

STATENS GEOTEKNISKA INSTITUT

SWEDISH GEOTECHNICAL INSTITUTE



# SÄRTRYCK OCH PRELIMINÄRA RAPPORTER

**REPRINTS AND PRELIMINARY REPORTS** 

Supplement to the "Proceedings" and "Meddelanden" of the Institute

# **Recent Quick-Clay Studies**

- 1. Recent Quick-Clay Studies, an Introduction by Roland Pusch
- 2. Chemical Aspects of Quick-Clay Formation by Rolf Söderblom
- 3. Quick-Clay Microstructure by Roland Pusch

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RECENT QUICK-CLAY STUDIES, 1

# RECENT QUICK-CLAY STUDIES, AN INTRODUCTION

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(Received January 18, 1967)

A quick clay is characterized by such a large reduction of its shear strength when remoulded that it becomes a liquid. In practice a clay is called quick when its sensitivity  $(S_t)$ , i.e., the ratio of the shear strength in the undisturbed and remoulded states, exceeds a certain value, usually 30–50.

Quick-clay deposits are frequently found in Scandinavia and North America. In some cases, sensitivity values of more than 500 (cone test) have been determined. A correspondingly large reduction of the shear strength by remoulding is of great practical importance. Thus, in certain parts of Scandinavia such as the Göta River Valley in Sweden, extremely quick clays make necessary special precautions in road and house building. For instance the very small lateral support of the surrounding clay mass on driven piles must be taken into consideration in extreme cases.

The effect of quick clay in a slide area is especially important. Thus, while a slide in a normally sensitive clay usually involves only relatively small movements of the unstable soil mass, a slide in quick clay may cause a flow over a large area. Well-known examples of large slides in quick-clay areas are the Vaerdalen (Norway, 1893) and the Anchorage (Alaska, 1964) slides.

After the occurrence of the large slides at Surte (1950) and Göta (1957), research work was initiated in Sweden to find out the physico-chemical background of the formation of quick clay. Also in the U.S.A. and Canada the quickclay problem has been studied during the last decade.

In Norway, where slides in quick clays have occurred frequently, research on quick-clay formation was started earlier. The initial research work, the results of which were published by Ivan Th. Rosenqvist in 1946, showed that leaching of originally sea-water deposited clays brings about increased repulsive inter-particle forces, which means that the clay turns into the liquid-like state when the clay structure is broken down by remoulding.

Systematic, recent investigations in several countries have shown that a quick condition can be effected by introducing into a normally sensitive clay,

chemicals such as organic and inorganic acids, carbonates and phosphates with and without concomitant leaching. Also, it has been shown that fresh-water deposited clays can become quick.

Thus, the formation of a quick clay obviously cannot be explained by one single process in nature. Furthermore the complexity of the quick clay problem means that no simple way of strengthening or treatment for transformation of quick clay into more normally sensitive clay can be suggested.

Research work on the formation of quick clay has been intensified during the last years, leading to an increased knowledge of the action of dispersing agents, clay microstructure and several other factors. Some recent investigations which exemplify among others new experimental techniques, ideas and results, are going to be reported under the series title of "Recent quick-clay studies".

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RECENT QUICK-CLAY STUDIES, 2

# CHEMICAL ASPECTS OF QUICK-CLAY FORMATION

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(Received August 22, 1966)

#### SUMMARY

In connection with the systematical examination of the slope stability in the Göta River Valley in Sweden it was impossible to get a satisfactory explanation of the cause of the high sensitivity of quick clays which could be used in the continued examination as a basis for geotechnical investigations.

It was concluded that the quick-clay problem must be regarded as a problem of colloid chemical nature. Many of the results obtained pointed out the fact that the surface properties of the grains will play an important role, and in this connection the problem with the possible existence of surface-active agents (dispersing agents) in natural quick clays has been studied.

Systematic isolations of dispersing agents have been made by different methods, and the subsequent extracts have been analysed chromatographically. It is found to be easy to isolate dispersing substances by most of the methods tested, but it is very difficult to find any substances with a known composition. Instead, this part of the examination will approach the humic problem. In one case it has been possible to break up the complexes into individual components of known composition, i.e., in the case of fatty acids and soaps. Quick clay contains homologuous series of normal saturated fatty acids and for this reason soaps seem to be components which contribute to the quick-clay formation.

#### INTRODUCTION

The quick-clay problem has been studied in Sweden for several years. The studies were initiated by an investigation of slope stability in the Göta River Valley located in the western part of Sweden, where clays with very high sensitivity are common.

During the aforementioned period, quick clays were examined from several points of view. Systematic experiments were made to try to verify the "salt leaching theory" by means of salt soundings in the field, and laboratory experiments were performed to analyse the content of different ions in the double layers with the object of finding relationships between these inorganic ions and the sensitivity of

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the system. Systematic studies were also made to correlate mineralogical composition (by means of X-ray and DTA-studies) and sensitivity, and sedimentation analyses were made to correlate grain size and sensitivity.

These tests, carried out on various clays, have not given any definite picture of the cause of the high sensitivity and as summarized in a report to the Commission of the Göta River Valley, which was appointed for the slope investigation mentioned above (TULLSTRÖM, 1961), the conclusion drawn is that high sensitivity of a clay system cannot be explained in terms of simple differences in the composition of the system. Instead the phenomenon seems to be of a deeper colloid chemical nature and an understanding of the phenomenon thus requires colloid chemical studies of the systems.

This article is a short review of colloid chemical examinations of quick clays, particularly with regard to the occurrence of surface active substances in natural quick clays. A more detailed report will be published later.

As will be shown in the first part of this article, the mechanical behaviour of actual clay systems depends to a high degree on the properties of the surfaces of the particles, because it is on these surfaces that the binding forces act.

# INFORMATION ABOUT SWEDISH QUICK CLAYS

Quick clays in Sweden occur both in marine and fresh-water deposited sediments. In an analysis of 1848 consulting investigations of the Swedish Geotechnical Institute, it was found that quick clays were encountered in 62 cases (4.3%). Of these, 26 were varved quick clays (sedimented in fresh water) and 36 probably marine clays. It was also found that quick clays are sometimes located in the vicinity of organic layers. In particular, the layer succession, peat-varved quick clay (fresh water sedimented), can be found. Clays of this type are also common in Finland, as reported by KEINONEN (1963).

It is interesting to compare the curves of salt content and electrical resistivity obtained from the investigation in the Göta River Valley where 20 cases were reported by the Geological Survey of Sweden (TULLSTRÖM, 1961). In five cases correlation existed between the sensitivity curve and the salt content curve but in the others no correlation could be found. In most cases both quick and normal clays were found in completely leached profiles. In two cases the clay was quick where the salt content was highest in the profile (ca. 2.3 g Na/l pore water).

In some cases varved clays are overlain by flocculated clays and it is possible to find examples where the curve of sensitivity values shows no discontinuity at the boundary of the two layers.

JERBO et al. (1961) have detected a hitherto little-known effect in quick clays. An undisturbed sample of quick clay will change its sensitivity with time without any loss of water and finally reach normal sensitivity. Changes in pH-value and resistivity are also noticed.





All these facts are difficult to explain if the "card-house" structure, typical for salt-water deposited clays, is accepted as the main factor giving rise to high sensitivity.

One can show that a coagulation theory cannot be generally valid for Swedish conditions, because salt-free, natural clays will show different coagulation properties if salt is added.

Results from an instructive experimental series are shown in Fig.1–7. Increasing amounts of salt were added to a completely remoulded clay sample and the  $H_1$ -values obtained (the  $H_1$ -value is a relative strength value determined by fall-cone tests on completely remoulded clay, cf. HANSBO, 1957) have been plotted against the electrical resistance determined by a penetration electrode (cf. Söder-BLOM, 1957). Each diagram thus illustrates, for different clays, the relative variation of the shear strength caused by addition of salt. The quick clay in Fig.1 shows a



Fig.2. The same clay as in Fig.1 but stored for 3 months.

typical stiffening effect (coagulation) when treated with salt, but the same clay stored for about three months did not coagulate (Fig.2).

A low sensitive, salt-free clay (marine) from the Göta River, showed a very small stiffening effect (Fig.3) when treated with salt.

Fig.4 shows that a quick clay will lose its stiffening properties if treated with hydrogen-peroxide, and, in fact, will even show the reverse effect. Pure kaolinite shows a very small increase in shear strength when treated with salt (Fig.5).

In the last two cases (Fig.6, 7) the materials show a stiffening effect if treated with a dispersing agent before the coagulation experiments.

These series of experiments, reported here for one case only, but being generally valid for the types of Swedish clays studied, seem to indicate that the coagulation properties and, indirectly, the sensitivity are dependent on the surface properties of the clay particles.

Fig.4–7 show particularly that surface active agents must play an important role in quick-clay formation.

If the surface layer is destroyed by hydrogen-peroxide, the typical salt stiffening effect, present in quick clays, is lost, but it will re-appear if a dispersing agent is added. It seems, therefore, that the quick-clay problem, including the salt leaching effect, can be attributed to the properties of the surfaces of the mineral grains and especially to their content of surface active agents.





This will partly explain the effect on sensitivity of storage of a quick clay, as being due to an instability of the surface layers.

These results are in accordance with those from salt-leaching experiments reported by QUIRK (1952) and EMERSON (1954). They found that different English clays behaved differently if treated with salt and then leached and in some cases no deflocculation effect was obtained. They are also in accordance with the results from leaching experiments obtained by the present author for certain Swedish clays.

LAMBE (1958) and SÖDERBLOM (1959) have reported laboratory experiments where clays with normal sensitivity have been changed into quick clays by dispersing agents. The effects seem to occur in most Swedish non-saline clays, regardless of whether they have originally been sedimented in strongly salt water or in fresh water. In recent years a few authors have pointed out that processes of this kind can play an important role in nature, cf. JERBO (1964) and KERR and LIEBLING (1965). None of these authors have tried to analyse the active components.



Fig.4. Quick clay from Intagan, Göta River Valley, in which the surface layer has been destroyed with hydrogen-peroxide. The original clay had a very pronounced stiffening effect (about 70 times). After treatment the clay will become softer due to the addition of salt.

#### NATURAL DISPERSING AGENTS

The occurrence of varved quick clay below a layer of peat may thus be explained by the fact that brown organic peat water contains substances with dispersing properties. The first problem is thus to isolate the active substances in peat water and to discover whether they are found in the underlying quick clay or not. After having gained some insight into the nature of the active substances in this special quickclay process, one must discover whether substances of this kind can be found in quick clays in general.

To obtain some information about which substances should be studied, it is necessary to examine systematically the dispersing properties of those substances, which, according to the literature, can be expected to be found in actual systems.

#### TESTING EXPERIMENTS

Both inorganic and organic substances were tested. Different amounts of dispersing agents were added to the clay and its  $H_1$ -value, electrical resistivity and pH-value

#### CHEMICAL ASPECTS OF QUICK-CLAY FORMATION



Fig.5. Untreated kaolinite.

were determined after every addition; 25 substances were tested and the results are shown in Table I. A typical dispersing curve for a natural substance, "hymatomelanic acid", isolated from peat, is shown in Fig.8.

The results indicate that in the analytic study a search must be made for substances belonging to a great variety of chemical groups. It appears to be impossible to obtain any relationship between the dispersing properties and the constitution of any particular substance.

Many substances can be excluded, e.g., the amino acids, urea and others. Some well-defined substances are significant, such as oxalic acid.

The strong dispersing effect of soluble silicates may be one explanation of the quick-clay problem. If the clay minerals are chemically decomposed in any way, the reaction products may thus have dispersing properties which will make the material quick.

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# TABLE I

EXAMINATION OF THE DISPERSING PROPERTIES OF DIFFERENT WELL-DEFINED SUBSTANCES. IN THE CASE OF ACIDS, THE SODIUM SALTS HAVE BEEN STUDIED

Chemical group	Substance	Max. decrease of H1-value (°/ <sub>0</sub> )	Dispersing agent causing max. effect g/100 g clay
Inorganic	Sodiumdiphosphate (pyro, Na4P2O7)	96.7	0.2
	Sodiumorthophosphate (Na <sub>3</sub> PO <sub>4</sub> .12H <sub>2</sub> O)	89.0	1.0
	Commercial water glass	96.7	.0.5
	Sodium silicate, according to VESTERBERG (1914)	72.9	2.0
	Na2SiO3 Kebo S 3540	30.0	1.5
Natural substances	Humic acid, EGA-Chemie KG	96.7	0.3
·	Hymatomelanic acid from Rosshyttan	96.7	0.5
Carbohydrates	Galacturonic acid	58.0	2.0
	Cane sugar	No effect	-
	Saponine	86.0	0.7
Organic acids	Oxalic acid	92.0	0.5
	Malic acid	83.0	1.0
	Lactic acid	49.0	2.0
	Succinic acid	59.0	1.0
Higher fatty acids	Commercial soap	96.7	1.0
	Sodium stearate	17.0	2.0
	Sodium oleate	96.7	1.0
	Saponified bitumen from Rosshyttan	59.0	0.4
Amino acids	Valine	Coagulates	
	Alanine	Coagulates	
	Glutamic acid	Coagulates	_
	Urea	Coagulates	-
Polyphenols	Hydroquinone + NaOH	96.7	1.0
	Resorcin + NaOH	Coagulates	_
	Gallic acid	96.7	0.5
	Tannic acid	96.7	0.3
	Quinone + NaOH	96.7	0.3

## CHEMICAL ASPECTS OF QUICK-CLAY FORMATION



ELECTRICAL RESISTANCE IN OHMS

Fig.6. The hydrogen-peroxide treated quick clay in Fig.4 dispersed to minimum viscosity with sodium pyrophosphate and somewhat dried before examination.

#### ANALYTICAL STUDY

Peat and quick clay were first examined by means of ion exchangers as used by NYKVIST (1963). The main examination was made on a profile containing the above-mentioned layer succession of peat-quick clay, located near the railway station in Rosshyttan adjacent to the railway line Sala-Krylbo in the middle part of Sweden, cf. SÖDERBLOM (1960).

Investigations were also made on quick clays and waters from different places located in the Göta River Valley in the western part of Sweden.

## TABLE II

THE DISPERSING PROPERTIES OF DIFFERENT FRACTIONS OF EXTRACTS FROM PEAT AND QUICK CLAY

Substance	Dispersing properties						
	real acids	amino acids, etc.	phenols, etc.				
Peat water from Rosenkälla (somewhat aged)	Will disperse obviously, but not so strongly	Coagulates	Will disperse strongly				
Peat water from Rosenkälla (fresh)	Will disperse obviously but not so strongly	Coagulates	Will disperse strongly				
Aceton extract from quick clay from Dalsland	Will disperse strongly	Coagulates	Will disperse very weakly				
Peat, 1.5 m	Weak	Verv weak	Very good				
Peat, 2.0 m,	Weak	Very weak	Very good				
Peat, 2.5 m,	Good	Weak	Good				
Contact, 2.9 m <sup>1</sup>	Rather good	Good	Good				
Clay, 3.0 m	Rather good	Verv weak	Good				
Clay, 3.5 m	Rather weak	Weak	Good				
Clay, 4.0 m	Rather good	Weak	Good				
Clay, 4.5 m	Bad	Weak	Good				

<sup>1</sup> Peat-clay.

To extract organic substances with dispersing effects from quick clay, 90% aceton-water was found to be suitable. The extract was evaporated to dryness and the water-soluble part examined.

Filtered extracts of natural water were first allowed to pass a cation exchanger column, IR 120. The fluid passing the column contained all anions (organic and inorganic) and non-ionic substances, i.e., carbohydrates. This solution was passed through a column of anion exchanger resin, IRA 410  $CO_3^{2-}$ . The solution which passed the two resins thus contained the non-ionic substances.

The cation exchanger containing the cations and amino acids was eluated with  $1NNH_3$  and the anion exchanger containing the acids in the water was eluated with 1NHCl. The original extract was thus divided into three parts, one containing cations and amino acids, one with real acids and one non-ionic fraction. The three extracts were evaporated to dryness, qualitatively examined for dispersing properties (Table II) and analysed by paper chromatography.

Amino acids were present in the extracts in very small amounts. As has been stated above, amino acids are of no further interest in this examination since they have no dispersing effect. For the sake of completeness, the amino acid fraction in the Rosshyttan peat water was examined chromatographically in the system, butanol-acetic acid-water (4-1-5), and it was possible to identify  $\alpha$ -alanine, leucine and glutamic acid.

#### CHEMICAL ASPECTS OF QUICK-CLAY FORMATION



Fig.7. The kaolinite in Fig.5 dispersed to minimum viscosity with sodium pyrophosphate, the resulting slurry being examined.

The real acids are of greater interest. As will be seen in Table I the sodium salt of this fraction has dispersing properties. The real acids were examined by onedimensional paper chromatography with the solvent system, butanol-formic acidwater (4-1-5). The chromatograms were developed by spraying with brom-phenol blue.

Neither in the waters nor in the quick clays was it possible to find any identifiable acids. Instead, the chromatograms in general had the appearance as shown in Fig.9. Four or sometimes five acids with low  $R_F$ -value and a tendency to form tails were found. Sometimes an acid with the same  $R_F$ -value as lactic acid appeared. Hitherto it has not been possible to determine the structure of these acids. The acids with the highest  $R_F$ -values can be isolated by means of a cellulose column. They have dispersing properties and may be quinoidic.

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Experiments were made to relate the acids found to defined acids found in

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KAOLIN HYMATOMELANIC ACID



Fig.8. Typical dispersion curve.

soils by different authors, e.g., picolin-carbonic acid, dihydroxystearic acid and others, but gave negative results.

The acids were methylated with  $CH_3OH-H_2SO_4$  (90–10) and the esters obtained examined with thin layer chromatography, Silicagel G, solvent diethylether-petrolether (3–7). An obvious spot of fatty acids was found. The acids in question will thus contain fatty acids, probably in the form of complex lipids. As mentioned above, the sodium salts of fatty acids (soaps) are good dispersing agents. Fatty acids, therefore, seem to form part of the components giving the surface properties of the clays.

The non-ionic fraction showed, in every case, strong dispersing properties, as seen in Table II. It was examined for reducing sugars but with negative results. It was also examined for polyphenols chromatographically in the solvent system, 2% HAc-H<sub>2</sub>O (spraying with 1% FeCl<sub>3</sub>-1%K<sub>3</sub>Fe(CN)<sub>6</sub>). No simple polyphenols could be detected but instead, substances with high  $R_F$ -value, following the solvent front, were found.

Experiments were also carried out to remove the adsorbed substances in the Rosshyttan quick clay by means of acids as proposed by MICHAELS (1958). Instead of  $6NH_2SO_4$ , as proposed, formic acid was used to avoid disturbances in the chromatography. The mixture was allowed to react for 24 h and the solution was then filtered and evaporated, the residue being dissolved in a small quantity of water. A small part of the solution was examined directly by paper chromatography using

#### CHEMICAL ASPECTS OF QUICK-CLAY FORMATION



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Fig.9. Acids isolated from Swedish quick clay. Four acids exist with low  $R_F$ -values and one with a relatively high value corresponding to that of lactic acid. Standards: oxalic, malic and lactic acids.

different solvents. The remainder was treated in the ion exchangers as described previously. The different fractions were then analysed.

The chromatogram from the evaporated, untreated formic acid solution, sprayed with a reagent for phosphoric acid, (WALDI, 1961) indicated the presence of some type of, probably organic, phosphoric acid. This acid can be purified by ion exchangers and column chromatography. It has an  $R_F$ -value of 0.4, somewhat lower than orthophosphoric acid and has good dispersing properties.

Also soluble silicates are present and can easily be shown with the qualitative reagent, ammonium molybdate-benzidin-sodium acetate.

The eluate from the cation exchanger showed only traces of amino acids and the eluate from the anion exchanger acids of the same character as described before. No sugars, but complex polyphenols, could be detected in the non-ionic fraction.

By means of this experiment it was possible to isolate two typical dispersing substances from the Rosshyttan clay, namely, phosphoric acid and silicic acid.

In addition to the experiments reported here, a large number of isolation methods have been tested for the removal of dispersing agents from peat and quick clay, e.g., the methods described by O. Schreiner and E.C. Shorey in the years 1908–1930 (e.g., SCHREINER and SHOREY, 1908a, b, 1910), the isolation method of RAMAUT (1955) and of FORSYTH (1947). It is possible to isolate dispersing substances by all these methods but, at present, with the exception of fatty substances, it

seems to be impossible to separate and to analyse the resulting substances. None of the well-defined substances existing in soil according to KONONOVA (1961) was found.

The most interesting results have been obtained by a method described by SUNDGREN and RAUHALA (1949, 1965) and used by the present author as reported below.

# SPECIAL STUDIES OF FATTY ACIDS IN PEAT AND QUICK CLAY

Fatty acids are common, well-defined components in peat and have been examined in detail in the Peat and Oil Laboratory, Helsinki, under its Director, Professor A. Sundgren. By means of thin layer and gas chromatography they have succeeded in isolating and identifying eleven saturated fatty acids in peat wax. One of the most important was lignoceric acid,  $C_{23}H_{47}COOH$ .

As shown by the thin layer chromatography in the present investigation (p. 426) it was possible to find normal saturated fatty acids in the eluate from the anion exchanger.

Acids of this kind are thus components that can be expected to play an active role in the quick-clay process and they are of special interest because of the possibility of separating them into well-defined individual components by relatively simple and rapid methods.

The quick clay or peat was dried at 105 °C and was then treated with a mixture of benzene-ethanol (9-1) in a modified Soxhlet apparatus for 4 h. The brown solution of bitumen was evaporated to dryness in vacuo, the evaporation residue was dissolved in boiling 95% ethanol and the filtered solution was cooled to -20 °C. A brown yellow precipitate of wax was formed. This was separated from the resin solution by means of a centrifuge and then saponified for 8 h in 0.5 N ethanolic KOH-solution. Some 30% ethanol was then added and the unsaponifiable substances were extracted with petrol ether. The soaps were then precipitated by means of 2.5% water solution of CaCl<sub>2</sub>. The lime soaps, after drying, were methylated by means of methanol-sulphuric acid (90-10) for 4 h. The methylesters were collected in CCl<sub>4</sub> and separated from the methanol solution, after which they were examined by means of thin layer chromatography.

A typical chromatogram from the Rosshyttan peat and quick clay is shown in Fig.10. It is evident that the waxy part of the clay and of the peat has a similar composition and that the same components will be found in the clay as in the peat. Similar experiments have been undertaken with several waters, peats and clays and the results are, in every case, similar to that from Rosshyttan.

A clay wax, extracted from a quick clay from Vesten, Göta River, was further examined. The methyl esters were dissolved in a small amount of carbon tetrachloride and allowed to pass a column of aluminium-oxide according to



Fig.10. Thin layer chromatogram of methylesters from saponified bitumen extracted from peat and quick clay from Rosshyttan. I = palmitic acid; 2 = peat from Rosshyttan; 3 = clay from Rosshyttan; 4 = palmitic acid.

Brockman, activity 1 (Fluka). The column was firstly eluated with CCl<sub>4</sub> which removed the substances with higher  $R_F$ -value. After that it was eluated with CHCl<sub>3</sub> which removed almost pure fatty acid methyl esters, which were tested for purity by means of thin layer chromatography.

The purified methylesters were subjected to gas chromatography in a fractometer, "Perkin Elmers", type F6, with hydrogen flame detector, column Q, apiezon packing, temperature 250 °C.

The chromatograms seem to show the same tendencies and the same acids seem to be present as those found in peat by SUNDGREN and RAUHALA (1965). It has hitherto been possible to identify acids with 14, 16 and 18 carbon atoms, but even higher acids are present.

#### DISCUSSION

The present work is a first attempt at studying the importance of surface active substances in the natural quick-clay process. The introductory rheological experiments (Fig.1-7) indicate the presence of dispersing substances in quick clay, and in the following experiments attempts have been made to remove and analyse these substances. It is easy to isolate dispersing substances of different types of chemical composition, such as acids, polyphenols, tannins, etc. from either quick clay, peat or natural waters, but unfortunately in most cases the substances isolated are of complicated composition and are impossible to separate further and analyse.

The experiments using the Finnish methods gave an indication of the occurrence of homologous series of fatty acids in the clays. The extractable bituminous material varies from about 0.5-1.0 g/kg of dried clay, but the extraction with benzene-ethanol is relatively mild and it is possible that the amount is larger. The quantity seems to be somewhat too small to explain completely the process of quick-clay formation. But of prime interest is the fact that there are substances present which can be broken up into individual components. These components are surface active and must necessarily affect the clay.

All quick clays also contain substances of the type "hymatomelanic acid", probably a mixture of resin- and quinoidic acids, having very strong dispersing properties, as shown above, but at present it seems to be impossible to analyse this complicated mixture. However, one can expect that the chemical individuals in this "acid", as in the case of fatty acids, will occur in homologous series. Hitherto suitable analysing methods have been worked out only for the fatty acids but this will not preclude the possibility of developing methods for other types of substances as well.

The results obtained show some interesting aspects of the time-dependent constants of geotechnical properties. Experiments have shown that in the laboratory it is easy to change the properties of a clay sample in a few days, but the question arises as to whether or not it is possible to change the geotechnical properties of a natural clay profile by means of dispersing agents. In the vicinity of Timrå railway station in northern Sweden a waste dump giving off phosphates was placed on a clay with normal sensitivity, the profile of which had been examined in 1934 with no quick clay being found (JERBO, 1964). In 1961 the profile was quick and, because the dump was placed in 1958, a quick-clay formation seems obviously to have taken place in three years. It must be pointed out that the clay contains silt layers.

These indications should encourage systematic field examinations into the possibility of changing the geotechnical properties by chemical means. A dispersing agent should be injected into permeable layers in the clay and the expected resulting formation of quick clay studied. At the same time quick-clay profiles must be injected with substances which will disturb the natural chemical equilibrium and start the alteration process mentioned above. Some preliminary tests seem to indicate that trivalent iron ions may be suitable.

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RECENT QUICK-CLAY STUDIES, 3

## QUICK-CLAY MICROSTRUCTURE

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#### SUMMARY

The article deals with a quick clay which was originally deposited in sea water. By leaching in situ the salt content has been reduced to a very small value.

By using a special technique for step-wise replacement of pore water with acrylate plastic, 500 Å thick sections were cut with a precision microtome. The sections were photographed in an electron microscope and the micrographs obtained could be used for a study of the clay microstructure.

The microstructure is characterized by a linkage of groups or chains of small particles in and between denser flocs or aggregates or between bigger particles. There is no preferential orientation either of small or of bigger particles.

A preliminary study of the quick clay and of unleached parts of the same clay stratum has not revealed any microstructural differences.

The extreme thinness of the clay sections means that the micrographs reveal pores larger than about 500 Å. Thus the micrographs give a fairly complete picture of the size and shape variation of the micropores in the clay. By measuring the maximum dimension of all pores which could be identified and by using suitable methods for statistical condensation, representative values of mean pore size and two-dimensional "porosity" were obtained. These characteristics are discussed in relation to the permeability and strength properties of the quick clay and of some fresh- and brackish-water deposited clays which have been investigated previously.

Finally, on the basis of the micrographs a hypothesis is made concerning the rate of settlement.

#### INTRODUCTION

In connection with a current investigation of clay microstructure, some quick clays from the Göta River Valley have been studied at the Swedish Geotechnical Institute. Thin sections were prepared according to a method described previously by the author (PUSCH, 1966) involving replacement of the pore water by acrylate plastic in a diffusion process. Comparative studies, as yet unpublished, have shown this method to be satisfactorily accurate as regards the preservation of the original microstructure.

Undisturbed clay samples were taken from 6 m depth with the Swedish 50 mm standard piston sampler at Lilla Edet about 45 km north of Gothenburg.

At this locality there is a 15 m thick stratum of Late-Glacial quick clay with the ground water level about 1 m below the ground surface. According to micro fossil analyses made by the Swedish Geological Survey, the clay was formed under cold and temperate marine conditions about 15,000 B.C. The clay stratum has been leached as can be seen from the very low present salt content. Since the sensitivity is of the order of 150, this seems to be an application of ROSENQVIST'S (1946) wellknown theory concerning the cause of sensitivity. The most important geotechnical data of a representative clay sample are given in Table I.

#### TABLE I

GEOTECHNICAL DATA OF THE INVESTIGATED SAMPLE (4643) TAKEN AT 6 M DEPTH

γ (g/cm³)	$ au_{fu}^1$ $(kg cm^2)$	Sensitivity (St)	w (°/₀)	₩L (°/₀)	w <sub>P</sub> (°/ <sub>0</sub> )	Activity (ac)	Ignition loss (°/0)	Clay content (°/ <sub>0</sub> )
1.50	0.16	147	91	58	29	0.40	3.0	72

<sup>1</sup>Cone test.

#### BASIC IDEAS ABOUT THE MICROSTRUCTURE OF MARINE CLAYS

Sedimentation in marine environments is generally supposed to involve flocculation and rapid settlement, leading to a porous system of particles. Different idealized images of the particle arrangement have been suggested, the most wellknown being CASAGRANDE'S (1932), TAN'S (1957) and LAMBE'S (1958). The two last-mentioned types are based on the supposition that clay particle size is of no importance in the flocculation process. According to Lambe, "salt flocculation" gives an orientation approaching parallelism due to the following reasoning. Assuming the particle edges to be positively charged and the flat surface to carry a negative charge, a "non-salt flocculation" gives a perpendicular array whereas a high salt content gives a more parallel particle arrangement due to the decreased attraction (because of edge charge reduction by anion adsorption) and to the decreased extension of the electrical double layers.

Tan's hypothetical picture of a marine clay shows a regular arrangement of uniform particles connected edge to flat surface.

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Casagrande's microstructural arrangement indicates—at least on the silt fraction scale—the importance of particle size, the flocculated smallest particles forming links between bigger particles.

Only indirect evidence concerning the real existence of these suggested types of mutual orientation of adjacent clay particles has been given with the exception of some investigations of replicas of freeze-dried clay samples. Such an investigation was made by ROSENQVIST (1958) who stated, on the basis of a study of about a hundred stereoscopic photos of Scandinavian virgin marine clays, that the clay particle arrangement is of the type suggested by Tan.

### PREPARATION OF THIN SECTIONS AND ELECTRON MICROGRAPHS

In the present microstructural investigations which are being carried out at the Swedish Geotechnical Institute, very good opportunities exist for investigating the particle arrangement as well, but until now the work has been concerned only with the system of the smallest pores. Some preliminary results will be given in this article.

Although there are certain difficulties in the preparation of ultra-thin (300– 700 Å) sections of plastic-treated clay by using a microtome in the study of small pores, the advantage is great compared with working with thicker sections or with replicas. This is because the very thin section represents a two-dimensional cross section through the clay whereas a section with a thickness of several  $\mu$  may not reveal the majority of the smallest pores. Besides, such thick sections—generally used in the field of pedology—are not penetrable by the electron radiation in ordinary electron microscopes. The smallest pores, however, can only be identified in such instruments. The replica only gives a rough picture of the microstructure depending on the "topographic" variation of the fracture surface under investigation.

The author's investigations were made with a Siemens Elmiskop I, which belongs to the Department of Histology, University of Lund. The microscope, which was equipped with a platinum-iridium aperture of 50  $\mu$  in the objective lens, was operated at 80 kV voltage.

Ocular inspection at a magnification of  $400,000 \times$  at the utmost, was performed and representative parts of the thin sections were photographed, the micrographs being taken at random. By identifying the special shape of the sections, their orientation in relation to the horizontal plane in situ was determined. All sections correspond to vertical planes in situ.

The electronic magnification during photographing was about  $5,000 \times$  to  $10,000 \times$  depending on the size of the object. The plates used were Ilford "Special lantern".

Almost all thin sections from pure clay can be used. When the diamond edge

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of the microtome meets coarser particles being harder than the plastic, these coarser particles are sometimes pushed up in front of the edge or are cut loose. This can cause a change in the clay structure in the vicinity of the coarse particles. By observing the cutting process through the eye-piece of the microtome, those sections which appear to be intact can be selected.

From electron micrographs it is possible to study in detail the structure and the pore system of clays. The photographs can also be used to investigate statistically the particle arrangement and then a mathematical expression for the degree of orientation can be obtained.

In Fig.1 and 2 two representative micrographs are shown of the quick clay. The most typical features of this clay compared with fresh-water clays, which are being studied, are the great number of dense aggregates and the open network of particles and aggregates. Also, the number of partly split particles is noticeable. As can be concluded from the intact film of the embedding substance the splitting is not an artifact. There is no preferential orientation either of small or of bigger particles. A very distinct feature is the linkage of groups or chains of small particles in and between denser flocs or aggregates or between bigger particles (Fig.3A, B). This kind of particle arrangement is almost identical with the hypothetical floc or aggregate structure suggested earlier by the author (PUSCH, 1962, pp.53, 55). Of special interest is the fact that there is no significant difference between the microstructures of samples from 3 m depth (the dry crust) and 6 m depth (the quick clay). Furthermore, the same type of structure was observed in a sample from 19 m depth taken from another bore hole at a distance of 150 m. This sample represents a part of the same clay stratum which has not been leached and where the sensitivity is of the order of 10. A more detailed study may, however, reveal differences between the structures of the quick and normally sensitive clays; for instance as regards the frequency of weathered minerals. The presence of weathered particles, which are mechanically weak due to partial splitting, may be connected with the sensitivity.

DEFINITION AND DETERMINATION OF SIZE AND SHAPE OF MICROPORES AND OF TWO-DIMENSIONAL "POROSITY"

Direct comparison between the microstructures of different clays necessitates a description based on statistics. A simple method is suggested in this report. It includes determination and description of the size and shape distributions of the pores and of the pore area in percent of the total area of the thin section studied. By this means, information may be obtained concerning the permeability and strength properties.

As regards these properties of a clay material in bulk, macrostructural larger pore systems, fissures, cracks and discontinuities are also important or even decisive. Although the results therefore may not always be directly applicable to the macrosystem, the statistical description of the microstructure indicates a difference between different clay types which is in some respects related to the mechanical properties in bulk.

The dimensions of the pores were defined in accordance with the author's concepts used for clay particles (PUSCH, 1962). Thus, the longest intercept  $a_p$  and the measure  $b_p$  of the widest part perpendicular to the long axis were measured (Fig.4). Since the pore image represents a cross section without reference to the real extension and orientation of the pore, the dimensions  $a_p$  and  $b_p$  only describe the sectioned pore. Because of the small thickness, about 500 Å, of the clay slice, the microstructural image corresponds to a two-dimensional vertical section through the natural clay mass. A convenient expression of the pore shape was found to be the quotient  $a_p/b_p$ . In the distribution diagrams, the pore size is represented solely by  $a_p$ . The calculation of the pore area was based on planimeter measurements.

The measurement was based on drawn images, Fig.5 and 6, of the micrographs. On the drawings, which show no discrete particles, the pores are marked white. Pores extending outside the micrograph edges were measured as if they had this truncated shape. Because of this, the pore size and shape distributions are somewhat affected. It is, however, of almost no importance as regards the calculated values of the statistical characteristics. Due to the limited area of the micrographs, the maximum pore size which could be registered was about 6  $\mu$ . Studies over areas of the order of 50  $\mu \times 50 \mu$  seem to give the same size of the majority of the pores as in the smaller areas.

Histograms representing  $a_p$  are given in Fig.7 and 8. The class intervals agree in the main with the ones in the graphical representation of clay particle size. The median measures, skewness measures and sorting coefficients are shown in Table II which also gives the total pore area in percent of the total studied area. The frequency of smaller pores is probably not representative. This is because the thickness of the clay sections did not reveal a certain number of very small pores. Furthermore, pores with an  $a_p$ -value smaller than 0.031  $\mu$  could not be identified with certainty. Such pores will, however, act more or less as the surrounding minerals due to the high water viscosity and are therefore probably of minor importance.

The median values of the pore dimension  $a_p$  and of the ratio  $a_p/b_p$  are of the same order in the Lilla Edet Clay and in fresh- and brackish-water deposited clays. Also, the average distance between the pores seems to be the same, but the micrographs of the Lilla Edet Clay show the presence of a certain number of large pores. The existence of these pores, which can also be shown statistically for instance by using quartile analyses, is a typical feature of the marine clays in Lilla Edet. They belong mainly to the "fine pores" (20-2  $\mu$ ) as defined by ODÉN (1957) whereas the majority of the pores in the sections investigated belong to the "ultra pore" fraction (<0.2  $\mu$ ) and to the finer part of the "micro pore" fraction (2-0.2  $\mu$ ).

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#### TABLE II

#### PORE PARAMETERS

Sample	Micro- graph	$a_p$ $(\mu)$			$a_p/b_p$			$S_K^4$ of $a_p$	S <sub>K</sub> of a <sub>p</sub> /b <sub>p</sub>	$log S_0^5$ of $a_p$	log S <sub>0</sub> of	Pore area (°/ <sub>0</sub> of total
		$M^1$	$Q_{1^2}$	Q3 <sup>3</sup>	М	$Q_1$	Q3				$a_p/b_p$	area)
4643 (Strand-	16252	0.13	0.09	0.25	1.92	1.58	2.40	1.33	1.03	0.221	0.091	47.8
backen)	16257	0.17	0.10	0.33	1.99	1.54	2.60	1.14	1.01	0.259	0.114	48.2

<sup>1</sup>Median value.

<sup>2</sup>Lower quartile.

<sup>3</sup>Upper quartile.

<sup>4</sup>Skewness (TRASK, 1932) ( $Q_1 \cdot Q_3$ )/ $M^2$ .

<sup>5</sup>S<sub>0</sub> = sorting coefficient  $\sqrt{Q_3/Q_1}$ ; log S<sub>0</sub>- values are directly comparable.

## MICROSTRUCTURAL FEATURES WITH REFERENCE TO PHYSICAL PROPERTIES

In comparison with 18 micrographs of a fresh- or brackish-water deposited clay  $(S_t = 10-20)$ , the relative pore area of the clay from Lilla Edet is about three times greater. There seems to be a logical relationship between the observed "porosity" and the permeability. Thus, the permeability in the vertical and horizontal directions, expressed in terms of the permeability coefficient, was  $7 \cdot 10^{-8}$  cm/sec for the Lilla Edet Clay and about 25% of this value for the fresh- and brackish-water deposited clay investigated. The permeability was calculated from oedometer tests in which the influence on the percolation process of a few very large pores is much less than in direct permeability tests. Although disturbances, especially the successive break-down of the structure during the compression, influence the drainage process (PUSCH, 1962, pp. 70–72), investigations of different clays probably give at least an idea of the relative permeability.

The relative pore area in marine clays is considerably larger than in freshwater deposited clays with similar unit weight and water content values. This indicates that the particle groups and aggregates represent different densities in marine and fresh-water deposited clays. Also, a different density may imply a different permeability of the particle aggregates themselves. The largest openings in the sectioned, black aggregates have  $a_p$ -values smaller than 0.031  $\mu$  or 310 Å since larger areas would have been registered as pores in the histograms. Using Low's (1958) value 60 Å for the approximate extension from the mineral surface of the ordered water with reduced mobility, it is obvious that a certain, very small permeability may also characterize the aggregates. The pictures indicate that the thickness of the adsorbed water does not govern the average permeability of this clay.



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Fig.1. Vertical section through a quick clay (sample 4643 : 1). Electronic magnification  $\times 8,000$ .

![](_page_30_Picture_2.jpeg)

Fig.2. Vertical section through a quick clay (sample 4643 : 2). Electronic magnification  $\times 8,000$ .

![](_page_31_Picture_1.jpeg)

Fig.3. Linkage in clay structure.

A. Vertical section through a quick clay (sample 4643:2). Electronic magnification  $\times 8,000$ .

B. Hypothetic floc or aggregate structure (PUSCH, 1962).

![](_page_31_Figure_5.jpeg)

Fig.4. Characteristic dimensions of pores.

As regards the strength properties of the Lilla Edet Clay, some information can be obtained from the micrographs. Firstly, the densest aggregates probably represent the strongest parts of the clay structure because of the small distance between the particles and of the increased average viscosity, or rather ordering, of the pore water. The weakest parts of the continuous particle network consist of the chain-like arrangements of particles between the aggregates. These features indicate that the clay would behave like a frictional material where the grains correspond to the clay particle aggregates. Although this idea is not contradictory to the well-known brittle behaviour of these marine clays, microstructural studies of remoulded quick clays show a complete break-down of the structure.

![](_page_32_Picture_0.jpeg)

Fig.5. Drawn image of Fig.1. The pores are marked white.

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![](_page_32_Picture_2.jpeg)

Fig.6. Drawn image of Fig.2. The pores are marked white.

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![](_page_33_Figure_1.jpeg)

Fig.7. Histogram of the maximum pore dimension (sample 4643 : 1).

![](_page_33_Figure_3.jpeg)

Fig.8. Histogram of the maximum pore dimension (sample 4643 : 2).

In comparison with the fresh- and brackish-water sedimented clays, an arbitrary theoretical failure plane passes through a smaller number of particle aggregates in the Lilla Edet Clay. Nevertheless, the shear strength is greater in this

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clay which would be explained by different types of particle bonds as well as differences in strength of the individual particle aggregates. However, since failure probably develops successively within a certain zone, the strength of the weakest parts, the connecting particle links, is of major importance. This question, which may also have to do with macrostructural features, will be dealt with in a separate article.

Finally, the micrographs give some support to the following hypothesis concerning the deformation properties of clays. In the time-dependent compression of a clay element, the smallest resistance is caused by an approach between aggregates, accompanied by a successive destruction and restoration of their connecting links. During this process, which may be rapid because of the high permeability of the porous system, the aggregates are also being compressed but the compression rate is low on account of the much lower permeability. When the first compression, which might well correspond to the "primary compression", has been developed, the "secondary compression" will dominate.

The experimental background has to be considerably extended before a general relationship between microstructural features and physical properties can be suggested.

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