.00	44.42	0.1398	6.65	32.86	0.84
43.00	52.12	0.1235	7.70	39.34	1.00
51.00	60.98	0.1493	8.75	46.83	1.19
61.00	71.38	0.1969	10.51	55.78	1.35
73.00	82.16	0.2434	10.78	66.73	1.38
87.00	90.94	0.2456	9.77	79.69	1.15
103.00	96.26	0.1632	9.32	94.66	0.73
123.00	98.96	0.2504	2.70	112.84	0.35
147.00	100.00	0.0000	1.04	134.47	0.13
175.00	100.00	0.0000	0.00	160.39	0.00
$x_{10} = 4.10 \mu\text{m} \pm 0.03 \mu\text{m}$	$x_{50} = 41.07 \mu\text{m} \pm 0.11 \mu\text{m}$	$x_{90} = 85.51 \mu\text{m} \pm 0.39 \mu\text{m}$			
$x_{16} = 8.88 \mu\text{m} \pm 0.08 \mu\text{m}$	$x_{84} = 75.93 \mu\text{m} \pm 0.39 \mu\text{m}$	$x_{99} = 123.99 \mu\text{m} \pm 1.01 \mu\text{m}$			
$Q_2 = 5.43 \% \pm 0.03 \%$	$Q_{50} = 17.05 \% \pm 0.08 \%$	$Q_{99} = 59.78 \% \pm 0.14 \%$			

Fig. 2: Measurement specification and statistical evaluation based on 6 measurements of the fraction < 63 microns

MEASUREMENT SPECIFICATION

Product name	: Sand
Sample preparation	: Separation of the coarse fraction with a 6.3 mm sieve wet sieving of the finer fraction (90.45 % < 6.3 mm) at 63 µm (60.06 % > 63 µm); sample splitting of the fraction between 63 µm and 6.3 mm with rotating riffler
Sensor	: QICPIC
Dispersing system	: GRADIS
Measuring range QICPIC	: 2.00 63.0 mm - M8 (20...6820µm)
Mass requirement	: 21.00 g
Start condition of measurement	: c.opt >= 0.01%
Stop condition of measurement	: 25s c.opt <= 0.01% oder 1300s Real time
Frame rate	: 100.00 Hz
Number of frames	: 110264.00 -127466.00
Number of measured particles	: 12471042.00 - 14501653.00
Measuring time	: 1102.91 s - 1275.01 s
Optical concentration	: 0.1847 % - 0.1887 %
Feed rate VIBRI	: 5.00 %
Funnel height	: 3.00 mm
Slit width GRADIS	: 10 mm
Number of measured partial samples	: 3
measurements per partial sample	: 1
QICPIC evaluation mode	: FERET_MIN
Class limits	: M8

Statistical evaluation of analytical data

QICPIC (Q0153) & GRADIS, 2.00 63.0 mm - M8 (20...6820µm)

Product: Sand		Evaluation:			
density:	1.00 g/cm ³	evaluation mode:	FERET_MIN (5.5.0.0)		
Sample number:	90506/005	number of particles:	13646843.33		
		class limits:	M8		
Measuring condition: 100Hz, copt0.01%,mea5-1300s...		Dispersing method: GR_50x10mm.feed5.gap3.vibri_c...			
frame rate:	100.00 Hz	doser:	VIBRI		
start condition:	c.opt >= 0.01%	feed rate:	5.00 %		
stop condition:	25s c.opt <= 0.01% oder 1300s Real time	gap width:	3.00 mm		
measuring duration:	1206.84 (1102.91 - 1275.01) s	height of drop:	50.00 cm		
opt. concentration:	0.1868 (0.1847 - 0.1887) %	additional info:	--		
Preparations:		Customer: First Institute of Oceanography			
sample splitting:	yes	operator:	TH		
amount of sample:	21.00 g	database:	QICPIC Januar 2009		
Measurements:		Identifiers:			
2009-05-25 10:19:45.7060 0153 Q		Sand_6.3mm_101			
2009-05-25 11:07:26.1240 0153 Q		Sand_6.3mm_102			
2009-05-25 13:04:51.8720 0153 Q		Sand_6.3mm_103			
upper band limit	pct. under cumulative	standard deviation	in band different	mean size for band (log)	distribution density (log)
x_{50} [µm]	Q_2 [%]	abs. [µm]	dQ_2 [%]	x_{50} [µm]	q_{50}
20.00	0.00	0.0000	0.00	14.14	0.00
43.52	0.36	0.0162	0.36	29.50	0.01
94.72	20.11	0.9559	19.75	64.21	0.58
115.04	42.54	1.6886	22.43	104.39	2.66
139.73	94.94	1.9674	12.40	126.79	1.47
169.71	60.88	2.0770	5.94	153.99	0.70
206.13	62.93	2.1121	2.05	187.03	0.24
250.36	64.31	2.1101	1.38	227.17	0.16
304.08	65.30	2.0866	0.99	275.91	0.12
369.32	65.93	2.0897	0.63	335.12	0.07
448.57	66.66	2.0686	0.73	407.02	0.09
544.83	67.80	1.9762	1.14	494.36	0.14
661.73	69.12	1.8733	1.33	600.44	0.16
803.73	70.49	1.8180	1.37	729.28	0.16
976.19	72.01	1.7869	1.52	885.77	0.18
1185.65	74.14	1.6167	2.13	1075.83	0.25
1440.07	77.38	1.3395	3.24	1306.68	0.38
1749.08	81.89	1.0857	4.53	1587.07	0.53
2124.39	86.38	1.0851	4.49	1927.62	0.53
2580.23	90.48	1.1955	4.11	2341.24	0.49
3133.89	92.95	1.6162	2.46	2843.62	0.29
3806.35	94.25	1.8932	1.30	3453.79	0.15
4623.11	96.32	0.9613	2.07	4194.90	0.25
5615.12	99.10	1.5526	2.79	5095.03	0.33
6820.00	100.00	0.0000	0.90	6188.31	0.11
$x_{10} = 68.55 \mu\text{m} \pm 1.26 \mu\text{m}$	$x_{50} = 129.96 \mu\text{m} \pm 3.73 \mu\text{m}$	$x_{90} = 2574.35 \mu\text{m} \pm 211.86 \mu\text{m}$			
$x_{16} = 84.13 \mu\text{m} \pm 2.02 \mu\text{m}$	$x_{84} = 1925.30 \mu\text{m} \pm 91.98 \mu\text{m}$	$x_{98} = 5662.79 \mu\text{m} \pm 619.06 \mu\text{m}$			

Fig. 3: Statistical evaluation based on 3 measurements of the fraction between 63 microns and 6.3 mm

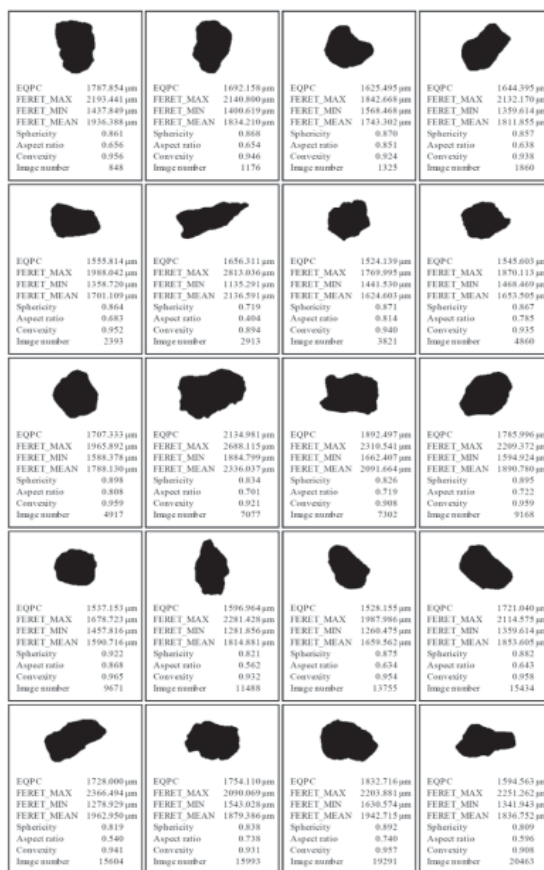


Fig. 4: Image analysis, fraction between 63 µm and 6.3 mm: Excerpt of 20 particles out of 106 particles with EQPC > 1500 µm; total amount of measured particles: 157605

As a consequence of the measurements the sample was identified as a silty sand with a minor amount of clay and a reasonable fraction of fine gravel.

3 Conclusions

For measurements of the particle size distribution of broadly distributed sediments and soil samples a separation in fine and coarse fractions by sieve procedures is necessary. As classic measuring methods sedimentation techniques for the finest fraction and sieve procedures for the coarser fractions are mandatory. In terms of sedimentation techniques, the disadvantages like long measuring times and an overestimation of the finest fraction are reported since several years. Modern measuring techniques are able to overcome these problems, but a correlation between classic and modern methods is often not feasible.

Merged curve of the sample “Sand”

measuring system : HELOS/QUIXEL, QICPIC/GRADIS, sieve: Merged curve

	x_0	Q_3	x_m	q_3^*
	μm	%	μm	
1	0,90	0,72	0,67	0,03
2	1,10	1,02	0,99	0,04
3	1,30	1,31	1,20	0,04
4	1,50	1,58	1,40	0,04
5	1,80	1,95	1,64	0,05
6	2,20	2,39	1,99	0,05
7	2,60	2,79	2,39	0,06
8	3,10	3,24	2,84	0,06
9	3,70	3,72	3,39	0,06
10	4,30	4,14	3,99	0,06
11	5,00	4,57	4,64	0,07
12	6,00	5,12	5,48	0,07
13	7,50	5,83	6,71	0,07
14	9,00	6,44	8,22	0,08
15	10,50	7,00	9,72	0,08
16	12,50	7,73	11,46	0,10
17	15,00	8,65	13,69	0,11
18	18,00	9,84	16,43	0,15
19	21,00	11,11	19,44	0,19
20	25,00	12,90	22,91	0,24
21	30,00	15,18	27,39	0,29
22	36,00	17,89	32,86	0,34
23	43,52	21,27	39,58	0,41
24	94,72	49,43	64,21	0,83
25	115,04	64,65	104,39	1,80
26	139,73	72,81	126,79	0,97
27	169,71	76,51	153,99	0,44
28	206,13	77,74	187,03	0,15
29	250,36	78,57	227,17	0,10
30	304,08	79,16	275,91	0,07
31	369,32	79,54	335,12	0,04
32	448,57	79,98	407,02	0,05
33	544,83	80,66	494,36	0,08
34	661,73	81,46	600,44	0,09
35	803,73	82,28	729,28	0,10
36	976,19	83,19	885,77	0,11
37	1185,65	84,47	1075,83	0,15
38	1440,07	86,42	1306,68	0,23
39	1749,08	89,13	1587,07	0,32
40	2124,39	91,82	1927,62	0,32
41	2580,23	94,29	2341,24	0,29
42	3133,89	95,76	2843,62	0,18
43	3806,35	96,55	3453,79	0,09
44	4623,11	97,79	4194,90	0,15
45	5615,12	99,46	5095,03	0,20
46	6300,00	100,00	5947,71	0,06
47	30000,00	100,00	13747,73	0,00

$x_{10} =$	20,81 μm	$x_{50} =$	102,37 μm	$x_{90} =$	5502,29 μm
$Q_2 =$	1,97%	$Q_{63} =$	28,99%	$Q_{2000} =$	82,43%

Fig. 5: Merged curve

Merged and linked result of the Sample “Sand”

measuring system : HELOS/QUIXEL, QICPIC/GRADIS, sieve: Merged and linked curve

	x_0	Q_3	x_m	q_3^*
	μm	%	μm	
1	0,90	0,65	0,67	0,03
2	1,10	0,93	0,99	0,03
3	1,30	1,19	1,20	0,04
4	1,50	1,43	1,40	0,04
5	1,80	1,76	1,64	0,04
6	2,20	2,17	1,99	0,05
7	2,60	2,53	2,39	0,05
8	3,10	2,94	2,84	0,05
9	3,70	3,37	3,39	0,06
10	4,30	3,75	3,99	0,06
11	5,00	4,14	4,64	0,06
12	6,00	4,64	5,48	0,06
13	7,50	5,28	6,71	0,07
14	9,00	5,84	8,22	0,07
15	10,50	6,34	9,72	0,08
16	12,50	7,00	11,46	0,09
17	15,00	7,84	13,69	0,11
18	18,00	8,92	16,43	0,14
19	21,00	10,07	19,44	0,17
20	25,00	11,70	22,91	0,21
21	30,00	13,76	27,39	0,26
22	36,00	16,22	32,86	0,31
23	43,52	19,28	39,58	0,37
24	94,72	44,81	64,21	0,76
25	115,04	58,60	104,39	1,63
26	139,73	66,01	126,79	0,88
27	169,71	69,36	153,99	0,40
28	206,13	70,47	187,03	0,13
29	250,36	71,23	227,17	0,09
30	304,08	71,77	275,91	0,06
31	369,32	72,11	335,12	0,04
32	448,57	72,50	407,02	0,05
33	544,83	73,13	494,36	0,07
34	661,73	73,85	600,44	0,09
35	803,73	74,59	729,28	0,09
36	976,19	75,42	885,77	0,10
37	1185,65	76,58	1075,83	0,14
38	1440,07	78,35	1306,68	0,21
39	1749,08	80,80	1587,07	0,29
40	2124,39	83,24	1927,62	0,29
41	2580,23	85,48	2341,24	0,26
42	3133,89	86,82	2843,62	0,16
43	3806,35	87,53	3453,79	0,08
44	4623,11	88,66	4194,90	0,13
45	5615,12	90,17	5095,03	0,18
46	6300,00	90,45	5947,71	0,06
47	30000,00	100,00	13747,73	0,14

$x_{10} =$	20,81 μm	$x_{50} =$	102,37 μm	$x_{90} =$	5502,29 μm
$Q_2 =$	1,97%	$Q_{63} =$	28,99%	$Q_{2000} =$	82,43%
Clay (fraction < 2 microns)	1,97%	Silt (fraction 2- 63 microns)	27,02%	Sand (fraction 63- 2000 microns)	53,48%

Fig. 6: Merged and linked curve

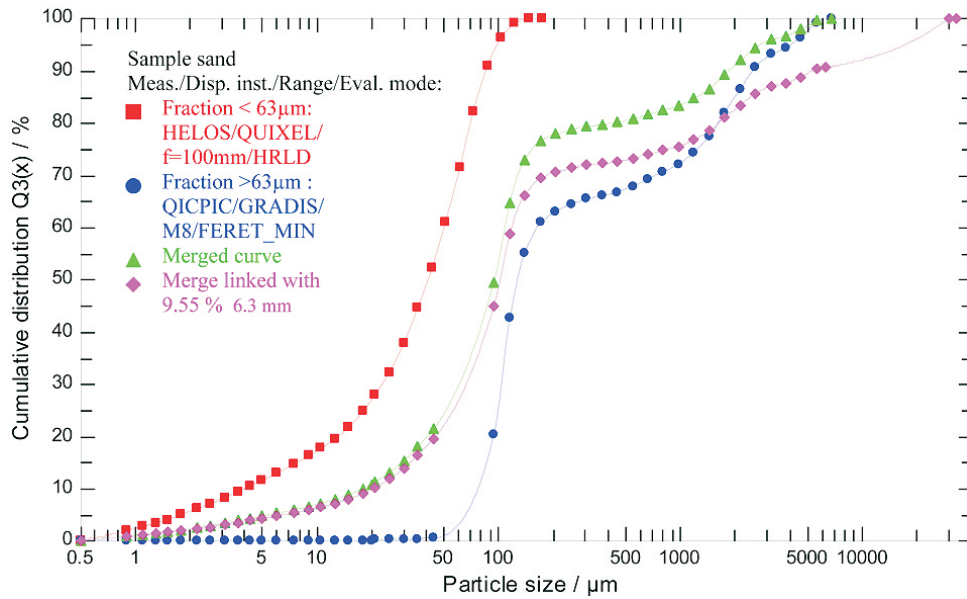


Fig. 7: Cumulative distribution

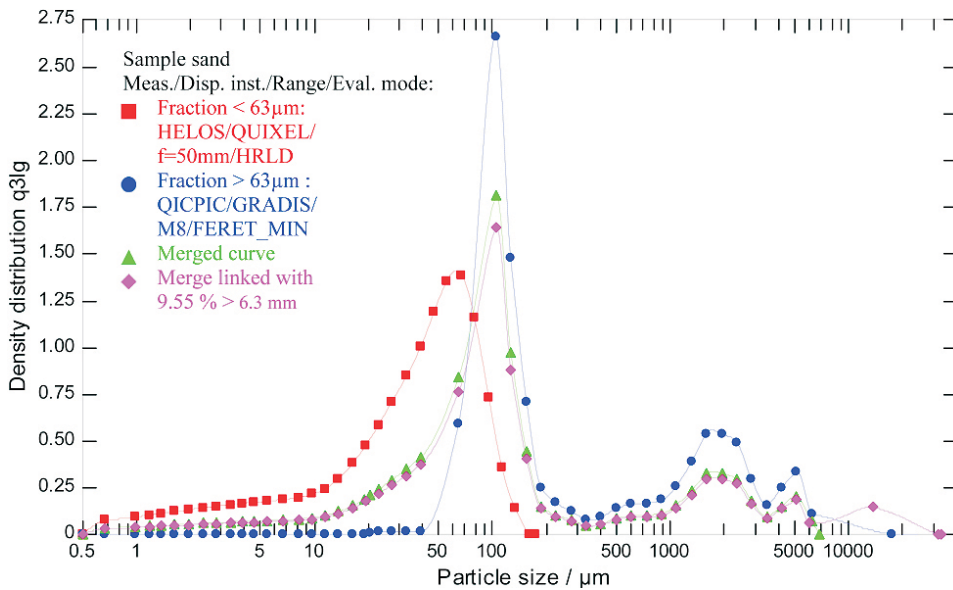


Fig.8: Density distribution curve q_{3lg}

The proposed step by step procedure in combination with the presentation of results will introduce the measurement of the particle size distribution and the characterisation of the shape with modern measuring techniques. The duration of the procedure is much shorter than the classic one and the result is not any longer based on a sedimentation method with all the oddities caused by incorrect assumptions of shape and density of the particles.

4 Bibliography

- SCHEFFER, F. & SCHACHTSCHABEL, P. (2002): Lehrbuch der Bodenkunde.- 15. Aufl.; 593 p.
- ISO 11464 (2006): Soil quality – Pretreatment of samples for physico-chemical analyses.- 15 p.
- DIN 19683 Blatt 2 (1973): Bodenuntersuchungsverfahren im Landwirtschaftlichen Wasserbau, Physikalische Laboruntersuchungen, Bestimmung der Korngrößenzusammensetzung nach Vorbehandlung mit Natriumpyrophosphat.- 3 p.

- DIN 18123 (1971): Baugrund, Untersuchung von Bodenproben, Korngrößenverteilung.- 9 p.
- DIN 66115 (1983): Partikelgrößenanalyse, Sedimentationsanalyse im Schwerefeld, Pipette-Verfahren.- 8 p.
- DIN ISO 11277 (2000): Soil quality: Determination of particle size distribution in mineral soil material: Method by sieving and sedimentation following removal of soluble salts, organic matter and carbonates.- 44 p.
- RÖLLIG, G. (1971): Verwitterung.- In: Autorenkollektiv (Hrsg): Die Entwicklungsgeschichte der Erde.- Band I, Nachdruck 1974, 535 p.; Hanau (Dausien).
- HATHAWAY, J. C. (1979): Clay Minerals, in: Reviews in Mineralogy.- Mineralogical Society of America, **6**: Marine Minerals, 380 p.; Department of Geological Sciences, Virginia Polytechnic Institute and State University
- DIN 13220-1 (1999): Particle size analysis – Laser diffraction methods – Part 1: general Principles.- 33p.
- BERTHOLD, C., LÜHMANN, J., KLEIN, R. & NICKEL, G. (2000): Particle measurement techniques compared – Tests on the grain size determination of clayey raw materials.- *Aufbereitungs Technik* **41,7**: 310-318.
- BEUSELINCK, L., GOVERS, G., POESEN, J., DEGRAER, G. & FROYEN, L. (1998): Grain-size analysis by laser diffractometry: comparison with the sieve-pipette method.- *Catena* **32**: 193-208 .