STATENS GEOTEKNISKA INSTITUT

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Ultrasonic Dispersion of Clay Suspensions

by Roland Pusch

ULTRASONIC DISPERSION OF CLAY SUSPENSIONS FOR GRANULOMETRIC AND MORPHOLOGICAL INVESTIGATIONS

By ROLAND PUSCH

ABSTRACT. — The influence of ultrasonic dispersion of clay suspensions on size and shape of the clay-size particles is investigated.

There is a slight deviation from the median values of size and shape obtained when ultrasonic treatment is not used. It is assumed that this deviation is mainly due to the increased possibility of identifying small particles when ultrasonic treatment is applied.

Scope of the test

In connection with a study of the size and shape of clay particles, ultrasonic dispersion of clay suspensions has been tried. This study of the particle morphology which forms a part of an investigation of clay microstructure, is based on a technique previously described by the author (Pusch 1962). In these earlier investigations, clay suspensions were obtained by shaking 20 grams of undisturbed clay in 1 000 cc of 0.005 M solution of sodium pyrophosphate for 30 minutes. After sedimentation during 8 hours using 100 mm depth of sedimentation, droplets of the suspension were placed on grids covered with zapon film.

Although the electron micrographs of the dry sediments generally showed a large number of discrete particles which could be measured, each micrograph generally showed also a number of dense, unresolved aggregates which could not be studied in detail. The presence of these aggregates indicated the need of a better dispersion, which was achieved by the following technique:

1. 100 grams of clay was placed at its natural water content in a solution of 20 cc of hydrogen peroxide in 100 cc of distilled water and was thereafter kept at a temperature of 110° C for one hour.
2. The clay was washed by centrifugation (20 minutes at 2000 r.p.m.). This process was repeated four times.
3. The clay was thereafter dried at 110° C for 24 hours. (This operation should be avoided since it may affect the particle morphology and may create particle groups which cannot be separated later. However, drying is generally used for sedimentation analysis at the Swedish Geotechnical Institute where this work was carried out.)
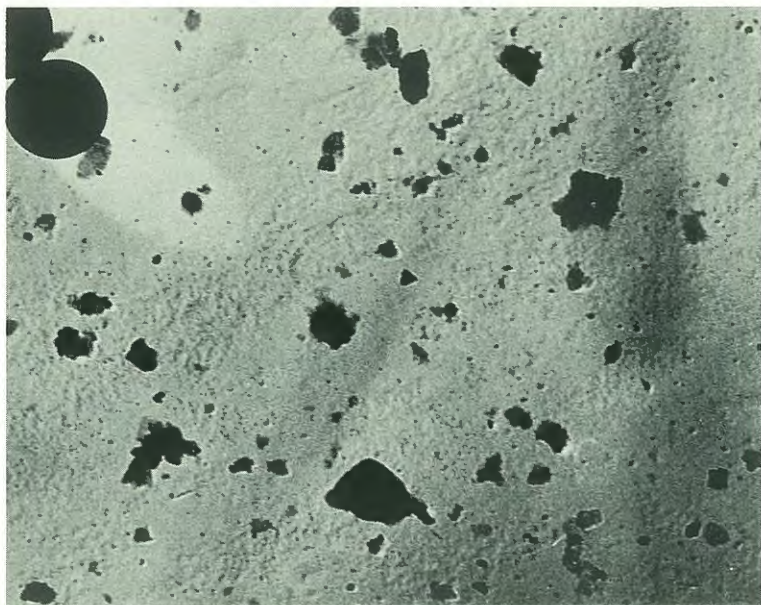
Fig. 1. Electron micrograph 243. Suspension treated with ultrasonic waves. (Electronic magnification 7000 ×, total magnification 23500 ×.)

4. The dry clay was placed in the sedimentation cylinder used for sedimentation analysis according to the pipette method (cf. Akroyd 1957). The cylinder was filled with 80 cc of 0.005 M solution of sodium pyrophosphate.

5. The clay suspension was thereafter agitated for four hours by a motor-operated, axially moving perforated lid. Minute quantities of the suspension were then extracted for the sedimentation analysis.

6. The remaining suspension was transferred to a 1000 cc glass container which was filled completely with distilled water.

7. After sedimentation for 250 minutes and subsequent decantation, 25 cc of the suspension was taken at four, equally spaced levels in the decanted, thoroughly shaked suspension. Theoretically, the equivalent diameter of the particles still in suspension should be less than about 5 μ.

8. The 100 cc suspension thus obtained was thereafter treated for 30 minutes in an 80 kc/s, 80 W ultrasonic apparatus.

9. Droplets were taken from the suspension. The droplets were applied to 400 mesh copper grids covered with a film of pure carbon prepared in a Hitachi evaporator.

A micrograph of the clay sediment is shown in Fig. 1. Very few or no aggregates can be observed, which indicates a good dispersion. Since the effect of ultrasonic waves on the particle morphology was not known, a separate investigation was performed on the same clay material, using the same test programme, but excepting the ultrasonic treatment (mom. 8). Fig. 2 shows a typical micrograph of aggregates obtained in this investigation.
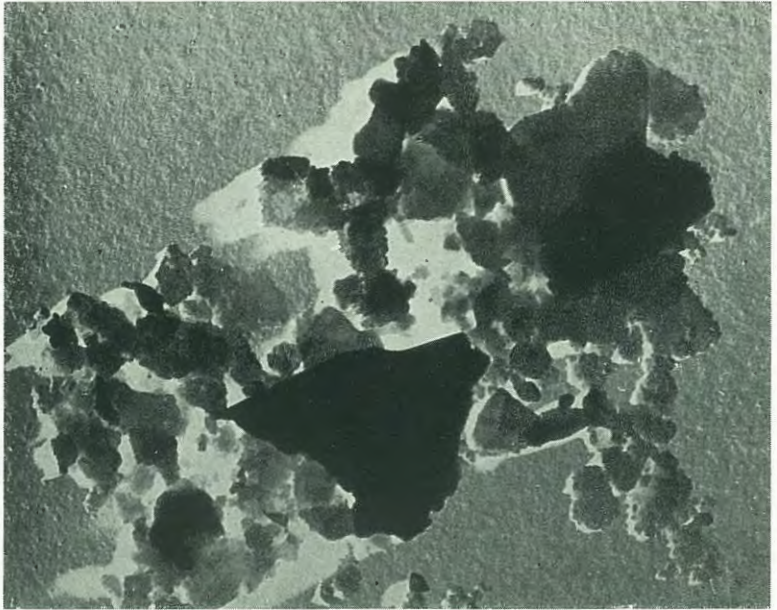
Fig. 2. Electron micrograph 174. Untreated suspension. (Electronic magnification 7000 ×, total magnification 23500 ×.)

Clay material

A clay sample obtained from a depth of 2 m by a 50 mm piston sampler at the Skå-Edeby test field was used in this investigation. The sample was taken from a layer of grey-green Postglacial organic clay. The average ground water table was located at 0.75 m depth and the dry crust of fissured clay was about 1.5 m thick at this location. Some geotechnical data are given in Table 1.

TABLE 1. *Geotechnical data.*

Sample (central)	Water content in %	Plastic limit	Liquid limit	Unit weight in g/cm³	Shear[1] strength in t/m²	Sensitivity	Clay content in %	Ignition loss in %
	w	w_P	w_L	γ	τ_{fu}	S_t		
5698	106	39	118	1.42	1.5	9	56	7.4

[1] Cone penetration test.

The clay has been examined by using DTA and X-ray diffraction methods with regard to the mineralogical composition. Detailed results and descriptions of these investigations will be published in a general report on the Skå-Edeby clay. The main constituents of the clay were illite and quartz.
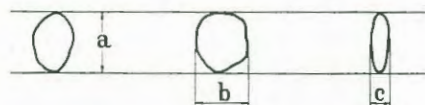
Fig. 3. Characteristic dimensions of a particle according to Krumbein (1941). *a* is the longest intercept, the long diameter. *b*, the intermediate diameter, is the measure of the widest part of the "maximum projection plane". The direction of *b* is perpendicular to the long axis. *c*, the short diameter, is the measure of the widest part when the particle is rotated 90° from the position shown in the figure illustrating the measure *b*. The three diameters are mutually perpendicular but do not necessarily intersect at the same point.

Determination of particle size and shape

In order to make it possible to determine accurately the particle dimensions, droplets of suspensions of latex particles with an average diameter of 0.264 and 0.557 μ were placed on the grids with the sedimented clay particles. After drying at room temperature the grids were shadowed with chromium at an angle of about 30° in the evaporator.

A series of micrographs were taken in a Zeiss EM9 electron microscope and the electronic magnification of the micrographs, which were randomly chosen, was 7 000 × to 21 000 ×. The electron microscope was equipped with a 25 μ aperture in the objective lens. The film used was a Gevaert Scientia Film 23 D 56, 70 × 70 mm.

A linear optical magnification of 2 × was used as well and the micrographs thus obtained were utilized to determine the particle dimensions. They were measured directly on the micrographs by using a Hensoldt ocular provided with a built-in precision 0.1 mm interval scale of metal. The dimensions of the particles were defined in accordance with Krumbein's (1941) concepts, (Fig. 3). These definitions are especially suitable in the case of clay minerals (Pusch 1962). The particle shape is described solely by the a/c ratio. A series of shape investigations of clay minerals has shown that this is a convenient shape factor.

TABLE 2. *Number of measured particles and number of measurements, respectively.*

Micrograph	a	c	a/c	Suspension treatment
240	141	136	136	
242	49	49	49	ultrasonic
243	79	57	57	
157	118	117	117	
173	151	108	108	none
174	93	66	66	("untreated")

Fig. 4. Histogram of the dimension *a*. Suspension treated with ultrasonic waves. Micrographs 240, 242, 243.
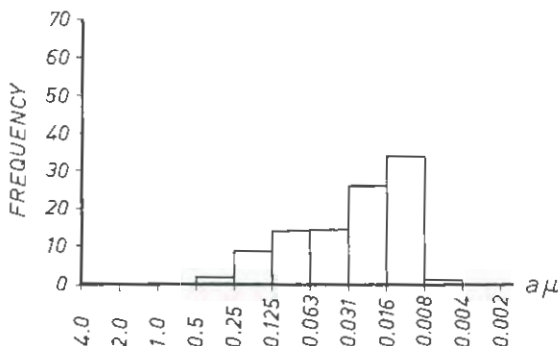
The dimensions a and c were determined from three micrographs where ultrasonic dispersion had been used and from three micrographs of particles which had not been subjected to such treatment. The total number of particles measured in each micrograph is given in Table 2. It should be noticed that the dimension c could not always be measured due to overlapping or unsharp shadows. All particles were measured, which were present within a representative, small area of each micrograph.

The total number of particles which were measured in each suspension exceeded 200. According to previous investigations this number is sufficient to obtain reliable results.

Test results

In order to treat the results statistically, histograms and cumulative curves were constructed and median measures, skewness measures, and sorting coefficients were calculated. The histograms, which offer the clearest, direct interpretation of the frequency curves, are shown in Figs. 4—7. Only the histograms which are based on all measurements of each suspension are shown. In Table 3 the values determined from the statistical analysis are given.

It can be seen from Table 3 that there is a small difference in the calculated median values M between the suspensions treated with ultrasonic waves and the untreated suspensions. This is apparently due to the increased possibility to identify small particles when the ultrasonic dispersion method is used, although it cannot be excluded that a certain size decrease has been caused. Thus, the disappearance of the bi-modality of the histogram of the suspension treated with ultrasonic waves (Fig. 4) when compared with the histogram of the untreated suspension (Fig. 6) may indicate some particle size decrease. It can also be seen from Table 3 that the skewness measure and sorting coefficient vary considerably between the micrographs. This is probably caused by the different number of measured particles in the two cases. However, one can
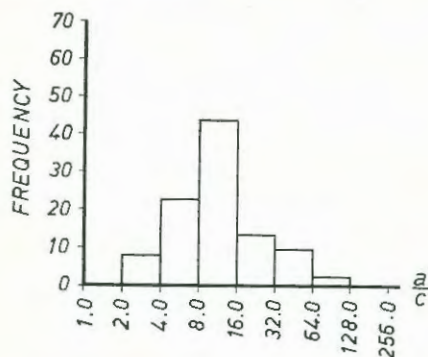
Fig. 5. Histogram of the ratio a/c. Suspension treated with ultrasonic waves. Micrographs 240, 242, 243,
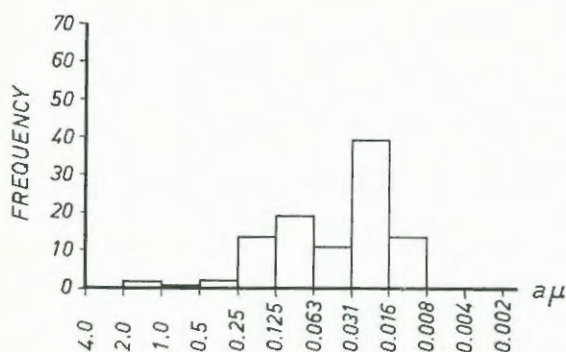

Fig. 6. Histogram of the dimension a. Untreated suspension. Micrographs 157, 173, 174.

notice that the difference is small between the average values. The significance of the skewness measure and of the sorting coefficient will not be discussed here.

Also we shall refrain from a detailed discussion of the statistical data. It may, however, be noted that the magnitudes of the a-value and of the a/c-value are of the same order of magnitude as the corresponding values found in a similar investigation of Postglacial clays from Norrköping (Pusch 1962). It can also be seen that the reproducibility is good and that the accuracy of the test results is in all probability satisfactory, as may be inferred from the small variation of the calculated median values between the six micrographs. The accuracy has been discussed with special reference to the sources and probable degrees of errors in connection with the investigation of the Norrköping clays.

It may be expected that small particles, because of their insignificant mass, would be less influenced by ultrasonic treatment than relatively large particles. In order to estimate the influence of particle size, all particles with a-values larger than 0.031 μ and the corresponding a/c-values were treated statistically separately. It can be seen from Table 4 that there is a slight difference in the measured a-values between the treated and untreated suspensions as in the previous case. As before, this difference is probably due to the fact that ultra-
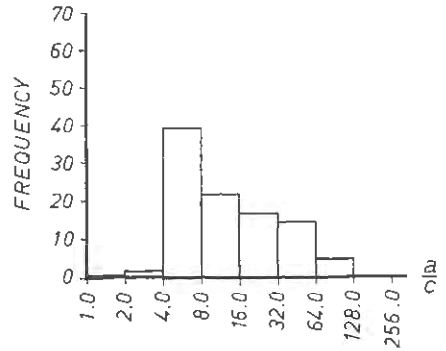
Fig. 7. Histogram of the ratio a/c. Untreated suspension. Micrographs 157, 173, 174.

TABLE 3. *Median values, quartiles, skewness numbers, and sorting coefficients.*

	Micrograph	a in μ			a/c			Skew-ness number of a[4]	Sorting coefficient of a[5]
		M[1]	UQ[2]	LQ[3]	M	UQ	LQ		
Ultrasonic treatment	240	0.03	0.05	0.02	8	10	6	1.11	1.58
	242	0.04	0.17	0.02	9	28	6	2.12	2.92
	243	0.05	0.11	0.03	6	14	4	1.32	1.91
Untreated	157	0.04	0.05	0.03	8	16	6	0.94	1.29
	173	0.04	0.13	0.03	5	20	4	2.44	2.08
	174	0.06	0.17	0.03	10	18	5	1.42	2.38
Average	240—243	0.03	0.09	0.02	8	13	5	2.00	2.12
Average	157 –174	0.04	0.13	0.03	7	18	4	2.44	2.08

[1] Median value

[2] Upper quartile

[3] Lower quartile

[4] Skewness number expressed as $\dfrac{LQ \cdot UQ}{M^2}$ (Trask)

[5] Sorting coefficient expressed as $\sqrt{\dfrac{UQ}{LQ}}$

sonic treatment gives a better dispersion and thus an increased possibility of identifying small particles.

It can also be seen that there is a difference between the a/c-values of the treated and untreated suspensions. The investigation of the clays from Norrköping indicated different a/c-values for particles of different sizes. Thus, it was found that the a/c-value increased markedly with the magnitude of a. If this tendency is valid for the Skå-Edeby clay, it supports the assumption that the difference in the a-values and the a/c-values is caused by the increased possibility to identify small particles in the case of ultrasonic treatment. It can be concluded that the ultrasonic treatment has not caused a greater splitt-

TABLE 4. *Median values of the dimension a and the quotient a/c of clay particles > 0.031 μ.*

	Micrograph	a in μ	a/c
Ultrasonic treatment	240—243	0.12	15
Untreated	157—174	0.15	21

ing of particles and strongly bound groups of particles than in the other case. If this had been the case, the a/c-value should have been higher for the suspension treated with ultrasonic waves than for the untreated suspension. The decrease of the a/c-value when using ultrasonic treatment may, to a certain extent, be the result of a particle size decrease.

It can be concluded that the ultrasonic treatment of the clay suspensions has not caused large changes of the particle size and shape. This means that such a treatment may be useful for granulometric analysis. It should be noticed, however, that the possible influence on the particle size and shape of larger mica grains has not been studied in this investigation. Most certainly such influence — especially splitting of particles parallel to the crystallographic ab-plane — will be caused in suspensions of montmorillonite, kaolinite and weathered minerals regardless of size.

It should also be pointed out that no conclusions can be drawn from this investigation as regards the influence of ultrasonic treatment as a method of disintegration of natural clay and sedimentary rock samples.

It is important to realize that size and shape distributions determined from dispersed suspensions do not represent the real distributions of the sediment in question during formation in situ. This is of course regardless of the method of dispersion.

Summary

In order to study clay particles morphologically, droplets of dilute clay suspensions which are placed on grids, are photographed in an electron microscope. By measuring conveniently defined particle dimensions, representative values can be calculated by using simple statistical methods. A good dispersion of the particles on the grids is desired for such measurements, since the accuracy of the results is affected by the degree of dispersion. Ultrasonic treatment of the suspensions has proved to be a very efficient method but the effect of this treatment on the mechanical break-down of the thin layer-silicates is not known. This article describes an investigation of a Postglacial clay where the median values of the maximum particle dimension and of the shape, expressed as the ratio of this dimension and the particle thickness, were determined.

Both untreated suspensions and suspensions treated with ultrasonic waves were used. The measurements indicated only a small difference. However, this difference was probably caused by the dispersion effect of the ultrasonic treatment so that a greater number of small particles were exposed rather than by mechanical disintegration. The investigation shows that ultrasonic treatment may be accepted as a preparative method in granulometric analysis.

Since the investigations were exploratory, the results should be regarded as preliminary.

REFERENCES

AKROYD, T. N. W., 1957: Sedimentation test. *Laboratory testing in soil engineering.* P. 68. Soil Mechanics Ltd, London.
KRUMBEIN, W. C., 1941: Measurements and geological significance of shape and roundness of sedimentary particles. *Journ. Sedim. Petr.* Vol. 2, No. 2.
PUSCH, R., 1962: Clay particles. *Byggforskningen, Handlingar* No. 40, Stockholm.