

Improvement of grain-size analyses using the automated SEDIGRAPH 5100

by Stanislas WARTEL *, Jean-Paul BARUSSEAU ** & Luc CORNAND *

* Section of Mineralogy and Petrography
Royal Belgian Institute of Natural Sciences
Vautier Street, 29
B-1040 BRUSSELS, BELGIUM

** Laboratoire de Recherches de Sédimentologie Marine
Université de Perpignan
Avenue de Villeneuve
F-66025 PERPIGNAN, FRANCE

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ABSTRACTS : The method of analysing fine grained sediments is discussed and an improved technique using a water-glycerol solution of known density instead of water is proposed. Inaccuracies resulting from wet sieving may contribute to explain the observed difference between the pipette method and the Sedigraph 5100 analyses.

Key words : sedimentology, granulometry, fine-grained sediments

RESUME : La méthode d'analyse des sédiments très fins est discutée et une méthode améliorée utilisant une solution aqueuse de glycérol de densité connue est proposée. Des erreurs dues au tamisage sous eau peuvent expliquer la différence observée entre la méthode de la pipette et les analyses effectuées à l'aide du Sedigraph 5100.

Mots clés : sédimentologie, granulométrie, sédiments fins

Introduction

Grain-size characteristics are frequently used in view to interpret the sedimentary dynamic processes in recent and ancient sedimentary deposits. For instance the relationship between clay content and geotechnical or geochemical properties of a sediment is well known and may be very useful for scientific or applied purposes. The utility, however, of such a correlation strongly depends on the precision and the accuracy of the results obtained.

Since the use of automated instruments, among which the SEDIGRAPH, for measuring the size of sedimentary particles has now become widespread, several authors have compared the results obtained with these instruments with the results of classical techniques based on the sedimentation of particles in water. Stein (1985), Singer et al. (1988) and Syvitsky et al. (1990) showed that the SEDIGRAPH gives results that are both accurate and highly reproducible. The individual modes in polymodal samples are correctly determined and correctly measured at sample concentrations < 2 vol%. For higher concentrations a small shift (0.25ϕ) in the mode may occur (Singer et al., 1988). A striking outcome of these studies was that for natural clay-sized samples more clay (20% or more) was detected by the SEDIGRAPH than was detected by decanting each sample (Stein, 1985; Singer et al., 1988). The authors related this higher clay content to the use of high volume concentrations (2 to 3 vol. %), and the resulting particle-to-particle interactions or hindered settling. A similar trend was also observed by Syvitsky et al. (1990) who mentioned furthermore that the SEDIGRAPH showed a tendency to spread out the sample's grain-size distribution and provided results that were too fine-grained. These divergences, that affect not only the clay fraction ($< 2 \mu\text{m}$) but also the silt fraction (63 to $2 \mu\text{m}$), are relatively important. Indeed, the grain-size distribution in the silt fraction is often used as an environmental indicator. Also small variations in the hydrographic regime may cause detectable changes in the silt fraction. An accurate knowledge of this part of the grain-size spectrum is thus of primary importance. Since older analytical techniques based on the Atterberg method are unable to produce reliable results (Syvitsky et al., 1990) further improvement of the methodology using the more accurate and reliable modern instruments (SEDIGRAPH, Malvern Laser Sizer, ...) must thus be attempted.

It appears from literature that one of the major problems encountered in grain-size analyses based on Stokes's law comes from particle-to-particle interactions and hindered settling. Both phenomena are related to frictional forces and thus to the viscosity of the fluid. Collisions between particles settling through a fluid result from the difference in settling velocity on the one hand and from Brownian motion on the other hand (Einstein & Krone, 1962). It follows that in a liquid with higher viscosity the decrease of the settling velocities and the suppression of the Brownian motion will result in a proportional reduction of the number of collisions between particles. Therefore it can be safely assumed that using a fluid with a viscosity higher than that of water will improve the accuracy of the SEDIGRAPH analyses. This study deals with different

series of tests that were performed to check the accuracy of the SEDIGRAPH using a suspending medium (glycerol solution) denser than water and to compare the results so obtained with a classical sedimentation technique (Atterberg method) using water as the suspending medium.

Methods

Sample preparation

For each test a large bulk sample was lyophilised and after homogenisation split into several sub-samples that were further prepared for grain-size analyses. The organic matter was removed using 30% diluted technical H_2O_2 as an oxidising agent. Afterwards the carbonates were removed using a HCl 1N solution. On completion of the removal of organic matter and carbonates, the sample was rinsed using demineralized water until a more or less stable suspension was obtained. The sample was then oven-dried at $105^\circ C$ and weighed. The dry sample was brought into suspension using 100 ml of demineralized water with 5 ml of a peptising agent (1.33 g $NaCO_3$ and 8.93g Na-oxalate in 1 litre of water) added. The suspension was stirred using a magnetic stirrer for at least one hour and, for the samples of test 5 to 8, further dispersed in an ultrasonic bath for approximately 5 minutes. The prepared sub-sample was then wet sieved using a FRITSCH ANALYSETTE vibrating sieving apparatus. The fine fraction was concentrated by slow evaporation on a hot plate (approximately $60^\circ C$) in order to avoid complete drying of the sample, and stored in a closed container. The container was rotated continuously to prevent settling of the sediment before being further analysed. Before each analyses with the SEDIGRAPH the suspension was stirred for 9 minutes followed by stirring together with ultrasonic dispersal for 1 minute unless otherwise stated.

The operational characteristics of the SEDIGRAPH have been described in Stein (1985) and Jones et al. (1988). The instrument determines the size distribution of particles dispersed in a liquid assuming settling of particles according to Stokes's law. Analyses were performed using demineralized water or a 50 weight % mixture of glycerol and water with a density of 1.12 and a viscosity of 3.696 cp. The X-ray absorption coefficients of the liquid and of the particles are assumed to be constant for the suspension components. In this study an interval of $1/4 \phi$ for the sieves as well as for the SEDIGRAPH was used.

Discussion

1. Measurement error.

The measurement error of the SEDIGRAPH was tested by performing repeated analyses on a mud sample (91B05, test 1) from the Schelde estuary, Belgium, that contained approximately 40% of sediment finer than $2 \mu m$ (clay). The prepared clay sample was wet sieved at $32 \mu m$ and the finer fraction was brought into suspension with a concentration of approximately 9 g/l. The time span between successive analyses varied from a fraction of an hour to several days. In

between the analyses the samples were continuously rotated to prevent the suspension from settling. The results are summarised in table 10 (in annex).

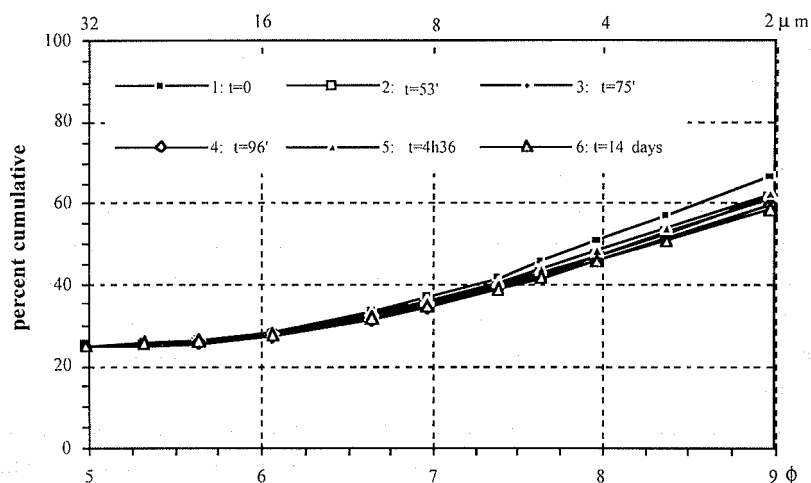


Figure 1 : Cumulative grain-size distribution of repeated analyses of sample 91B05 (test 1).

The measured clay content ($>9 \phi$ or $<2 \mu\text{m}$, figure 1) of the sample ranged between 37.5 and 41.2%, only one analysis (run 1) gave a lower clay content of 33.5%. The average clay content was 38.3% with a the standard deviation of 2.7%.

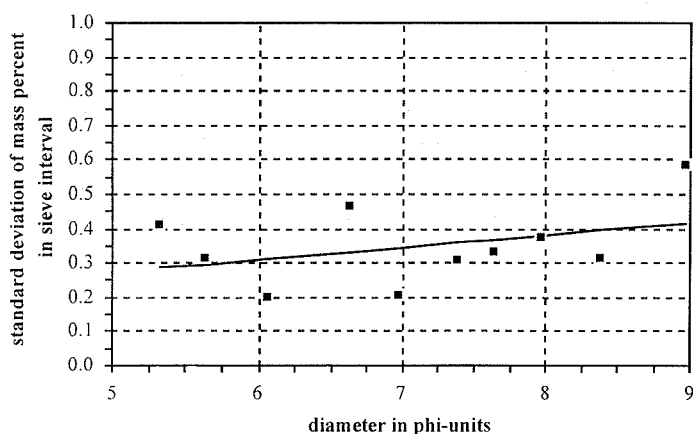


Figure 2 : Standard deviation per grain-size interval for the repeated analyses of sample 91B05 (test 1).

The standard deviation for the concentration of particles in each grain-size interval ranged between 0.2 and 0.5% showing an average of 0.3%. The standard deviation increases slightly from the coarser to the finer fractions (figure 2).

2. Comparison between analyses in water and in glycerol solution

A prepared sample (Boom Clay) (test 2, table 11) was wet sieved at $63\mu\text{m}$ and analysed a first time using water, with 5 weight-percent of a peptising agent added (run 41), as the suspending medium and afterwards, after drying and resuspending, using a 50% by weight glycerol solution (run 42, 46 and 49). The average clay content for the duplicate runs in water was 21.2% with a standard deviation of 0.3%. This very low standard deviation can be attributed to the fact that only 2 analyses were carried out. The average clay content using a glycerol solution was 27% with a standard deviation of 3.8%. It can be observed that the standard deviation using a glycerol solution does not vary significantly from the standard deviation of 2.7% detected for sample 91B05 (test 1) using water as the suspending medium.

The analyses also indicate that the clay content increases with increasing rotation time (table 11). One of the analyses using glycerol solution (run 42) was subjected to the same rotation time as the analyses performed with water (run 41). Both analyses gave the same clay content (21.2%). An increase in clay content with increasing time of rotation was also observed for sample 91B05 (table 10) for which the clay content stabilized after approximately 100 minutes (figure 3). In all these cases the increase in clay content may be the result of a better dispersal due to either a longer rotation time or a repeated dispersal with ultrasonic vibrations as will be shown later on the hand of SEM analyses (test 7).

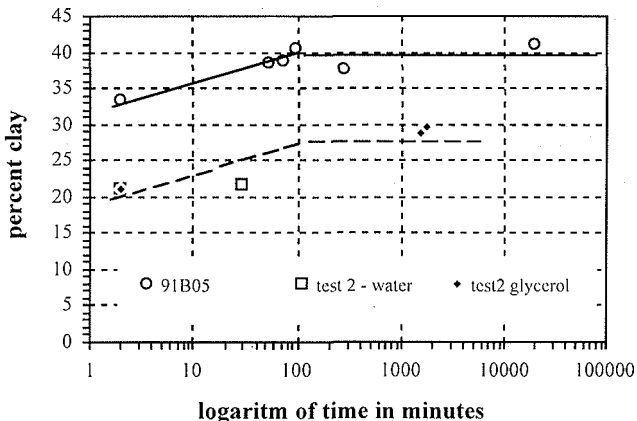


Figure 3 : Clay content as a function of rotation time

In a general way it can be observed that the difference in clay content detected between the analyses using water and using a water-glycerol mixture does not exceed the standard deviations detected for tests 1 and 2 and thus is not really significant. So it can be concluded that the use of a glycerol solution as the suspending medium does not affect the measured clay content to a significant degree. The rotation time, however, has probably a much more important effect. If it is

taken too short the measured clay content may be too low as a result of incomplete dispersal of the sample. This was already observed by Stein (1985) who suggested that a minimum of 15 minutes of ultrasonic treatment should be applied.

For the analyses performed with a glycerol solution the average mean was $8.92 \phi (\pm 0.17 \phi)$, the average sorting was 7.18ϕ -units ($\pm 0.04 \phi$), the average skewness was 0.42ϕ -units ($\pm 0.06 \phi$) and the average kurtosis was $0.94 \phi (\pm 0.03 \phi$ -units).

3. Effect of the sieve-size used for wet sieving.

Using a dense glycerol solution has the advantage that the maximum grain-size that can be analysed with the SEDIGRAPH increases to $106 \mu\text{m}$ for the solution used here. Therefore a test (test 4) was performed to examine the effect on the grain-size distribution of different sieve diameters used for wet sieving. A bulk clay sample was split into 4 parts, numbered A to D, each of which was split into 3 sub-samples that were prepared as described above. The samples of the C-series were not further analysed and will thus not be considered here. Test 4-A samples were wet sieved at $106 \mu\text{m}$ and SEDIGRAPH analyses were made in duplicate. The samples of test 4-B were wet sieved at $76 \mu\text{m}$ (and were analysed 3 times. The first analyses of each set was performed immediately after rotation, for the second analyses rotation was stopped one hour before the analyses and for the third analyses rotation was stopped 24 hours before the analyses. The mixing time in the SEDIGRAPH was kept constant at 3 minutes except for the third analyses of test 4-B samples for which the mixing time was doubled. The ultrasonic dispersion in the MASTERTECH was kept constant at 1 minute, coinciding with the last minute of mixing. A summary of the data is given in table 12 (in annex). Test 4-D samples were wet sieved at $32 \mu\text{m}$ and the finer fraction was analysed using a sedimentation technique.

TEST 4	number of runs	% clay 1 ^o run	average % clay all runs	standard deviation runs 1 and 2
A1	2	29.6	30.2	0.8
A2	2	37.6	37.8	0.4
A3	2	36.8	37.0	0.2
A - average		34.7	35.0	
A - st. dev.		4.4	3.8	
B1	2	34.8	33.0	2.6
B2	2	31.3	29.6	2.4
B3	2	31.2	29.8	2.0
B - average		32.4	30.8	
B - st. dev.		2.1	2.5	
A+B average		33.6	32.9	
A+B st. dev.		1.6	2.8	

Table 1 : Average clay content and standard deviation of test 4 samples

The average clay content of the A-series (first analysis only) is 34.7% with a standard

deviation of 4.4% (table 1). The high standard deviation results from the very low clay content detected for sample A1 and is much lower (1.1) if only samples A2 and A3 are considered. The standard deviation for duplicate analyses ranges between 0.2 and 0.8 and is lower than in the previous tests. The deviating result obtained for subsample A1 seems to indicate that this sample was different from the others, probably as a result of insufficient homogenisation before subsampling.

The sub-samples of the B-series show a clay content with an average of 32.4% (± 2.1) that is 2% lower than the average clay content of the A-series but within the limits given by the standard deviation for the sample of the A-series. Duplicate analyses of B-series samples show a larger standard deviation that ranges between 2.0 and 2.6. In the third analyses of B-series samples, performed after 24 hours of rest, a much lower clay content of 28.9% (± 0.3) is found.

The average values and their standard deviation, expressed in phi units, for the grain-size parameters are given in table 2. It can be seen that the mean is strongly affected by the clay content, test 4-B samples having a much finer mean than test 4-A samples. The sorting, skewness and kurtosis are not affected.

parameter	test 4-A	test 4-A2,3	test 4-B	test 4-B1,2
mean	4.8 (± 1.18)	4.0 (± 0.10)	7.5 (± 1.10)	7.0 (± 1.05)
sorting	4.8 (± 0.14)	4.8 (± 0.04)	4.7 (± 0.11)	4.7 (± 0.11)
skewness	0.6 (± 0.14)	0.5 (± 0.02)	0.8 (± 0.11)	0.8 (± 0.10)
kurtosis	2.1 (± 0.24)	1.9 (± 0.03)	2.4 (± 0.21)	2.3 (± 0.20)

Table 2: Average values and standard deviation for grain-size parameters of test 4 samples. For test 4-A2,3 the first analysis and for B1,2 the third analysis are not considered.

A comparison of the grain-size spectra (figures 4 to 6) indicates that the spectra for A-series samples overlap with these for the B-series samples. However, it struck that the sub-samples of the B-series, wet-sieved at 76 μm , have systematically more particles in the fraction 93-76 μm than the samples of the A-series, wet-sieved at 106 μm . In the example given in figure 4 (samples 4-A2.1 and 4-B1.1) the difference is 3% which corresponds fairly well to the observed 3% difference in clay content between both samples (table 12). It appears thus that finer particles, not passing the 76 μm sieve during the wet sieving process, account for the observed difference in clay content.

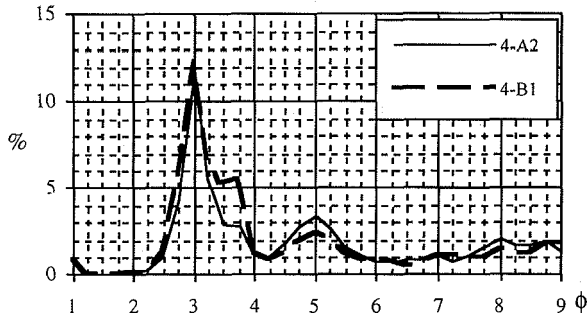


Figure 4 : Grain-size spectra of test 4-A2 and -B1 samples

Next to a retention of fine particles on the sieve used for wet sieving also agglomeration of fine particles or floc forming should be considered. The second and third analyses of the B-series, performed respectively after 1 hour and after 24 hours of rest, show systematically a lower clay content (up to 5%) than the first analyses. From the grain-size spectra of the first analysis (figure 5) it can be seen that in the fine sand and silt fractions two broad highs occur respectively around $32\ \mu\text{m}$ ($5\ \phi$) and, less pronounced, around $4\ \mu\text{m}$ ($8\ \phi$). These highs are separated by a low around $8\ \mu\text{m}$ ($7\ \phi$). For the second and third analyses the high at $32\ \mu\text{m}$ is visibly broadened and the low is much less pronounced compared to the first analyses. A similar pattern is observed in the spectra of samples from the A-series. The spectrum of sample A1 (figure 6), which was mixed only for a short time (5 minutes) and showed a relatively low clay content, also shows a broadened high around $32\ \mu\text{m}$ as compared to the spectra of samples A2 and A3. These results thus suggest that the lower clay content detected for some analyses is compensated by a larger amount of silt-sized particles, what most probably can be explained by the aggregation of particles after a prolonged period of rest and an incomplete resuspension in the MASTERTECH. With respect to this there seems to be not much difference between a period of rest of one hour (second analysis) and of 24 hours (third analysis).

The average particle concentration for each sieve, SEDIGRAPH or sedimentation fraction was calculated for all analyses of test 4-A and -B samples. The standard deviation on these averages are plotted against the average concentrations in figure 7. A weak correlation exists ($r^2 = 0.5$) between the percentage of particles present in a given fraction and the standard deviation for that fraction. The standard deviation per fraction exhibits a larger variation for the sedimentation analyses than for the SEDIGRAPH analyses, indicating that the SEDIGRAPH analyses are more reliable. From these data it can also be calculated that the relative error (standard deviation per unit of concentration) for each fraction is roughly between 8% and 25%.

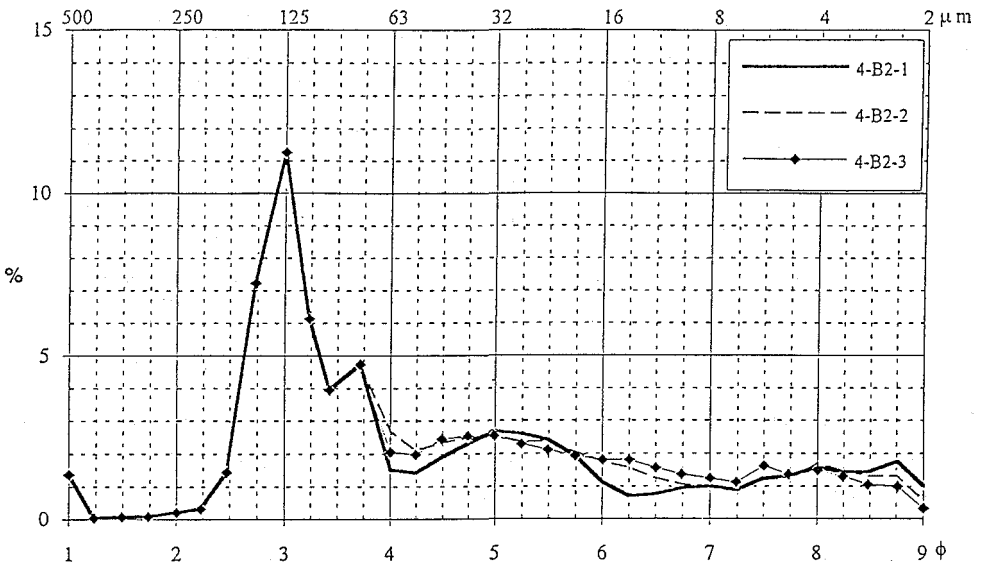


Figure 5 : Grain-size spectra of test 4-B2 samples, wet sieved at 3.72 phi.

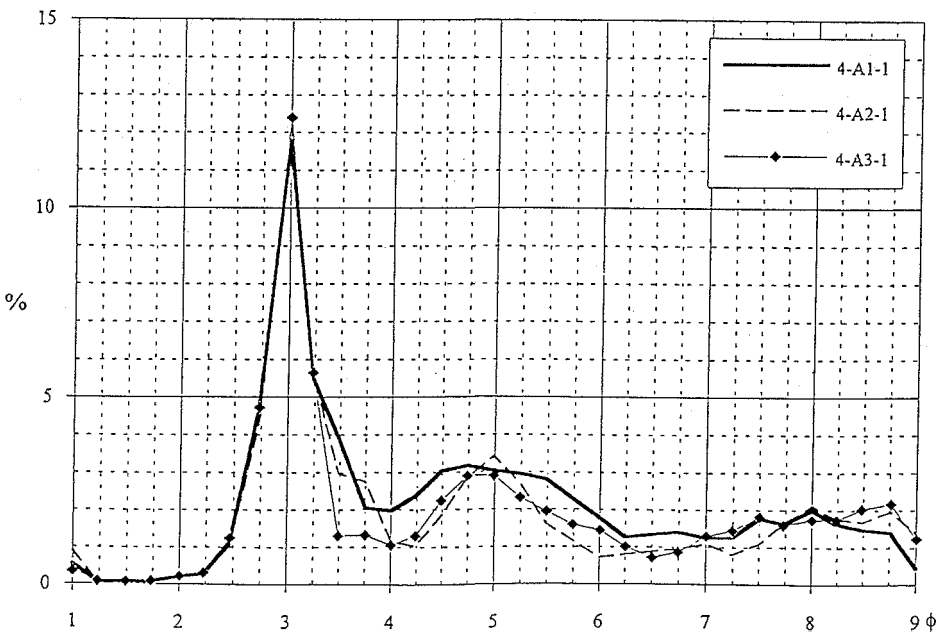


Figure 6 : Grain-size spectra for test 4-A samples, wet sieved at 3.25 phi.

fraction		samples 4-D %	samples 4-A %	difference %
phi	μm			
3.25	105	6.94	5.61	1.33
3.45	92	6.91	2.39	4.52
3.71	76	9.52	2.11	7.41
4.00	62	7.99	1.70	6.25
5.00	32	8.91	10.68	-1.77
total				16.45
< 9	< 2	19.73	34.70	-14.97

Table 3 : Comparison of the wet sieved fractions and the SEDIGRAPH fractions.

The test 4-D samples, analysed using a sedimentation technique, gave an average clay content of 20% (table 3) which is 15% less than the average clay content of 35% detected for the test 4-A samples, analysed with the SEDIGRAPH.

A comparison of the log-normal grain-size distribution curves of 4-D samples with sample 4-A2 (figure 8) shows that between 62 μm and 2 μm all curves are roughly parallel. However, the sedimentation curves diverge strongly from the SEDIGRAPH curve in the range 92 μm to 63 μm . The difference between the sedimentation and SEDIGRAPH analyses is given in table 3. It can be seen that the difference is especially important between 92 μm and 63 μm and is less important at 32 μm . The algebraic sum of the differences is 16.45% i.e. the sieve fractions gives 16.45% more particles in the grain-size interval between 92 and 32 μm than does the SEDIGRAPH. This value closely approximates the difference in clay content (15%) observed between the samples. Therefore it is suggested here that for these very fine sands sieving appears to be incomplete in the sense that particles finer than the sieve opening are retained on each sieve so that the grain-size distribution is biased resulting in a lower clay content.

From these tests it can be concluded that a difference in clay content may result from insufficient mixing of the sample and/or from incomplete wet-sieving. The sieve-size used for wet sieving must be taken as large as possible. In practice this size will be determined by the largest size that can be analysed with the SEDIGRAPH and thus by the density of the suspending medium. Using liquids denser than water thus will improve the precision of the analyses.

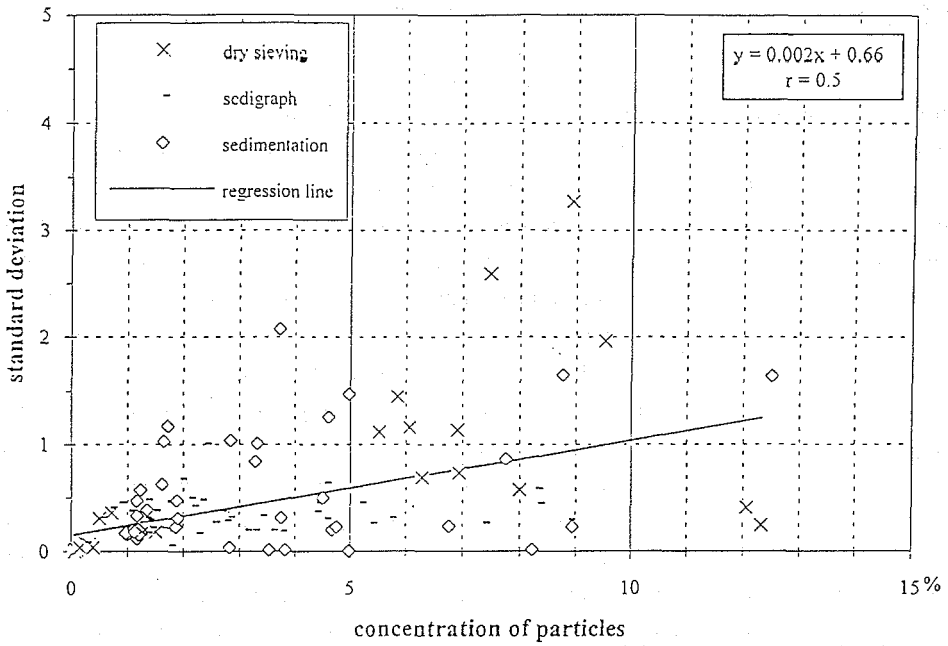


Figure 7 : Standard deviation for the concentration of particles in separate size-fractions

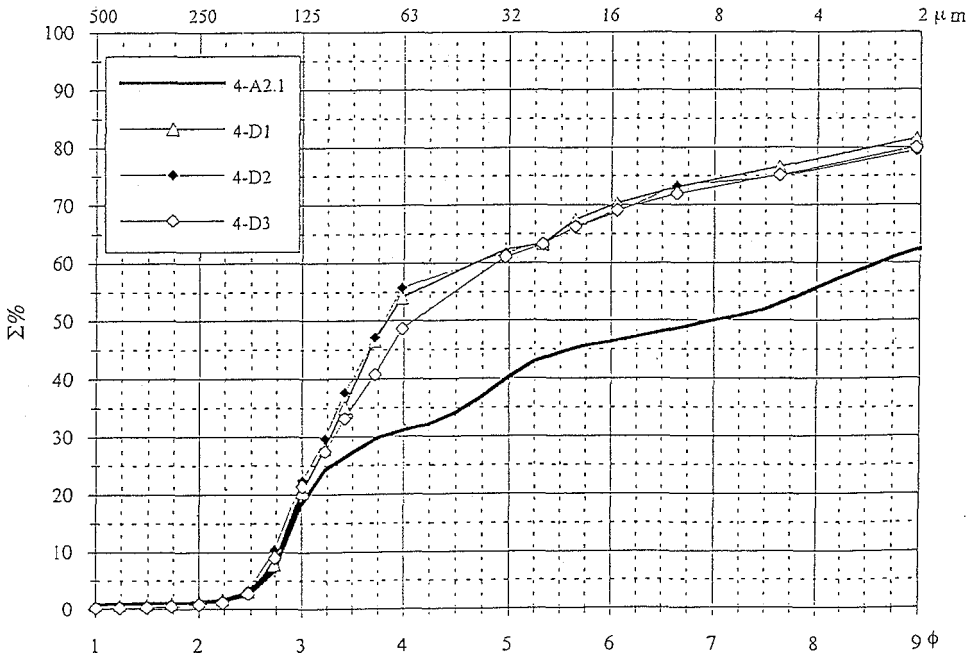


Figure 8 : Grain-size spectra for test 4-A2 and 4-D samples.

4. Effect of rotation time and mixing in the MASTERTECH

The MASTERTECH is a carrousel supporting up to 18 samples in suspension which are successively analysed by the SEDIGRAPH after being resuspended for a pre-defined time. A series of tests was performed to check an eventual effect of rotation time preceding the transfer of the sample to the MASTERTECH the effectiveness of resuspension in the MASTERTECH (several minutes of stirring and 1 minute of ultrasonic dispersion) even after a prolonged period of rest, called here the "dead time". A large sample was prepared and, after homogenisation, split into 8 parts numbered A to H. Each part was further subdivided into 3 sub-samples A1, A2, A3, B1, ... Special care was taken for the homogenisation of the sample in order to avoid the problem that arises with some samples test 4. Each of the sub-samples A to C was then analysed in duplicate or in threefold. The effect of mixing was tested on a relatively high concentrated suspension (50g/l) so that resuspension was made more difficult than for normal analyses, performed with lower concentrations. Each sub-sample was exposed to a different rotation time before being transferred to the MASTERTECH, to a different "dead time" and to different mixing times in the MASTERTECH. The ultrasonic dispersion in the MASTERTECH was kept constant at 1 minute, coinciding with the last minute of stirring. The first set of sub-samples (test 5-A) was not rotated before being transferred to the MASTERTECH. The second set (test 5-B) was rotated for 24 hours and the third set (test 5-C) was rotated for 48 hours. A summary of the results obtained in test 5 is given in table 13 (in annex).

TEST 5			
run series	number of runs	clay content average	clay content standard deviation
A	3	54.86	1.57
B	3	55.49	0.60
C	3	54.56	1.45
A+B+C	9	54.97	1.12
SED	3	40.64	1.94

Table 4 : Summary of test5 A to C analyses and of sedimentation (SED) analyses.

The average clay content for sub-samples of test 5-A, B and C is 54.97% with a standard deviation of 1.12% (table 4) which is better than the results obtained in the previous tests. When the samples are grouped according to the "dead-time" in the MASTERTECH (table 5) it can be seen that a dead-time of up to 60 minutes had no significant effect on the clay content. Sub-samples that had experienced stirring for only 5 minutes showed a somewhat lower clay content. No difference can be seen between samples that were stirred for 10 or for 20 minutes, whatever the rotating time or the "dead-time" was.

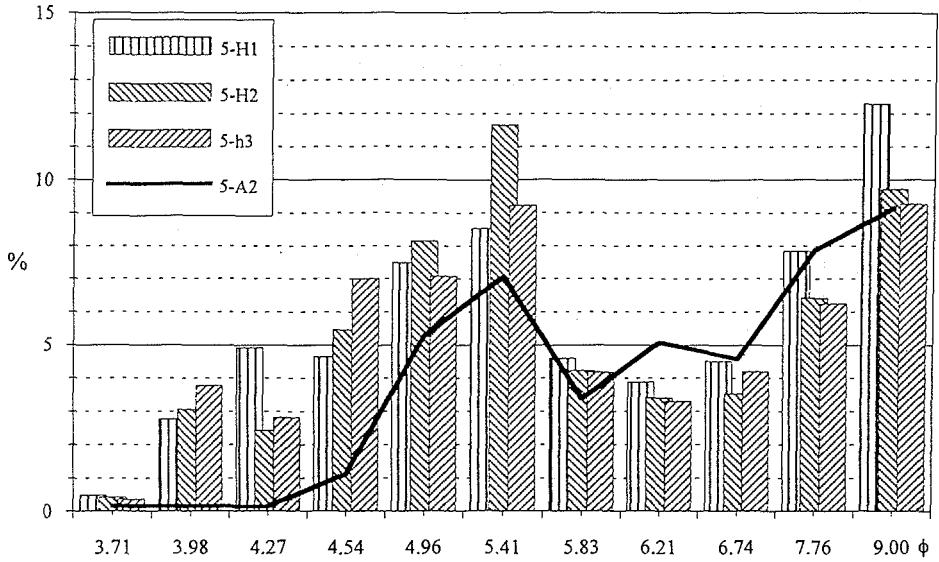


Figure 9 : Grain-size spectra for test 5-A and 5-H samples.

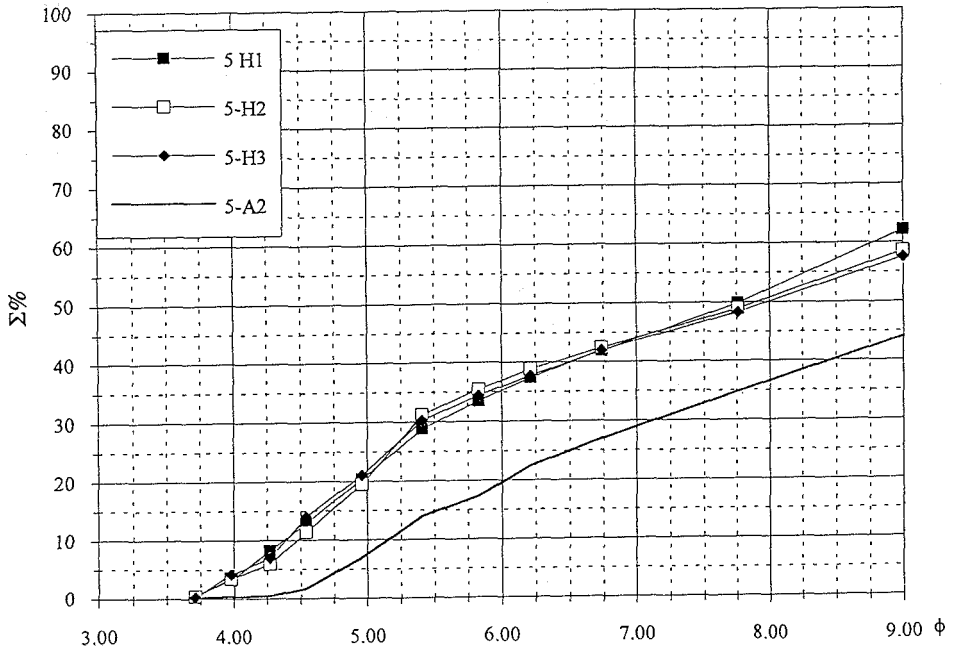


Figure 10 : Cumulative grain-size distribution of test 5-A2 and 5-H samples.

TEST 5				
sub-sample	rotating time	dead-time	stirring	%clay
A1	0	0	5	53.05
C1	48	5	5	54.18
A2	0	10	10	55.83
A3	0	20	20	55.71
C3	48	20	20	53.33
C2	48	32	10	56.16
B3	24	34	20	56.01
B1	24	60	5	54.84
B2	24	64	10	55.64

Table 5 : Average clay content of test 5-A to C samples as a function of mixing.

It can thus be concluded that in general the mixing process used in the MASTERTECH is efficient enough to give a good reproducibility of the clay content, even after a prolonged period of rest, provided that stirring between 10 and 20 minutes is applied.

Also for this test 3 sub-samples (test 5-H1, -2 and -3) were wet-sieved at $32\mu\text{m}$ and analysed using a classical sedimentation technique. The clay content detected is much lower and the standard deviation is larger (average 40.63%, standard deviation 1.94) than the average clay content and standard deviation observed for the SEDIGRAPH analyses 5-A to -C (average 54.97, standard deviation 1.12). The standard deviation on the average clay content is better than that for test 4 what is most probably a result of a better initial homogenisation.

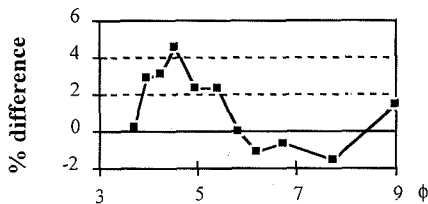


Figure 11 : Difference in concentration of particles in size fractions obtained with a sedimentation technique and with the SEDIGRAPH (test 5).

A comparison of the grain-size spectra of samples H-1 to -3 with samples of the A- to C-series (table 6, figures 9 and 10) indicates that although the general trend of the spectra are similar, each fraction coarser than $22\mu\text{m}$ (5.5ϕ) of the samples of the H-series contains up to 5% more particles than is the case for samples of the A- to C-series. No significant difference is seen in the fractions finer than $22\mu\text{m}$. Furthermore the algebraic sum of the differences between both series

(table 6 and figure 11) for each fraction (14.04%) matches closely the observed difference in clay content (14.33%) between the SEDIGRAPH and the sedimentation technique. A similar result was obtained for samples of test 4 series so that apparently the difference in clay content can be explained for a large part by the retention of clay-sized particles on the sieves during the wet sieving process.

The standard deviation for the concentration of particles in the separate fractions ranges between 0.06% and 1.65% for the sedimentation analyses and between 0.02% and 1.5% for the SEDIGRAPH analyses.

The average mean value for test 5-A to -C samples was 8ϕ with a standard deviation of 0.05 ϕ -units. The average sorting was 4.14 ϕ -units with a standard deviation of 0.01 ϕ -units. The average skewness was 0.24 ϕ -units with a standard deviation of 0.03 ϕ -units and finally the average kurtosis was 1.75 ϕ -units with a standard deviation of 0.01 ϕ -units. These standard deviations are better than for test 4 clearly demonstrating that much care must be taken to the homogenisation of the raw sample, as was done for test 5, before subsampling an aliquot part of it for the grain-size analysis.

microns	series H		series A		difference (H - A)
	average	standard deviation	average	standard deviation	
76	0.43	0.06	0.13	0.02	0.3
63	3.21	0.52	0.26	0.13	2.95
52	3.39	1.34	0.21	0.05	3.18
43	5.71	1.19	1.11	0.15	4.60
32	7.58	0.54	5.27	0.42	2.31
24	9.81	1.65	7.41	0.24	2.40
18	4.35	0.23	4.33	1.50	0.02
14	3.54	0.32	4.65	1.46	-1.12
9	4.08	0.50	4.70	0.61	-0.62
5	6.84	0.87	8.33	0.37	-1.49
2	10.43	1.64	8.90	0.25	1.52
total					14.04

Table 6 : Average concentration of particles in the different size fractions for the sedimentation analyses (series H) and for the SEDIGRAPH analyses (series A).

5. Sieve diameter versus settling diameter as measured with the SEDIGRAPH.

Three samples (test 6, 7 and 8) were prepared as stated for test 5 and wet sieved using a sieve series 93, 76, 63, 53, 45, and 32 μm , or a $1/4 \phi$ interval. The sediment retained on each sieve was subsequently analysed with the SEDIGRAPH using a glycerol solution as the suspending medium. For all three samples the mean diameter and the inclusive graphic sorting (Folk, 1966) was calculated and compared to the sieve diameter.

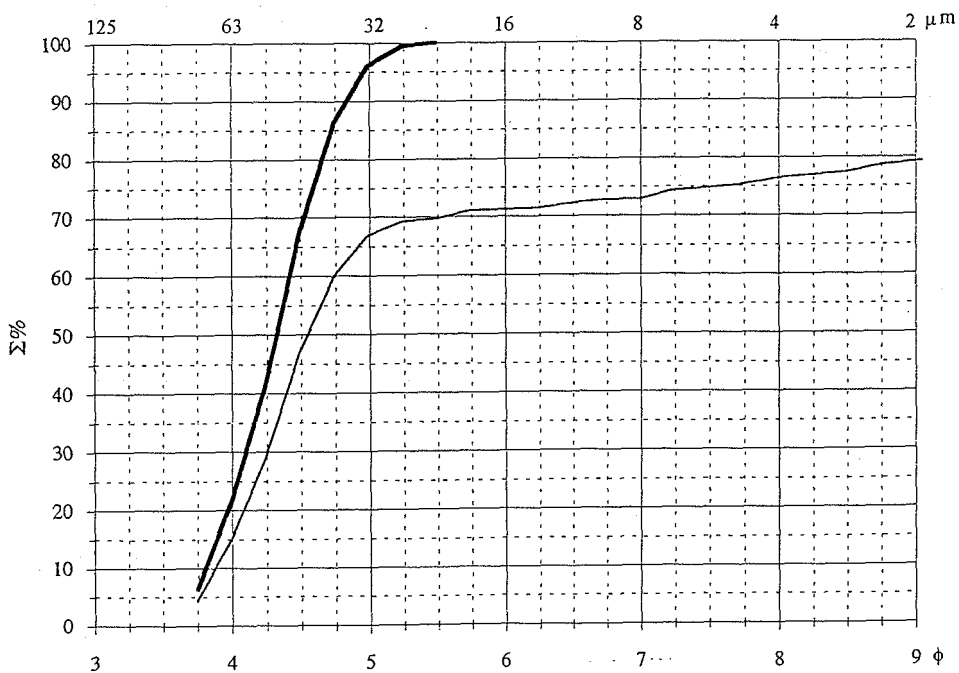
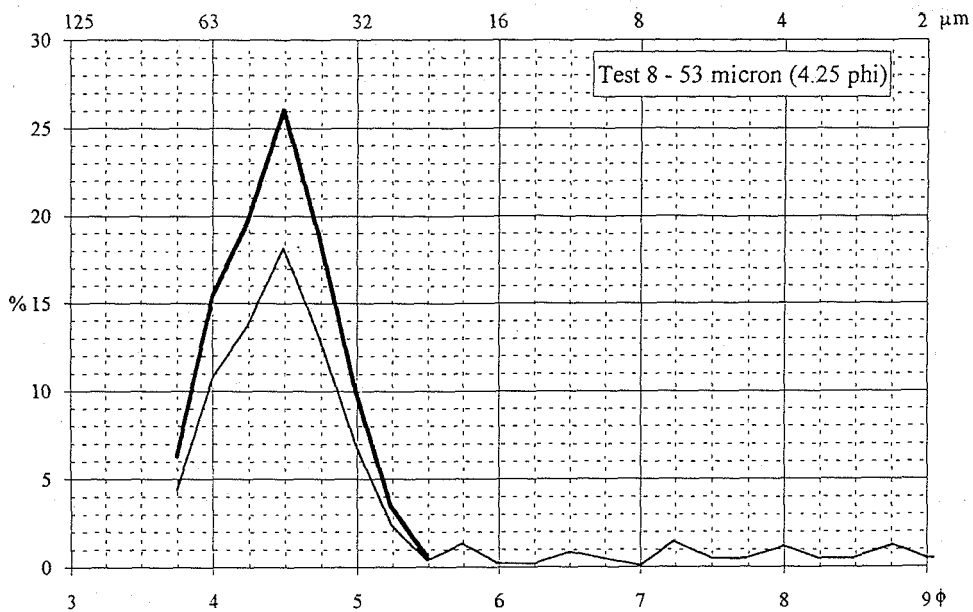


Figure 12 : Grain-size distribution of 53 micron sieve fraction; heavy line shows the distribution after removal of the fractions smaller than 5.5 phi.

From all analyses it appeared that a range of particle sizes were present more or less symmetrically distributed around a modal value (figure 12) that was close to the sieve value. Furthermore, it also appeared that an unexpected population of much finer clay-sized particles was present. This finer population, that was not expected to occur since each fraction were treated repeatedly with ultrasonics to achieve complete dispersal, will be discussed later; for evident reasons it was discarded for the calculation of the mean size of the distribution.

sieve microns	SEDIGRAPH microns			average microns	difference phi-units
	test 6	test 7	test 8		
32	34	32	31	32.33	-0.05
44	41	40	42	41.0	+0.1
53	52	46	50	49.33	+0.1
63	62	53	66	60.33	+0.05
76	65	61	72	66.00	+0.20

Table 7 : Sieve diameters and mean diameters as calculated from SEDIGRAPH analyses.

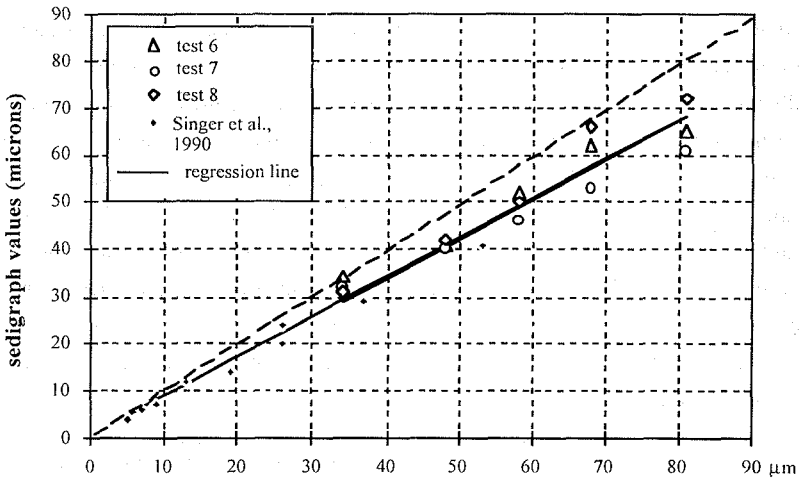


Figure 13 : Modal values of size fractions as obtained with the SEDIGRAPH compared to corresponding sieve diameters. The values of Singer et al. (1990) were obtained by decanting each fraction instead of by sieving.

The calculated mean diameters are given in table 7 and compared to the sieve diameters in figure 13. With the exception of the sieve of 32 μm the calculated averages of the "SEDIGRAPH"-diameters are smaller than the sieve diameters. The inclusive graphic sorting of the distribution (table 8) is more or less constant and ranges between 0.34 and 0.43 phi-units. The observed sorting falls in the range of standard deviation given by Singer et al. (1988) for fractions at $1/2 \phi$

interval between 4 and 63 μm . From the regression line (figure 13) it can be seen that the departure from the sieve diameter increases with increasing size. At 76 μm the deviation is approximately 0.2 phi and approaches the sieve interval used (0.25 phi).

sieve microns	SEDIGRAPH phi-units			average phi-units
	test 6	test 7	test 8	
32	0.35	0.36	0.58	0.43
44	0.33	0.37	0.51	0.40
53	0.38	0.39	0.41	0.39
63	0.36	0.39	0.32	0.36
76	0.27	0.40	0.34	0.34

Table 8 : Sieve diameters and inclusive graphic sorting as calculated from SEDIGRAPH analyses.

As was stated previously it appeared that in all the SEDIGRAPH analyses for test 6 to 8 a non-negligible amount of clay-sized particles occurred (figure 13). SEM observation of a sample (test 7), that was prepared in the normal way but was not subjected to stirring and ultrasonic dispersion in the SEDIGRAPH showed that clay-sized particles are indeed present. They occur either adhered to the surface of larger particles (photo 1, in annex) or as agglomerated fine particles (photo 2 and 3, in annex). It seems thus likely that these adhered or agglomerated particles are not completely released during the normal preparation of the sample, involving only stirring and a few minutes of ultrasonic dispersion, but that many, if not all, of them are freed after supplementary stirring combined with ultrasonic dispersion in the MASTERTECH. Furthermore it can be observed that the relative amount of these particles decreases with decreasing sieve size from 38% for 76 μm to 11% for 32 μm (table 9) and consequently with longer wet sieving time. This shows clearly that also during the wet sieving process at least part of the aggregates are destroyed.

sieve	% of total sample	% clay particles sieve fraction	% clay particles total sample
76	11	38	4.2
63	2.6	23	0.6
53	2.9	14	0.4
43	3.4	10	0.3
32	5.4	11	0.6

Table 9 : Percent clay particles in SEDIGRAPH analyses and % of these particles relative to total sample (test 6).

The total amount of these fine particles accounts for approximately 6% of the total sample. Since for a classic sedimentation technique such as the pipette-method no extra stirring or mixing is performed it can be assumed that part of the observed divergence in clay content results from

the adherence of clay-sized particles at the surface of larger particles or form the occurrence of aggregates that are not destroyed during the preparation or sieving of the sample.

Conclusions

A number of multiple grain-size analyses using the SEDIGRAPH 5100, combined with the MASTERTECH 51, of fine-grained estuarine sediments has been described. The analyses were performed in a glycerol-water solution with a density of 1.12 g/cm^3 and a viscosity of 4 cps. From this several conclusions can be drawn:

1. The advantage of a denser medium lies in the decrease of the setting velocity and consequently the possibility of increasing the maximum grain-size that can be analysed with the SEDIGRAPH. It is also assumed that the effect of hindered settling and particle aggregation will be less and thus that higher sediment concentrations can be used.

2. The instrumental accuracy of the SEDIGRAPH as detected in these tests is very good. The relative error on the clay content ranges between 1 and 4%. The concentration of particles in the separate fractions is determined with a standard deviation that is always better than 0.7 % and with a relative error that is inversely proportional to the concentration.

3. Some analyses showed deviating results that are to be attributed to insufficient homogenisation before subsampling. Although this observation is far from being new it is still worth being mentioned because of its importance for laboratory experiments that use large amounts of supposedly homogeneous fine-grained sediments and where subsampling may be problematical.

4. The tests performed in this study confirmed the observation that more clay-sized particles are measured than with a classical sedimentation technique. Aggregation of particles may be one of the causes and occurs if samples are insufficiently mixed. An important outcome of this study, however, is that the observed difference results mainly of the inefficiency of the wet sieving process. Incomplete separation of particles adhering to larger particles or forming aggregates makes that a non-negligible proportion of clay-sized particles are hold back at the sieve. A stronger mixing process in the SEDIGRAPH makes that these particles are freed. Since the mean of the grain-size distribution is negatively correlated with clay content an exact determination of the last parameter is important if sediments containing more than 15% of clay are to be analysed.

5. It could be observed in this study that even when an important difference in clay content occurred between subsamples their grain-size spectra was not affected. Therefore it is indicated that grain-size spectra should be used more generally for the description of fine-grained sediments instead. It is one of the advantages of the SEDIGRAPH, and other similar techniques, that the size distribution can be given using a constant class interval for the complete spectrum.

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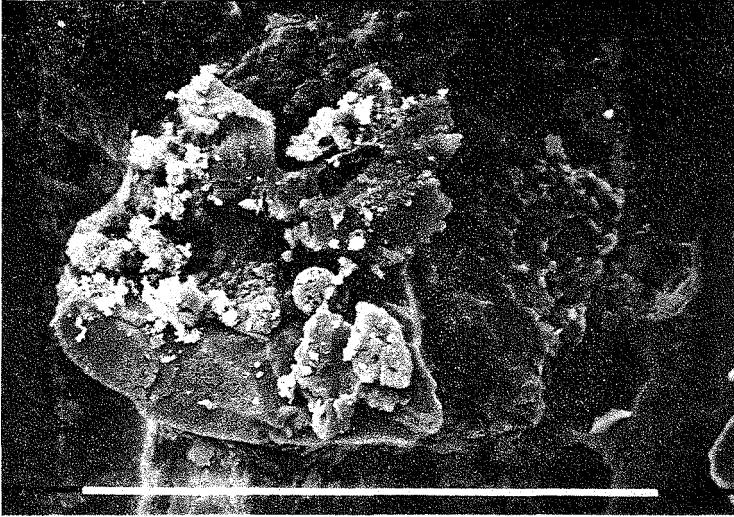


Photo 1 : SEM-photograph of sample test-7 (SEM 1950-18) showing very fine particles ($< 1 \mu\text{m}$) adhered to a larger particle, scale bar is $10 \mu\text{m}$.

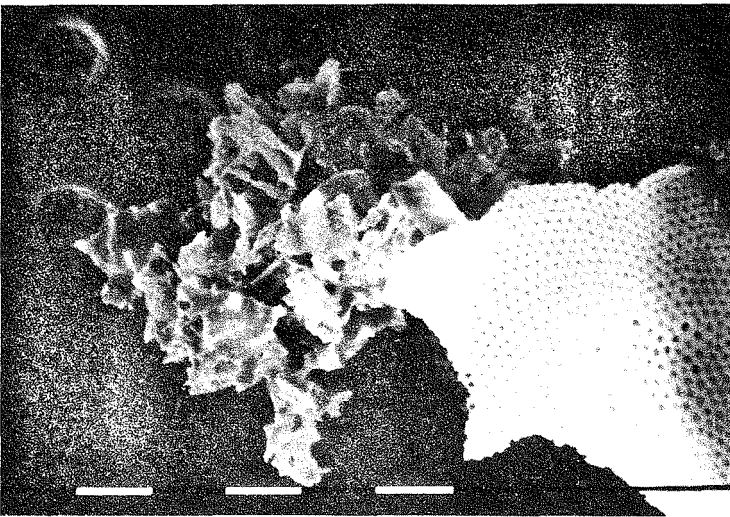


Photo 2 : SEM-photograph of sample test-7 (SEM 1950-04) showing an agglomerate of particles $< 10 \mu\text{m}$, scale bar is $10 \mu\text{m}$.

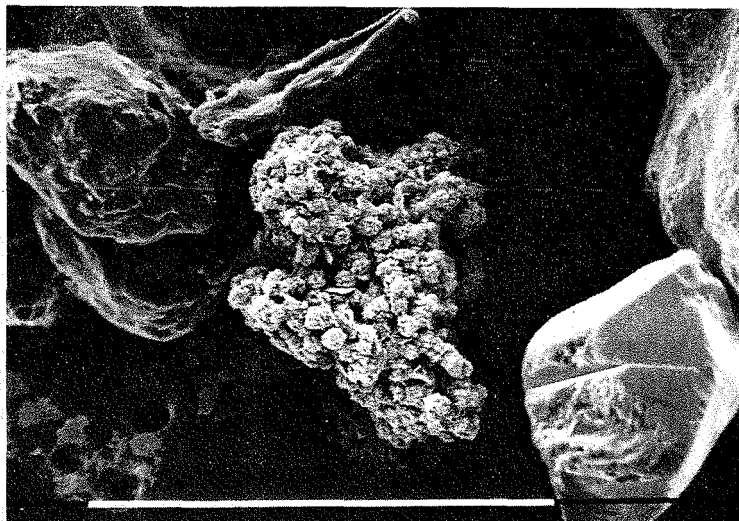


Photo 3 : SEM-photograph of sample test-7 (SEM 1950-06) showing an agglomerate of agglomerated particles with diameter $< 2 \mu\text{m}$, scale bar is $100 \mu\text{m}$.

TEST 1 91B05	micron	phi	run 1 : t = 0		run 2 : t = 53'		run 3 : t = 75'		run 4 = 96'		run 6 : t = 4H36'		run 7 : t = 14 days	
			data1.4	data1.6	data1.7	data1.8	data1.10	Schelde.3	%	S %	%	S %	%	S %
	250	2.00	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	
	177	2.47	0.5	0.9	0.5	0.9	0.5	0.9	0.5	0.9	0.5	0.9	0.9	
	125	3.00	2.5	3.3	2.5	3.3	2.5	3.3	2.5	3.3	2.5	3.3	3.3	
	88	3.64	12.4	15.8	12.4	15.8	12.4	15.8	12.4	15.8	12.4	15.8	15.8	
	63	3.99	5.3	21.1	5.3	21.1	5.3	21.1	5.3	21.1	5.3	21.1	21.1	
	31	4.97	3.8	24.8	3.8	24.8	3.8	24.8	3.8	24.8	3.8	24.8	24.8	
	26	5.32	1.2	26.0	0.2	25.0	0.6	25.4	0.4	25.2	0.8	25.7	1.1	
	18	5.64	0.2	26.2	0.5	25.4	0.9	26.3	0.6	25.8	0.9	26.6	0.2	
	16	6.06	2.1	28.3	1.9	27.3	1.6	27.9	1.6	27.4	1.7	28.3	1.8	
	9	6.64	5.2	33.5	4.2	31.5	4.3	32.2	4.2	31.6	4.7	32.9	3.8	
	8	6.97	3.4	36.8	3.3	34.7	3.1	35.3	2.8	34.4	3.1	36.0	2.9	
	6	7.38	5.1	41.9	4.5	39.3	4.5	39.8	4.3	38.8	4.7	40.8	4.2	
	5	7.64	4.0	45.9	3.5	42.8	3.4	43.1	3.1	41.9	3.4	44.1	3.0	
	4	7.97	5.1	50.9	4.4	47.2	4.2	47.4	4.3	46.2	4.3	48.5	3.9	
	3	8.38	6.3	57.2	5.8	53.0	5.7	53.0	5.5	51.7	5.8	54.3	5.3	
	2	8.97	9.3	66.5	8.5	61.5	8.2	61.2	7.9	59.6	8.2	62.5	7.6	
	% clay		33.5	38.5	38.8	40.4	37.5	41.2						
	average % clay		38.3											
	standard deviation		2.7											

Tabel 10 : Grain-size data for test 1, sample 91B05

TEST 2		water				glycerol solution					
		run 41a		run 41b (30 min.)		run 42		run 46 (25 hrs.)		run 49 (29 hrs.)	
micron	phi	%	Σ %	%	Σ %	%	Σ %	%	Σ %	%	Σ %
250	2.00	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6
180	2.47	2.2	2.8	2.1	2.7	2.1	2.8	2.2	2.8	2.2	2.8
125	3.00	23.1	25.9	22.9	25.5	22.5	25.9	23.1	25.9	23.1	25.9
90	3.47	11.7	37.6	12.6	38.2	12.4	37.6	11.7	37.6	11.7	37.6
63	4.00	19.8	57.4	18.8	56.9	18.5	57.4	19.8	57.4	19.8	57.4
44	4.50	1.1	58.5	1.1	58.0	5.2	62.6	3.1	60.4	3.6	61.0
31	5.00	2.4	60.9	2.4	60.4	4.4	67.0	2.5	62.9	2.2	63.2
22	5.50	3.0	63.9	3.0	63.4	2.7	69.7	1.8	64.8	1.4	64.6
16	6.00	2.5	66.3	2.5	65.9	2.0	71.6	1.2	65.9	1.1	65.7
11	6.51	2.1	68.4	2.1	68.0	1.2	72.8	0.8	66.7	0.7	66.4
8	7.00	2.1	70.5	2.1	70.0	1.2	74.0	0.8	67.5	0.9	67.3
6	7.51	2.0	72.5	2.0	72.1	1.2	75.2	1.0	68.5	0.7	68.0
4	8.00	1.9	74.4	1.9	74.0	1.1	76.3	0.9	69.4	0.8	68.8
3	8.48	2.1	76.5	2.1	76.0	1.1	77.5	0.9	70.3	0.8	69.7
2	9.00	2.3	78.8	2.3	78.4	1.4	78.8	0.9	71.2	0.7	70.4
% clay		21.2		21.7		21.2		28.8		29.6	
mean in microns		2.1		2.1		2.8		3.0		3.6	
mean in phi units		8.92		8.92		8.46		8.38		8.12	
sorting in phi units		7.02		7.02		7.14		7.17		7.22	
skewness in phi units		0.22		0.22		0.37		0.40		0.48	
kurtosis in phi units		0.89		0.89		0.92		0.92		0.97	
% clay average (glycol)		27.0						average			
% clay standard deviation (glycol)		4.7				mean, microns		mean phi units		skewness phi units	
% clay average (water)		21.2				2.10		8.92		7.02	
% clay standard deviation (water)		0.3				3.1 (0.42)		8.32 (0.17)		7.17 (0.04)	
						water		0.42 (0.03)		0.22	
						glycol		0.42 (0.03)		0.89	
								0.42 (0.03)		0.94	

Table 11 : Grain-size data for test 2

TEST 4	wet sieve microns	loss %	sand %	silt %	clay %	silt/clay	mean microns	mean phi units	sorting phi units	skewness phi units	kurtosis phi units
A1-1	106	20.69	32.25	38.17	29.58	1.29	10.8	6.53	4.57	0.82	2.45
A1-2	106	20.69	31.29	37.95	30.76	1.23	14.8	6.08	4.61	0.78	2.36
A2-1	106	21.43	31.07	31.35	37.58	0.83	63.4	3.98	4.86	0.50	1.94
A2-2	106	21.43	31.19	30.69	38.12	0.81	58.3	4.10	4.91	0.52	1.91
A3-1	106	20.56	28.86	34.34	36.80	0.93	65.2	3.94	4.79	0.52	1.98
A3-2	106	20.56	30.02	32.82	37.16	0.88	55.9	4.16	4.84	0.55	1.95
average (A)		20.89	30.78	34.22	35.00	1.00	44.7	4.80	4.76	0.62	2.10
standard deviation (A)		0.42	1.18	3.23	3.79	0.21	25.0	1.18	0.14	0.14	0.24
average (A)*		21.00	30.29	32.30	37.42	0.86	60.7	4.05	4.85	0.52	1.95
standard deviation (A)*		0.50	1.09	1.63	0.57	0.06	4.3	0.10	0.05	0.02	0.03
B1-1	76	20.96	39.59	25.58	34.83	0.73	23.7	5.40	4.90	0.62	2.02
B1-2	76	20.96	40.97	27.88	31.16	0.89	8.1	6.94	4.77	0.78	2.26
B1-3	76	20.96	41.88	30.52	27.60	1.11	2.2	8.80	4.60	0.95	2.62
B2-1	76	21.11	38.39	30.33	31.28	0.97	9.6	6.70	4.77	0.74	2.26
B2-2	76	21.11	39.57	32.51	27.92	1.16	3.1	8.35	4.62	0.89	2.56
B2-3	76	21.11	38.95	32.94	28.11	1.17	3.3	8.26	4.62	0.89	2.56
B3-1	76	20.79	38.37	30.44	31.19	0.98	9.3	6.75	4.73	0.79	2.30
B3-2	76	20.79	39.04	32.54	28.42	1.14	3.9	7.99	4.61	0.90	2.54
B3-3	76	20.79	38.86	33.03	28.11	1.17	3.5	8.18	4.59	0.92	2.58
average (B)**		20.95	39.32	29.88	30.80	0.98	9.6	7.02	4.73	0.79	2.32
standard deviation (B)**		0.14	0.97	2.72	2.48	0.16	7.4	1.05	0.11	0.10	0.20

* = analyses of A1-series not considered ** = third analyses of B-series not considered

Table 12. : Grain-size data for test 4

TEST 5	wet sieve microns	weight gram	loss %	sand %	silt %	clay %	silt/clay microns	mean microns	mean phi units	sorting phi units	skewness phi units	kurtosis phi units
A1	76	19.18	4.22	0.82	46.13	53.05	0.87	0.89	10.13	4.15	0.29	1.77
A2	76	19.08	4.59	0.52	43.65	55.83	0.78	0.76	10.36	4.12	0.22	1.75
A3	76	19.23	4.43	0.46	43.83	55.71	0.79	0.76	10.36	4.12	0.23	1.74
B1	76	19.14	4.34	0.94	44.22	54.84	0.81	0.81	10.27	4.15	0.24	1.75
B2	76	19.10	4.46	0.59	43.77	55.64	0.79	0.77	10.34	4.14	0.22	1.74
B3	76	19.31	3.65	0.65	43.34	56.01	0.77	0.75	10.38	4.12	0.22	1.74
C1	76	19.11	4.41	0.59	45.23	54.18	0.83	0.84	10.22	4.15	0.26	1.75
C2	76	19.08	4.56	1.09	42.75	56.16	0.76	0.76	10.36	4.15	0.20	1.74
C3	76	19.17	4.48	0.83	45.84	53.33	0.86	0.87	10.17	4.15	0.27	1.77
average		19.16	4.35	0.72	44.31	54.97	0.81	0.80	10.29	4.14	0.24	1.75
standard deviation		0.07	0.27	0.20	1.10	1.12	0.04	0.05	0.09	0.01	0.03	0.01
H1 (13)	32	18.76	6.28	3.26	58.78	37.96	1.55	NC	NC	NC	NC	NC
H2 (14)	32	18.81	7.60	3.49	55.05	41.46	1.33	NC	NC	NC	NC	NC
H3 (15)	32	18.71	6.72	4.15	53.36	42.49	1.26	NC	NC	NC	NC	NC
average		18.76	6.87	3.63	55.73	40.64	1.38					
standard deviation		0.04	0.55	0.38	2.26	1.94	0.12					

NC = not calculated

Table 13 : Grain-size data for test 5