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CLAY MICROSTRUCTURE

A Study of the Microstructure of Soft Clays with Special Reference to Their Physical Properties

By
Roland Pusch

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Lerans mikrostruktur

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Byggforskningen Sammanfattningar

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Mikrostrukturen hos några svenska lösa leror har undersökts med elektronmikroskopi och beskrivits statistiskt med hjälp av enkla strukturparametrar. Strukturmönstret karakteriserades av aggregat kopplade via länkar och grupper av små partiklar. Vissa mikrostrukturella egenskaper bedömdes ha samband med den ostörda lerans geotekniska egenskaper, t.ex. permeabiliteten och sensitiviteten. De mikrostrukturella förändringarna vid konsolidering och vid skjuvning har undersökts vilket gett underlag för ett antagande om mekanismen vid dessa processer. Aggregaten visade sig verka som fasta kroppar upp till en viss spänningsnivå. Denna fasthet kan till en del bero på aggregatens låga vattenhalt som innebär en mycket hög viskositet hos porvattnet.

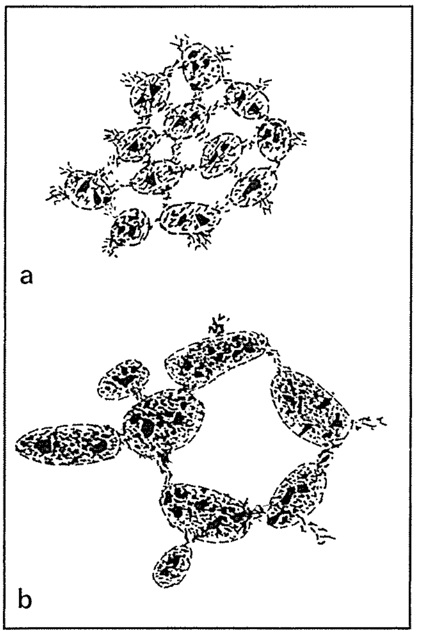
Under de sista årtiondena har åtskilliga hypoteser lanserats om den mikrostrukturella uppbyggnaden hos lösa leror. Kolloidkemiska betraktelser främst i form av teorier om elektriska dubbellager har legat till grund för dessa hypoteser som visat sig ha begränsad giltighet. Dubbellagerteorierna har lett till antagandet av en struktur karakteriserad av parallellställda partiklar. Andra teorier som gäller fördelning av elektriska laddningar på olika delar av partiklarna har fört till antagandet av partikelarrangemang av typen kant mot plan yta, speciellt i illitisk lera.

Åtskilliga undersökningar med hjälp av polariserat ljus, röntgendiffraktionsteknik, sedimentations- och krympningsobservationer samt hållfasthets- och deformationsbestämningar har utförts av olika forskare för att få indirekt information om mikrostrukturens uppbyggnad.

Ljusmikroskopiska undersökningar har givit värdefulla upplysningar om arrangemanget av mjälapartiklar och grövre lerpartiklar samt om t.ex. aggregatbildning, men den begränsade upplösningen har inte möjliggjort ett detaljstudium av arrangemanget hos huvuddelen av partiklarna i lerfraktionen.

Elektronmikroskopiska undersökningar grundade på replikametoder har givit information om mikrostrukturen men prepareringstekniken, lufttorkning eller frystorkning, kan i de flesta fall ha påverkat den naturliga strukturen. Ett bättre förfarande, som beskrivs i denna rapport, är att utföra elektronmikroskopisk undersökning av ultratunna snitt av plastpreparerad lera. I rapporten beskrivs en undersökning av tre svenska typleror: söt- eller brackvattenavsatt Skå-Edebylera, saltvattenavsatt Lilla-Edetlera och brackvattenavsatt organisk Morjärvlera. Lerornas kornstorleksfördelning — också inom lerfraktionen — undersöktes elektronmikroskopiskt och deras geotekniska egenskaper, såsom kompressibilitet, odränerad skjuvhållfasthet och sensitivitet bestämdes genom laboratorieförsök.

Mikrostrukturen beskrevs med användande av parametrarna a_p (pordiametern) och $\frac{P}{T}$ ("porositeten"). Den sötvattenavsatta leran hade lägre $\frac{P}{T}$ värde än den saltvattenavsatta. Det högsta värdet observerades för den organiska leran. Medianvärdet av a_p var av samma storleksordning för alla lerorna, men den saltvattenavsatta leran karakteriserades av en viss mängd mycket stora porer. Den mest typiska mikrostrukturella egenskapen hos alla de undersökta proven var uppbyggnaden i form av mer eller mindre täta aggregat förbundna av länkar eller grupper av små partiklar. I den saltvattenavsatta leran var aggregaten större och tätare än i de söt- och brackvattenavsatta sedimenten.



Schematisk bild av partikelarrangemanget i lera. a Sötvattenavsatt lera uppbyggd av relativt porösa aggregat åtskilda av små porer. b Marin lera med stora, täta aggregat åtskilda av grövre porer.

En jämförelse mellan strukturparametrarna och de geotekniska egenskaperna visade vissa samband. Sålunda observerades ett direkt samband mellan permeabiliteten och strukturparametern $\frac{P}{T}$. För de lerlager i Skå-Edeby som karakteriserades av de lägsta a_p- och $\frac{P}{T}$ -värdena gäller inte DARCYS lag enligt tidigare undersökningar av professor Sven Hansbo, CTH. Någon relation mellan strukturparametrar och kompressibilitet kunde inte observeras, vilket antyder att fakto-

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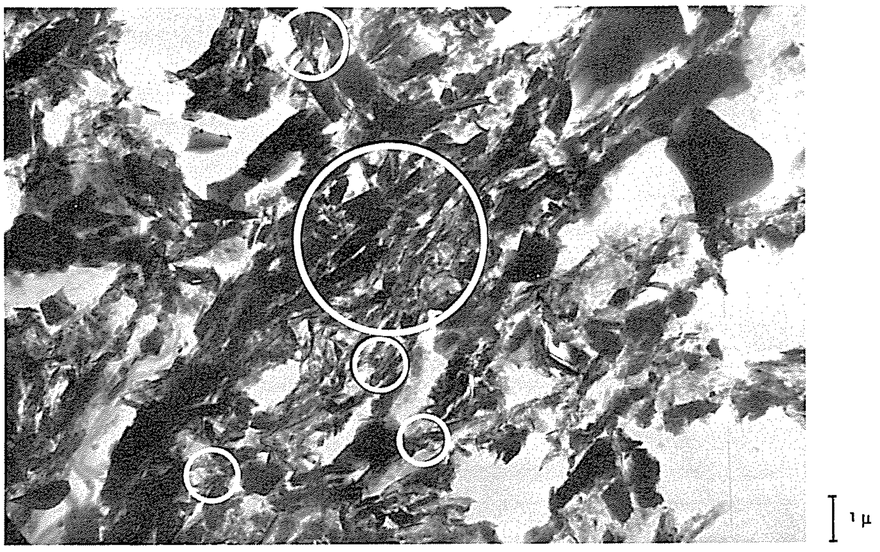

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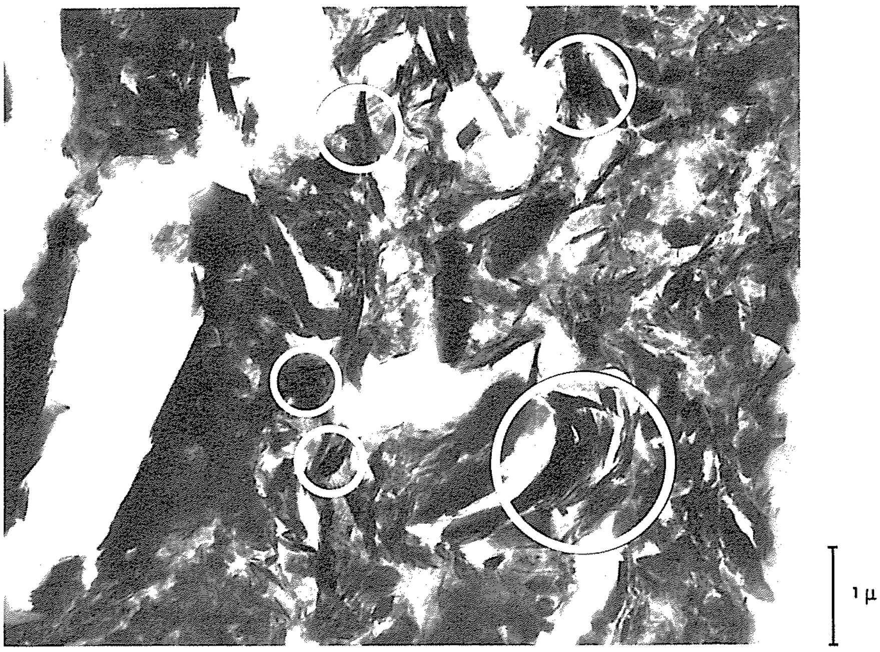

b

Domänbildning (cirkelmarkering) och aggregatdeformation vid ett konsolideringstryck av 128 N/cm². a Postglacial Skå-Edebylera från 2 m djup. b Glacial Skå-Edebylera från 8 m djup.

rer som organisk halt och kornstorleksfördelning är avgörande för kompressibiliteten.

Mikrostrukturen hos prov som konsoliderats under olika tryck undersöktes också, varav framgick att belastning utöver förkonsolideringstrycket leder till en nedbrytning och orientering av länksystemet (domänbildning). Upp till en viss spänningsnivå bedömdes aggregaten fungera som stela, hållfasta kroppar. Denna egenskap kunde också verifieras genom en undersökning i ett högvoltmikroskop av de interna deformationerna i en torkande lergel.

Inget säkert samband mellan de mikrostrukturella parametrarna och den odränerade skjuvhållfastheten kunde observeras. De högsta sensitivitetsvärdena erhölls för de lerprover som hade de högsta $\frac{P}{T}$-värdena vilket antyder att en mycket hög porositet är en nödvändig förutsättning för att en lera skall vara kvick.

De mikrostrukturella förändringarna under inverkan av olika överlagringstryck och skjuvspänningar studerades också. Av dessa studier drogs slutsatsen att mekanismen vid skjuvning av en lös lera är den, att aggregaten som verkar som stela kroppar förskjuts inbördes i samband med en nedbrytning av länksystemen som förbinder aggregaten. Länksystemen ombildas till domäner. Denna mekanism ger en förklaring till begreppet "residualhållfasthet" som antas vara den hållfasthet som provet har när de starkt deformerade länkarna är ombildade till domäner. En ytterligare störning av systemet i form av omrörning antas ge en nedbrytning också av aggregaten och med detta ett betydligt lägre hållfasthetsvärde hos provet.

Ett stöd för antagandet att aggregaten fungerar som stela kroppar upp till en viss spänningsnivå erhölls från preliminära NMR-undersökningar. De vid dessa undersökningar erhållna värdena på *spinn-spinn*koherenstiden T_2 visar att vid låga vattenhalter är vattenmolekylrörligheten mycket mindre än i fritt vatten. Eftersom den beräknade vattenhalten hos aggregaten var mycket låg har slutsatsen dragits att den höga vattenviskositeten bidrar till aggregatens stelhet.

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Clay microstructure

Roland Pusch

National Swedish Building Research Summaries

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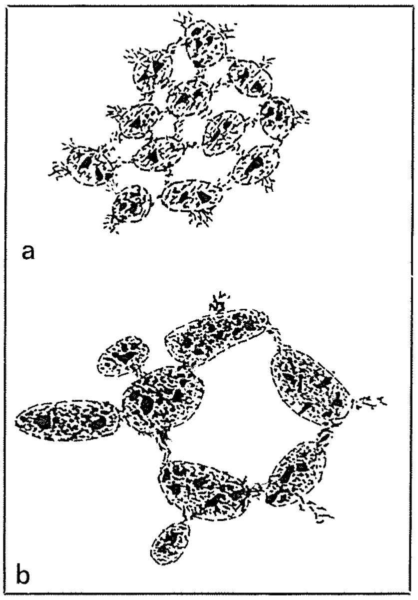
A large number of contributions to the knowledge of clay microstructure has been made in the last twenty years. Colloid chemistry has furnished most of the hypotheses suggested in literature but large discrepancies between theory and reality have been found. One of the basic ideas, the double-layer interaction of parallel colloidal particles, has been found to be applicable in specific cases only. The more recent hypotheses of different surface charges on edges and faces of clay particles form the basis of the common microstructural concept for illite clays, the edge-to-face coupling of adjacent particles.

Optical studies using polarized light, X-ray diffraction technique, settling and shrinkage observations as well as analyses of strength and deformation properties, have been tried by several authors to reveal characteristic features of clay microstructure but only rough indications have been obtained.

By direct observation of soil structure using light microscopy, a series of important observations have been made by several investigators but due to the limited resolving power no details of the clay matrix have been revealed. Electron microscopic investigations have contributed largely to the knowledge but practically all studies have been made on replicas of air- or freeze-dried specimens. This preparation technique does hardly preserve the original microstructure when applied to soft clays. Transmission electron microscopy applied to ultra-thin sections has been tried by the author and results from such investigations are presented in the paper. Acrylate-treated sections 0.05 μm thick of three Swedish soft clay types were investigated: fresh- and brackish-water sediments from Skå-Edeby, marine sediments from Lilla Edet and organic sediments deposited in brackish water from Morjärv. The clay particle size and shape within the clay fraction and the mineralogical compositions were also investigated. The geotechnical properties of the clays such as compressibility, undrained shear strength and sensitivity were determined as well.

The microstructural patterns were described by the structural parameters a_p (pore diameter) and $\frac{P}{T}$("porosity"). It was found that the fresh-water clays had



Schematic clay particle arrangement. a) Clay deposited in fresh water having relatively porous aggregates and small voids. b) Marine clay with large, dense aggregates separated by large voids.

the smallest $\frac{P}{T}$ -values, and that the highly organic clays had $\frac{P}{T}$ -values which were even higher than those of the marine clays. The median value of a_p was of the same order for all the clays, but the marine clays contained a small number of very large pores per unit sectioned area. The most important observation was that all the clays were characterized by micro-aggregates connected by links of particles. It was also found that the aggregates of the marine clays were much denser than those of the fresh- and brackish-water clays.

In the last part of the paper the correlation between microstructural features and physical properties is discussed.

A direct relationship was found between the permeability and the $\frac{P}{T}$ -value which means that the microstructural parameters are representative of clay volumes of several cubic centimeters. The clay strata with the smallest a_p- and $\frac{P}{T}$ -values at Skå-Edeby were found to be those for which professor Sven Hansbo observed that DARCY's law is not valid.

No relationship was found between the

The microstructure of some soft Swedish clays has been investigated by electron microscopy and described by statistical methods using simple structural parameters. The microstructural pattern was found to be characterized by aggregates coupled by links and groups of small particles. Certain microstructural properties seemed to be related to the geotechnical properties of the undisturbed clay material, such as the permeability and sensitivity. The microstructural changes by consolidation and shearing have been investigated. The results formed the basis of a hypothesis concerning the mechanism of these processes. The aggregates seemed to act like rigid bodies up to a certain stress level, the rigidity probably being connected with their low water content and the very high viscosity of the pore water.

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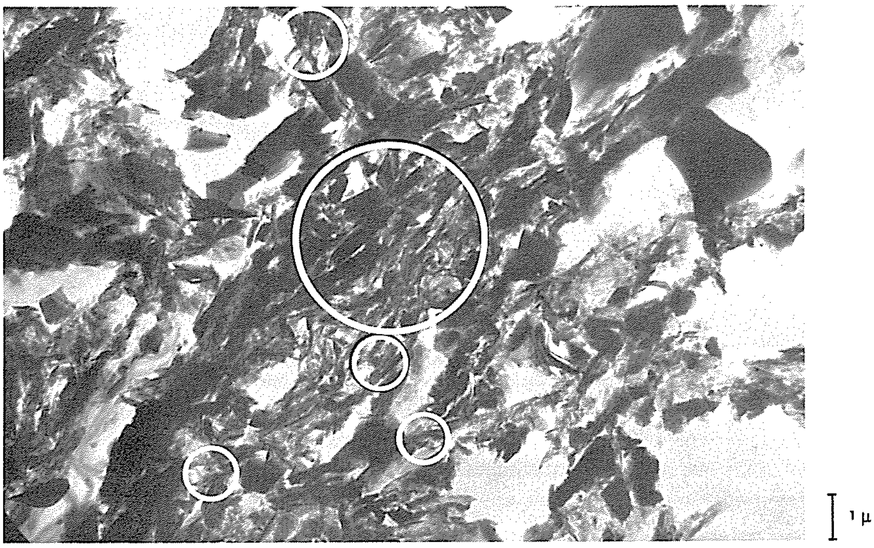

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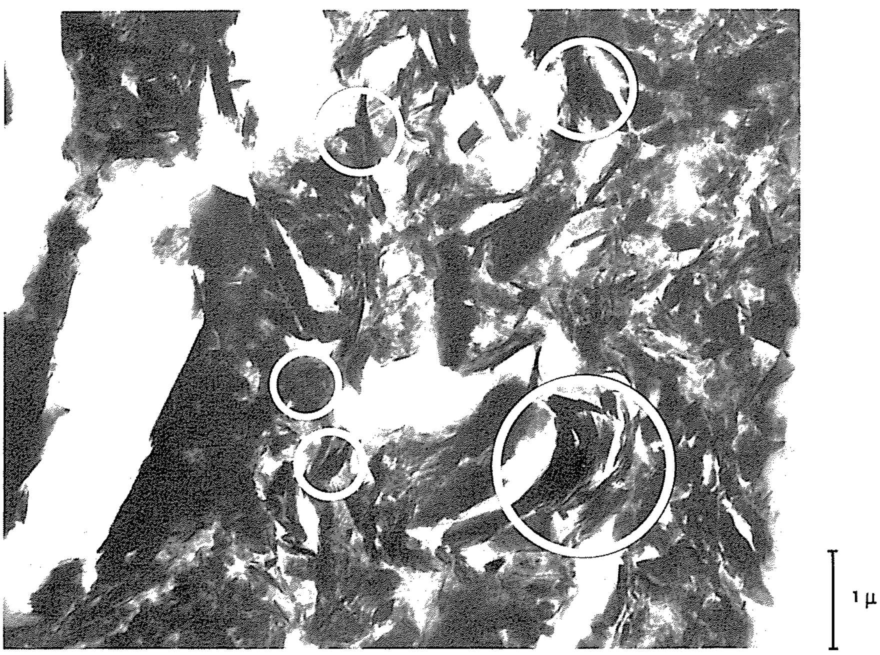

b

Domain formation (encircled) and aggregate deformation at a consolidation pressure of 128 N/cm^2. a) Postglacial Skå-Edeby clay from 2 m depth. b) Glacial Skå-Edeby clay from 8 m depth.

compression properties and the microstructural parameters. This indicates that other factors such as organic substance and particle size distribution may be most important. Special microstructural investigations of compressed samples showed that the haphazardly arranged linking particles between the aggregates were changed to domains. The aggregates behaved as fairly rigid bodies to a certain stress level. This behavior was also observed in an investigation of a drying clay/water gel in a cell placed in the electron path of a high-voltage electron microscope.

There was no relationship between the microstructural parameters and the undrained shear strength. As in the case of compression, physico-chemical and granulometric properties seem to be decisive for the strength. Very high sensitivity values were found only for clays with very high $\frac{P}{T}$ -values. Although physico-chemical processes, such as leaching, may provoke a very high sensitivity, an open particle network thus seems to be a necessary prerequisite. Special microstructural investigations of the detailed shear processes showed the same characteristic pattern as observed in compressed samples. It was concluded that the mechanism of shearing of soft clays is that the aggregates, which form strong units, are mutually moved in connection with a failure of their links. The motion of the aggregates involves a large deformation of the links which results in a parallel orientation of the linking particles. This mechanism may also furnish an explanation of the concept "residual strength". This strength may correspond to the state where the links are inactive through large internal deformations. A further distortion in the form of remoulding causes a break-down of the aggregates leading to lower bulk strength values.

The investigations indicated that the rigidity of the aggregates is basic to the fundamental properties of the clays. Certain evidence of the rigidity of the aggregates was obtained from a preliminary series of NMR tests. The calculated spin-spin coherence time T_2 at low water contents indicates that the molecular mobility of the water at mineral surfaces is much smaller than that of free water. Since the calculated water content of the aggregates was of this low order, it was concluded that the high water viscosity contributes to the rigidity of the aggregates.

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ROLAND PUSCH

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CONTENTS

INTRODUCTION

1 DEFINITIONS

The important physical properties of soils, such as permeability, compressibility and shear strength depend on the structure, which is defined as the organization of the soil constituents. The arrangement of the smallest soil units, primary particles and particle aggregates, illustrates the microstructure, while the appearance of an exposed soil profile reflects its macrostructural constitution (BAVER, 1956). Macrostructural features, such as laminations and cracks, have to be taken into account when solving soil mechanical problems in which larger volumes are involved. The microstructure of certain soft clays with special reference to the physical properties of fairly small specimens is the main topic of this report.

The purpose of the study was primarily to collect information about natural soft clay microstructure — mainly illitic — obtained from investigations using various techniques, and to select a suitable method for the author's work.

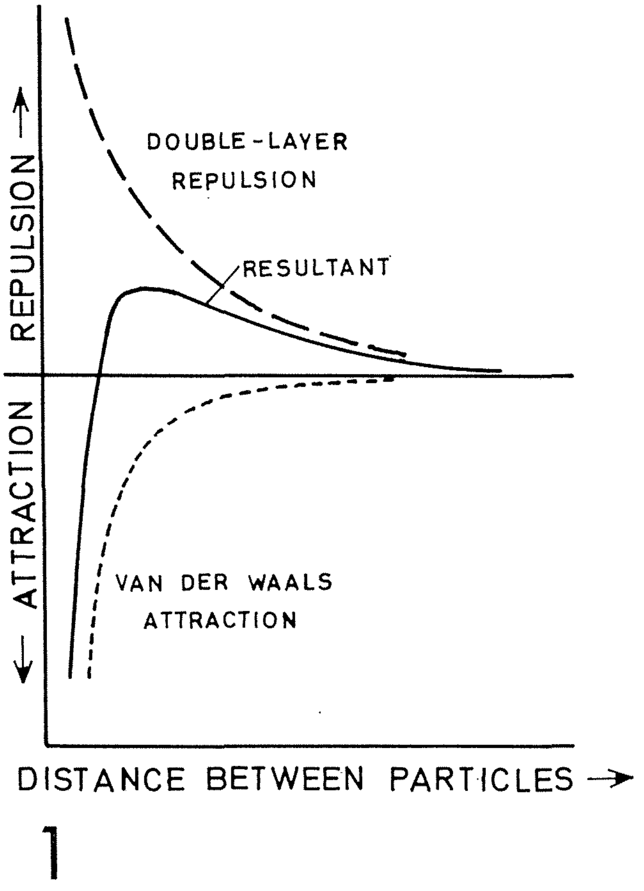
2 CLAY MICROSTRUCTURE CONCEPTS

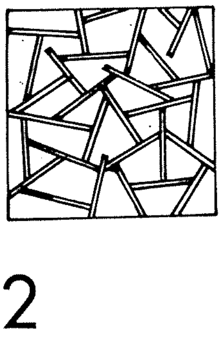
2.1 Theories of colloid chemistry

The earliest information regarding the detailed particle arrangement in clays has mainly been inferred indirectly from theoretical studies of interparticle forces operating between particles in colloidal suspensions. The interaction is supposed to contain a repulsive factor caused by electrical double-layers and an attractive factor due to (LONDON-) VAN DER WAALS forces. The total interaction is represented by superposing the repulsion and the attraction curves (Fig. 1). In the case of clays the double layers are formed by isomorphous substitutions in the mineral lattices or by broken bonds or lattice defects, leading to a net negative charge.

According to OVERBEEK (1952), the repulsion can be represented by an exponential function with a range approximately equal to the extension of the double-layer, whereas the attraction decreases at some inverse power of the distance. The VAN DER WAALS forces, which create the attraction, result from the nearness of one particle to another and the concomitant overlapping of atomic forces in the crystal lattices. The range of these attractive forces is assumed to be of the order of colloidal dimensions. The influence of the concentration and kind of cations in the liquid phase on the inter-particle distance is commonly explained by the theoretical properties of electrical double-layers. An increased concentration of cations reduces the zeta-potential and the range of the double-layer, thus reducing the repulsive force between the particles. This is a currently favoured explanation of the influence of electrolyte concentration on clay particle flocculation. According to BOLT (1956) parallel arrangement of the particles corresponds to the position of minimum free energy of the system, which means that deviations from this arrangement should be very small. Therefore the calculation of the interaction between double-layers and VAN DER WAALS forces is generally based on the assumption that all particles have parallel basal surfaces so that the forces induced by the particle edges are not considered. Certain experimental evidence supports this theory of interaction of forces, and a parallel orientation of the particles under certain conditions has in fact been proved. A series of tests with clay-water suspensions performed by BOLT & MILLER (1955), BOLT (1956) and MITCHELL (1960) provides an illustration. These investigators compared the actual mean distance between the particles with the theoretical mean distance calculated by using the GOUY-CHAPMAN diffuse double-layer theory and the VAN'T HOFF osmotic equation. The latter distance was assumed to correspond to the thickness of the double-layers between parallel particles. It was shown that for Na montmorillonite and very fine-grained ($< 0.2\ \mu m$) Na illite there was a rather close agreement between the actual and calculated particle distances at compression pressures in the range of 0—100 N/cm^2. In these tests the void ratio decreased with increased electrolyte concentration at constant pressure as predicted by the double-layer theories. Tests with coarser illite ($>1\ \mu m$) and with kaolinite showed, however, an inverse relationship between void ratio and electrolyte concentration, indicating flocculation with an irregular particle arrangement. Hence, the validity of the parallel particle concept depends on particle size. In fact, the classical double-layer theories give a much too simplified picture of interacting particles since they do not consider the structural order of the interparticle water molecules (FORSLIND, 1953).

It has been concluded from observations of the sedimentation volume, that flocculation may create various types of particle arrangement. VAN OLPHEN (1951) assumed that positive charges are present on the edges of the clay plates and that this would lead to flocculation resulting from attraction between positively charged particle edges and negatively charged particle faces (Fig. 2). The part of the edge surfaces at which octahedral sheets are broken, will carry positive double-layers in acid and neutral





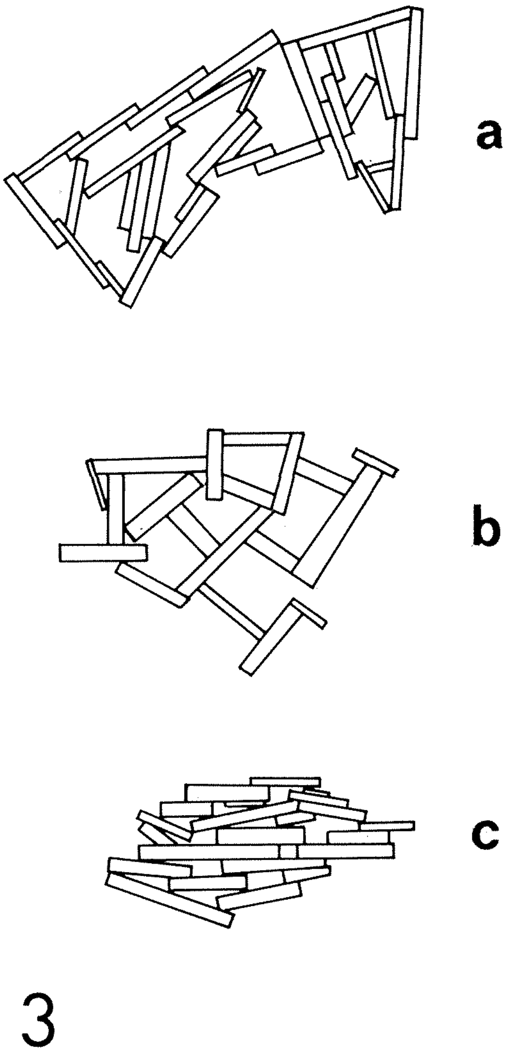


FIG. 1 Forces between basal (001) surfaces of parallel clay minerals as a function of interparticle distance, according to double-layer theories.

FIG. 2 Flocculation resulting from attraction between positively charged particle edges and negatively charged particle faces.

FIG. 3 Particle arrangement as a function of water salinity according to LAMBE (1958). a) Salt-flocculated. b) Non-salt flocculated. c) Dispersed.

solutions and negative double-layers in alkaline solutions. Where tetrahedral sheets are exposed positive double-layers may exist in the presence of very small amounts of aluminum ions in the suspension. Because of the slight solubility of the clay, such small concentrations of aluminum ions will occur in a clay suspension. Thus, under appropriate conditions, the entire edge surface may well carry a positive charge (VAN OLPHEN, 1965). LAMBE (1958) suggested that the sediment volume is influenced by the size of the hydrated ion, which decreases in the order Li>Na >K>Cs. It can be assumed, according to LAMBE, that the smaller hydrated ions allow the clay mineral particles to approach more closely, to flocculate more readily, and to orient more randomly. The structural order of the water molecules was ignored by LAMBE who assumed that particle edges are positively charged and that "non-salt flocculation" with an orientation approaching a perpendicular array may exist in clay suspended in fresh water (Fig. 3). It was assumed that addition of a small amount of electrolyte results in dispersion due to reduction of the positive edge charge by anion adsorption and that increasing salt content causes flocculation with a parallelic particle arrangement as a result of decreasing extensions of the double-layers. Experiments with kaolinite have in fact indicated that the sediment volume may have a minimum value at a certain salt content. Non-uniform distribution of surface charges (FORSLIND & DANIELSSON, 1955) and density gradients in adsorbed water layers (RESENDIZ, 1965) have also been assumed to cause edge-to-face arrangements.

Various types of bonding mechanisms have been assumed to cause other particle associations. Thus, attraction caused by an asymmetrical distribution of adsorbed cations by which simple electrostatic bonding is effected, VAN DER WAALS forces between polarized adsorbed cations, VAN DER WAALS forces between cations adsorbed on one particle and the lattice field

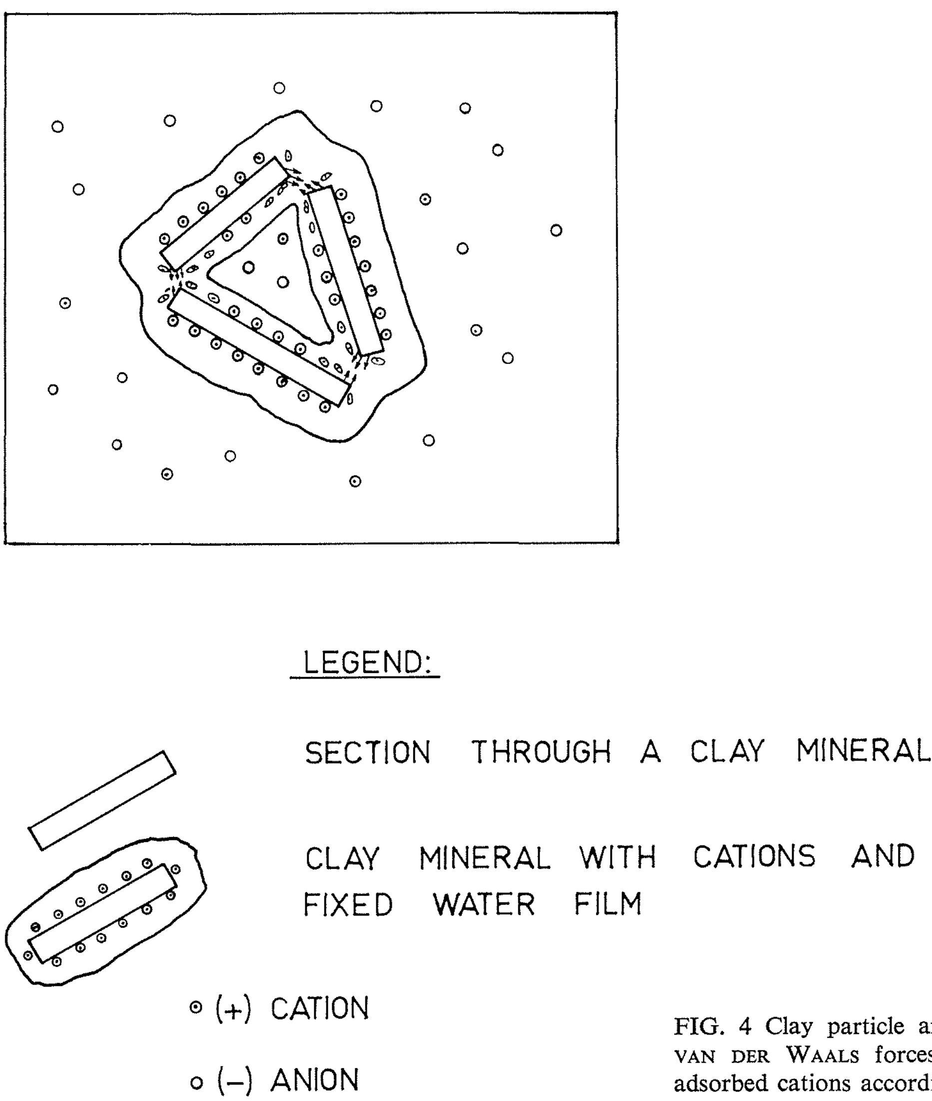


FIG. 4 Clay particle arrangement in consolidated clay. VAN DER WAALS forces established by polarization of adsorbed cations according to ROSENQVIST (1955).

of adjacent minerals, attraction by the action of adsorbed polar molecules or hydrogen bonds, may all lead to several kinds of particle associations such as edge-to-edge, edge-to-face or face-to-face (parallel) arrangements.

ROSENQVIST (1955) experimented with the sedimentation of mainly illitic material in sodium chloride solution of various concentrations. He showed that the sedimentation volume at equilibrium increased with increasing electrolyte concentration. Similar experiments with potassium and cesium chloride solutions showed that the sediment volume increased in the order NaCl, KCl, CsCl. ROSENQVIST suggested that the shear strength of the sediment was the same in all cases where equilibrium was obtained and that sedimentation equilibrium means that the sediment has reached a strength which is just sufficient to support the weight of the overlying mass. According to ROSENQVIST the volume of the sediment of a certain strength is dependent upon the degree of disorder of the clay particles. He also found a direct proportionality between the shear strength of remoulded clays and the polarizability of the ions with which the clays had been saturated. ROSENQVIST suggested the schematic arrangement of edge-connected particles shown in Fig. 4. Later he supported TAN'S (1957) idea of the edge-to-face type (Fig. 5).

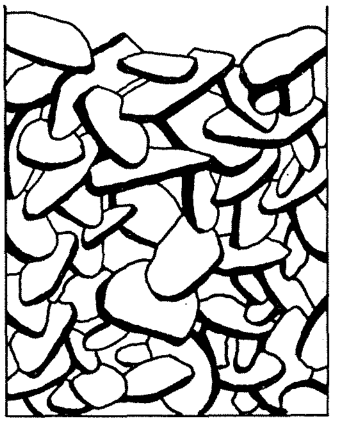


FIG. 5 Edge-to-face arrangement according to TAN (1957).

In a recent publication VAN OLPHEN (1965), still applying the concept of double-layer interaction, stated that three different modes of particle association may occur when a suspension of plate-like clay particles flocculates: face-to-face, edge-to-face and edge-to-edge. In the three cases the different combination of double-layers and the different VAN DER WAALS interaction energies due to different geometries mean that the net potential curves of interaction will be different. Thus, according to VAN OLPHEN, the three types of association will not necessarily occur simultaneously or to the same extent when a clay suspension is flocculated. Face-to-face association leads to thicker and larger aggregates, Fig. 6 (b), whereas the other modes will lead to three-dimensional, voluminous card-house structures. VAN OLPHEN suggested various combinations of particle association resulting from flocculation (Fig. 6).

Certain structural features have been reported in the literature as evidence of long-range particle interaction. Parallel layers of particles, known as Schiller layers, may be formed in stable sols at a distance of several thousand μm. These structures have been explained by the counteraction of gravity forces by double-layer

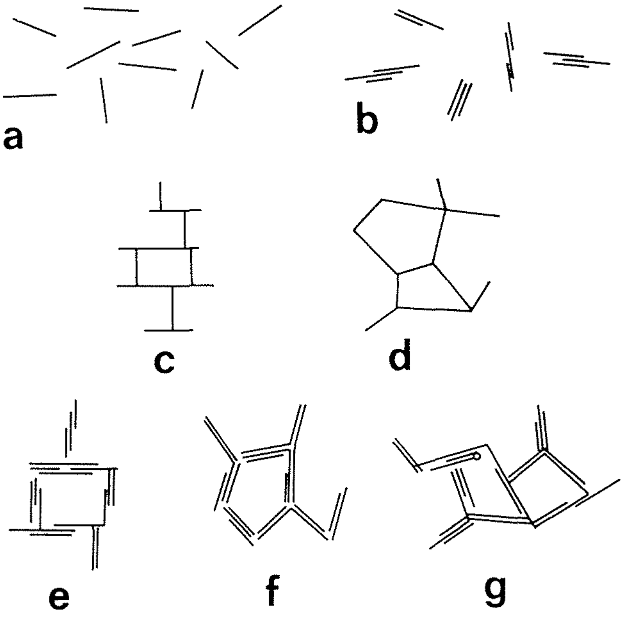


FIG. 6 Various modes of particle association in clay suspensions (VAN OLPHEN, 1965).

repulsion forces (VAN OLPHEN, 1965). Other structura features are regions of particles, tactoids, oriented parallel to each other at an interparticle distance of the order of 0.01 μm. A detailed discussion of tactoid formation has been given by OVERBEEK (1952).

According to MEADE (1964, p. B18), groups of parallel particles (domains or turbostratic units, Fig. 7) are assumed to form in clays under low pressure in laboratory tests whereas there is little evidence to support the formation of such structures in natural clay sediments. The formation of domains may be enhanced when calcium is the principal exchangeable cation and when domain-like aggregates, deposited as such, are available for nucleation of further material.

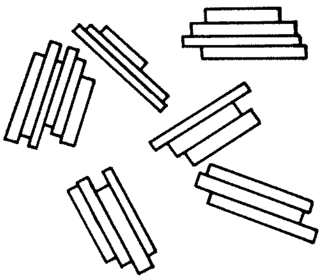


FIG. 7 Turbostratic arrangement (MEADE 1964).

Recently, modern Soviet research results concerning flocculation processes with special reference to clay microstructure have been published by OVCHARENKO *et al.* (1967). It was stated that the formation of a flocculation (coagulation) structure in a dilute suspension is induced by the cohesion between the colloidal particles driven by Brownian movement. The colliding particles, which are assumed to join at the least hydrophilic sites, are said to be separated by thin residual water interlayers at the points of association. An open network is promoted by an anisodiametric shape of the particles. According to DERYAGIN's cohesion theory two particles are connected where the radius of curvature of the mineral surfaces is minimum, that is where the dispersing medium is most easily displaced (Fig. 8). This implies that the flocculation structure consists of particles connected edge-wise. Furthermore, the type of exchange cation is assumed to be important as regards the particle arrangement. The authors claimed that Ca clays are more hydrophilic and bind water more firmly than Na clays and that the bonding between particles is much stronger in the former. In Na clays on the other hand, the links were supposed to be more numerous.

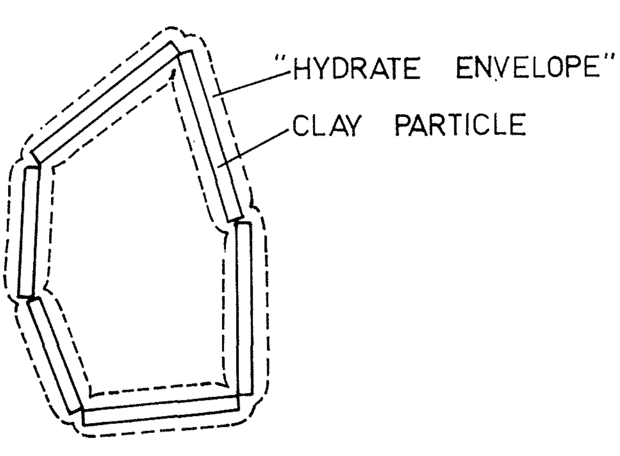


FIG. 8 "Reticular" structure in clay gels (GUMENSKII & KOMAROV).

It can be concluded from the wide variety of statements, that theories of colloid chemistry are of limited assistance in the establishment of any real concept of the clay particle arrangement in natural clays. It is also apparent that the common schematic pictures of clay particle arrangements, such as that of TAN, ignore variations in clay particle size.

2.2 Optical studies with polarized light

Optical study with polarized light has been applied in order to obtain information about microstructural features. The method is based on the fact that a randomly oriented group of clay particles appears uniformly grey in plane polarized light, whereas the particles behave optically as one large body and have definite optical properties if they are aligned parallel to each other. The preparation of sufficiently thin, undisturbed specimens has been the main obstacle in the practical application of this method. Early investigations by WILLIAMSON (1941), WEYMOUTH & WILLIAMSON (1953) and others were made by using thin sections prepared by impregnating the clay with plastic (pyroxylin). The plastic film with embedded clay was peeled off from the clay after solidification. MITCHELL (1956) introduced a preparation method which involved replacement of soil moisture with a polyethylene glycol compound (Carbowax 6000). This material is hard at room temperature, melts at about 55°C and is soluble in water in all proportions. Within three days, the heated glycol compound replaces the water in a small clay cube and, after cooling, the clay has a hardness comparable to that of talc. Thin sections of approximately 30 μm thickness can be produced by a grinding procedure similar to that used for the preparation of thin sections of rock material. Alternatively, thin sections (>5 μm) can be prepared by the application of microtomy without serious distortion of the sections. MITCHELL's preparation technique, which is assumed to preserve the main features of the natural clay microstructure, has been used in several investigations. A certain influence on the particle arrangement is probably not avoided since linear shrinkage of about 8 % has been observed in the preparation of various clay types.

A number of clays from the North-American continent were investigated by MITCHELL. He found that preferred orientation—identified as a large difference in intensity between extinction and illumination—characterized mainly non-marine clays. Remoulding of marine clays seemed to cause a small improvement in the clay orientation within small areas. MITCHELL observed that the undisturbed marine clays investigated contained significant amounts of parallel clay orientation in spite of their initial random, flocculated state. This orientation was said to be caused by the presence of tightly bonded oriented aggregates—possibly remnants of the parent rock—and by one-dimensional compression in nature. According to MITCHELL silt particles probably have little influence on the strength properties of soils with clay contents higher than about 25 % because the silt particles do not touch each other but float in a matrix of clay. Magnifications up to ×350 were used in MITCHELL's investigations.

WU (1958) investigated glacial lake clays from the Great Lakes area in the United States using the same technique as MITCHELL. The ratio of the light intensity at illumination to that at extinction was designated the orientation factor. WU found that the particle arrangement of stratified clays varied from well oriented (orientation factor from 2 to 3) to almost random. Of ten investigated samples only two showed definite orientation over areas larger than (25 μm)². The salinity of the pore water was only 0.85—3 ‰. According to WU the lack of orientation of the fresh-water deposited clays can only result from a random structure. WU stated that this structure gives the clays a high liquidity index, a high sensitivity, and a low c/p-ratio. The clays with pronounced parallel particle arrangement had a low sensitivity and a fairly high c/p-ratio.

PENNER (1963) used MITCHELL's thin section technique in the study of lacustrine ("Seven Sisters") and marine ("Leda") clays and of marine clay shale ("Bearpaw"). Microscopic investigation with polarized light revealed a preferred horizontal orientation in the lacustrine clay which was composed of illite and a minor amount of montmorillonite. The marine clay consisted of micaceous minerals, feldspars and quartz. It had a sensitivity of 30—1500 and showed only slight particle alignment. In the marine clay shale, which had been subjected to a pressure of the order of 100—1500 N/cm² in nature, the particle alignment was found to be less perfect than in soft lacustrine clays. The clay shale was mainly composed of illite and montmorillonite.

MORGENSTERN & TCHALENKO (1967 *a* and *b*) observed particle orientation in shear zones of large-scale slips. These authors also found that the consolidation properties of kaolin clay with a domain-like structure were strongly influenced by the mobility of the clay domains. Very interesting results were obtained in the study of sediment volumes of natural kaolin and kaolin treated with hydrogen peroxide to remove its organic content. A suspension of organic-free kaolin was found to yield an increasing sediment volume as the salt concentration decreased, which is in agreement with double-layer theory predictions. However, the inverse effect was found with natural kaolin and this was attributed to the presence of a small amount of organic material inhibiting deflocculation. A microscopic investigation of a natural kaolin sample showed that it consisted of clusters of slightly flocculated clay, 50 to 400 μm in diameter, surrounded by thin shells of organic matter. All the microstructural investigations of MORGENSTERN & TCHALENKO were made by using specimens prepared according to MITCHELL's method.

2.3 X-ray diffraction investigations

X-ray diffractometer methods for identification of preferred orientation in clays have been used by MEADE (1961), KAZDA (1963) and several others. The use of the X-ray diffraction technique is based on the fact that the intensity of the reflection from any crystallographic plane is proportional to the quantity of material oriented so that the plane reflects X-rays according to BRAGG's law. The practical application of the method involves the preparation of plane surfaces of the clay specimen and the determination of the peak height ratio for certain characteristic reflections. There are great difficulties in preparing suitable surfaces without changing the original particle arrangement.

The large majority of structural X-ray investigations have been concerned with stiff clays and clayey rocks but some studies have been made on soft, mostly artificially sedimented clay. Laboratory investigations have shown that even under slight pressure (20 N/cm²) initially randomly oriented illite particles become oriented perpendicular to the pressure direction, whereas montmorillonite particles do not seem to be influenced (MEADE, 1964, p. B 17). Compression at very high pressures in laboratory experiments was found to cause preferential orientation in most cases, whereas many natural clayey sediments, subjected to heavy loads, did not show an increasing degree of preferred orientation with increasing depth of burial.

An investigation of artificially sedimented clays by ROSENQVIST (1962 *a*) showed that montmorillonite clays, formed in a solution of sodium chloride containing 13.5 g Na per litre, were characterized by a random particle arrangement. Leaching with a potassium chloride solution which gave a partial ion exchange and a strength increase, did not change the arrangement.

Although investigations using polarized light or X-ray diffraction techniques may give an average measure of the degree of particle orientation, no conclusions concerning the detailed particle arrangement can be drawn.

2.4 Indirect observations

Studies of the settling velocity of river-transported clay have shown that many particles in nature are aggregated and that preferred orientation does not occur even under fresh water conditions (MEADE, 1964).

OLSON & MITRONOVAS (1962) found that a Ca illite sedimented from a dilute suspension obtained a larger void ratio than did one sedimented from a concentrated suspension. This suggests that the particles from the dilute suspension formed a more open flocculated arrangement.

Shrinkage measurements have been used for investigation of structural anisotropy. If, for instance, the shrinkage of a drying clay specimen is greater in the vertical direction than in the horizontal direction, it is assumed that there is a preferred horizontal orientation of particles in the clay mass. Although it is recognized that reorientation of particles by rotation may occur during the process of shrinkage, this method has been used for preliminary investigations of structural anisotropy.

ROSENQVIST (1955) mentioned two examples of marine illite clays which showed the following shrinkage characteristics:

Vertical	Horizontal	
7.0 %	6.5 % 6.4 %	"Clay Halden"
5.5 %	6.8 % 2.4 %	"Clay Töyen" (Quick)

WARKENTIN & BOZOZUK (1961) investigated marine clays ("Leda") and a lacustrine deposit ("Seven Sisters"). The dominant clay mineral was illite in the marine clays and montmorillonite and illite in the lacustrine clay. The following average shrinkage values were obtained (calculated from WARKENTIN's & BOZOZUK's values):

Vertical	Horizontal	
21 %	18 %	Marine clays
23 %	9 %	Lacustrine clay

The present author carried out shrinkage tests on three soft, normally consolidated, illitic Swedish clays, a clay deposited in fresh water from Västervik, a marine quick clay from Kungsbacka and an organic clay deposited in brackish water, from Morjärv. The mean shrinkage values were:

Vertical	Horizontal	
15.5 %	12.5 %	Västervik
8.4 %	8.5 %	Kungsbacka
25.7 %	21.8 %	Morjärv

The investigations show that marine clays are generally characterized by structural isotropy whereas clays deposited in fresh water are structurally anisotropic. This may be due to a tendency of the particles to be oriented horizontally or to the existence of thin horizontal intercalations of coarser particles in the fresh-water clays.

Studies of strength and deformation properties have led to concepts of soil structure. TERZAGHI & PECK (1948) claimed that the difference in strength of a soft clay tested *in situ* and in consolidated undrained laboratory tests is explained by the action of coarse particles in the following way. During deposition of a mixture of grains by sedimentation, the clay is composed of honeycombed clay aggregates and randomly distributed coarser grains (Fig. 9). During compression these grains come into contact in a very unstable but self-supporting arrangement. Slight disturbance such as gentle deformation will transfer the stresses previously carried by the coarse-grain network to the interstitial clay matrix. Therefore, the shear strength of this matrix is independent of the overburden pressure and exclusively caused by thixotropic hardening. In a sample taken from a boring in a soft clay, a slight disturbance is unavoidable. A renewed application of the overburden pressure, which acted *in situ*, will therefore cause a breakdown of the original structure with a concomitant consolidation of the clay matrix. According to TERZAGHI & PECK this brings the coarser grains into contact again which permits the development of frictional resistance between these grains.

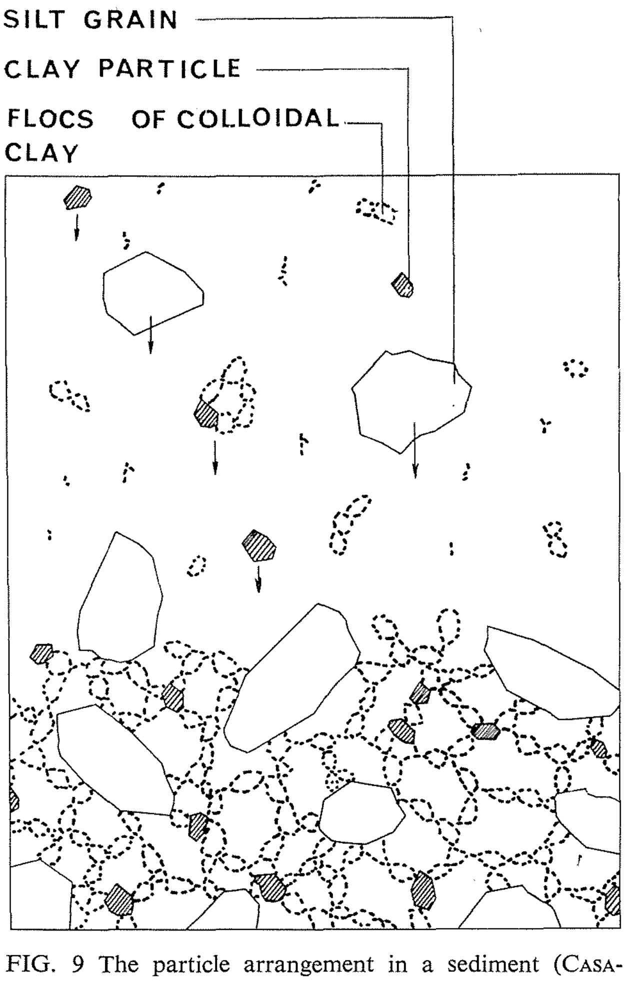


FIG. 9 The particle arrangement in a sediment (CASAGRANDE, 1940).

According to a similar hypothesis, suggested by TROLLOPE & CHAN (1960), sufficiently high shear stresses initiate a yield condition in the clay matrix and cause an orientation of colloidal particles in local regions. At failure there is a general particle orientation in the direction of the failure plane. In connection with this process, coarser grains migrate towards the failure zone resulting in additional yield strength of the matrix by intergranular friction. These authors also introduced the concept of stable units of soil structure, the "triangular" colloidal unit consisting of three particle members and the "diamond" colloidal unit consisting of four members. Theoretical studies of the rheological properties of clays by MURAYAMA & SHIBATA (1964) suggested the existence of similar units, a four-member structure with no relative sliding representing elastic joints and a structure of the triangular type with three mutually sliding members.

Recent investigations by ARNOLD (1967) showed, however, that the role of coarse grains in the clays may be of little importance only. He drew particle patterns (corresponding to hypothetical ultra-thin sections) by using grain-size distribution curves ranging down to colloidal dimensions, and found that in ordinary soft Swedish clays and even in boulder clay, fine silt and larger particles are so widely spaced that they have a negligible effect on the strength properties.

According to TERZAGHI & PECK (1948), the high sensitivity of soft clays may presuppose a well-developed skeleton structure. The sharply bent virgin compression curve of highly sensitive clays indicates a structural collapse when the preconsolidation pressure is exceeded (Fig. 10). Since highly sensitive clays are generally formed in brackish water or sea water, it indicates a relationship between (original) salt content and random, open structure. The fact that the water content is much higher than the liquid limit of many highly sensitive clays and that excess water is released by remoulding also indicates a very open type of structure.

The (shear) stress-strain relationship determined in controlled strain tests *in situ* has also been correlated with the microstructure. Tests performed by using a helical sounding borer driven by a boring machine (SÖKJER, 1961), illustrate a clear difference between sensitive inorganic clays and highly organic clays (muds). As is illustrated by Fig. 11, the maximum shear stress is developed at a large deformation in the organic clay indicating higher plastic deformability of the organic clay. This implies that the organic substance

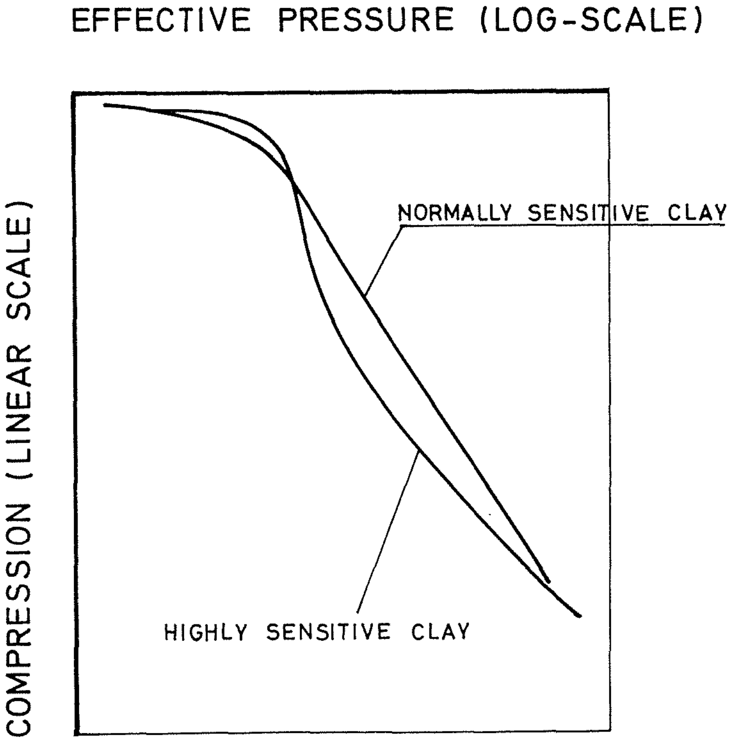


FIG. 10 Characteristic shape of compression curves for normally and highly sensitive clays.

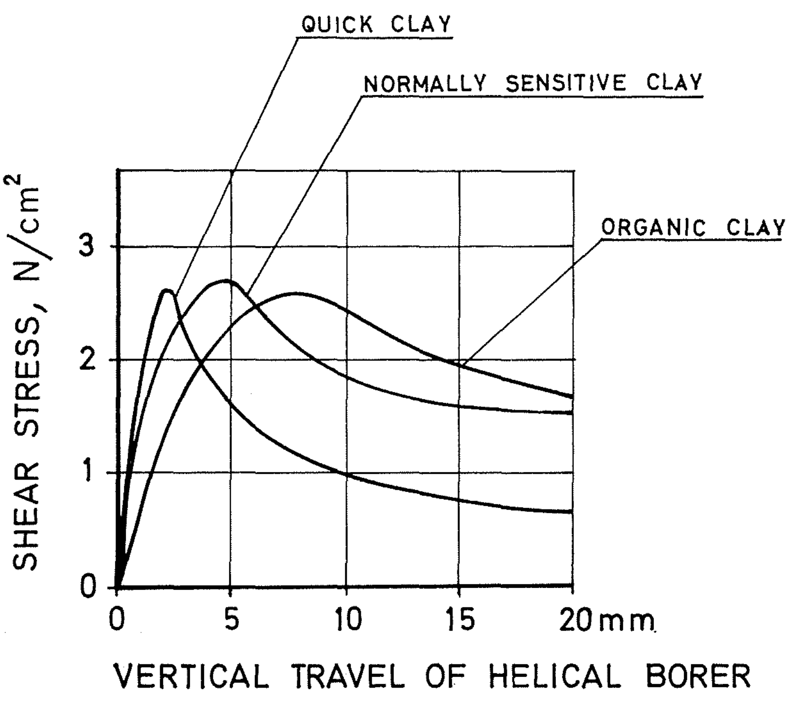


FIG. 11 Stress/strain relationship of different clay types according to A-sond tests (SÖKJER, 1961).

is an integrated member of the structural network. In the quick clay, the maximum shear stress rapidly decreased after exceeding the peak value, indicating the collapse of a very open structure.

Settling observations, shrinkage measurements and analyses of strength and deformation properties as well as of anisotropic physical properties such as permeability, electrical resistivity and thermal conductivity can only give a rough idea of the microstructural constitution. Yet, they may serve as standard tests for a simple microstructural classification.

2.5 Direct observation of structural details in light microscopes

The most important problem in microscopic studies of structural details is to find a preparation method which does not affect the particle arrangement. GOLDSCHMIDT (1935) suggested a technique based on drying and heating to about 500° C of the undisturbed clay sample. ROSENQVIST, being one of the pioneers within the field of structural investigations, realized that drying seriously affects the microstructure and introduced several suitable preparation methods which enabled him to draw important conclusions regarding clay structure (ROSENQVIST, 1955). By exchanging the pore water with alcohol, the alcohol with benzene (or toluene) and finally exchanging this with melted wax, the clay specimen consistency was found to be suitable for thin section preparation by using a microtome. Also various sulphonated alcohols, which are soluble in water, were found suitable for impregnation. After 14 days in a 60° C bath of such a substance (Jasperol), the clay samples were practically free from water and could be sectioned at —10°C.

By studying thin sections with a thickness of 2–10 μm, ROSENQVIST found that the smallest particles seen in the microscope were distributed in a haphazard way whereas flaky particles larger than about 10 μm were always parallel to the sea bottom. In Norwegian clays deposited in fresh water, most particles seemed to be placed nearly horizontally while the haphazard arrangement dominated the marine clays. Microphotographs of quick clays did not reveal any "honeycomb structure" or "card-house arrangement". Larger grains without mutual contact were found to be supported in a ground mass of finer particles and ROSENQVIST concluded that these coarser grains probably have no important influence upon the mechanical properties of the clays.

Within the field of pedology, a great many investigations concerning the microstructure of soils have been reported. In most cases, however, the investigated soil samples were taken from shallow layers, influenced by fertilizers and soil conditioners. Very seldom has complete information been given in such reports concerning the degree of water saturation, precautions to avoid disturbance at sample extraction, and exact depth position with special reference to the ground water level. However, certain investigations are of some interest in connection with the study of undisturbed fine-grained soils. A few reports will therefore be referred to in this chapter.

BREWER & HALDANE (1957) investigated the conditions for the development of parallel clay-size particles in the form of bands and coatings. Thin sections of drying mixtures of heavily flocculated clay-size and coarser material were obtained by grinding plastic treated soils with dry carborundum powder. It was concluded that the saturating cation had no effect on the degree of clay orientation, that clay flocculated in large pore space was only weakly oriented and that silt-size material had a disrupting effect on the orientation of clay-size material. An interesting result of the study of the drying mixtures is the gradual decrease in orientation of clay-size material as the proportion of such material increases. It was found that, up to 30 % clay content, the clay particles occurred as well-oriented coatings around coarser grains. Coatings and oriented bridges of clay between coarser grains were assumed to be caused by surface tension effects during drying. At higher clay content than 40 % only a small portion of the clay material occurred as oriented coatings. The clay fraction mainly consisted of illite (60 %) and kaolinite (30 %). Oriented clay material around coarser grains was further investigated by BREWER (1960), who introduced the concept of "cutan" for genetic pedological features of which "clay skins" is one of several types.

EMERSON (1959) suggested a probable structure of quartz-clay-organic material on the basis of microscopic investigations. He stated that the decrease in shear strength on remoulding is particularly obvious in organic Na soils, in which the remoulding both breaks the bonds between quartz, organic matter and clay and causes dispersion of the clay. The clay particles (illite or montmorillonite) form domains, defined by EMERSON as a group of clay crystals which are oriented and sufficiently close together to behave in water as a single unit. EMERSON used KUBIENA's thin section technique but impregnated with a cold-setting resin (KUBIENA, 1938; EMERSON & DETTMANN, 1959). KUBIENA started with dried soil samples and carried out the grinding by using carborundum powder and emery paper.

VAN DER WATT & BODMAN (1962), also dealing with organic contaminations (VAMA, vinyl acetate maleic anhydride, which was also the soil conditioner in some of EMERSON's studies) suggested that the particle linkage in H montmorillonite is of the edge-to-edge type through (lattice-)aluminum-carboxyl bonds while it is of the face-to-face domain-type in Al montmorillonite.

MCCRACKEN & WEED (1963) described micromorphological studies of various shallow soil layers (pan horizons) by thin-section technique with special reference to grain-to-grain relations. Undisturbed oriented blocks were collected from each site. In a layer with 6 % clay (kaolinite and mica type minerals) and 9 % silt, a random distribution of quartz sand with intergranular bridges of fine-grained material was found. A few groups of oriented clay were observed. In an organic layer with 4–7 % clay, the intergranular bridges of the fine-grained material had a random arrangement and seemed to block the pores.

SZABÓ, MARTON & PÁRTAI (1964) found photographic evidence for the interconnection of mineral particles by bridges formed by tightly adhering (organic) cells in mull-like forest soils. Fungi and actinomycetes formed a rich network among soil particles. The preparation of thin sections for examination under ordinary and polarized light was made by using an unsaturated polyester cast-resin (Polikon ES).

BREWER (1964) suggested a classification system of soil materials with morphological features, especially the extinction patterns observed in thin sections (25 μm) examined in polarized light. According to BREWER, preferred orientation in shallow soils is probably due to pressure and tension produced by wetting and drying. He also suggested that domains without any mutual preferred orientation may result from wetting and drying on a micro-scale within the soil mass or that they may be derived from older redeposited soil material. They may also be formed by weathering of mineral grains (MEYER & KALK, 1964). In this connection it should be mentioned that the microstructure of optically oriented clays is considered to be a very persistent feature, used as a pedogenetic indicator (MOROZOVA, 1964). This author also mentioned the existence of oriented clay caused by intrasoil weathering and argillisation in fossil soils.

An investigation by KORINA & FAUSTOVA (1964) of the microstructure of moraines (Würm and recent) revealed the existence of oriented clay. Detailed study of thin sections showed flow structures of oriented clay in pores and fissures and around grains. A perpendicular arrangement of large fibres with oriented clay particles between them was found to be a common feature. In moraines with a high content of sand and silt, the space between such grains often contained haphazardly arranged domains. Orientation of clay was observed around sand grains and around pores. The authors assumed that oriented clay in moraines was formed under ice pressure in the presence of water. The fibrous structure was supposed to be inherited from the structure of the moraine-carrying ice in which clay aggregates may have been distributed over the basal planes of ice crystals.

ALTEMÜLLER (1962, 1964) reported phase contrast investigations of thin soil sections. He concluded that in the marginal parts of sections of fine-grained material, a minimum thickness of 2–6 μm can be obtained if a polyester resin (Vestopal–H) is used as an embedding medium. The thickness will, however, be greater in those parts where coarser (and harder) grains are located. The sections were obtained by using diamond pastes in the grinding and polishing operations.

BECKMANN (1964) studied the frequency and shape of pores in thin sections of shallow soil samples, prepared by the method used by ALTEMÜLLER. GEYGER (1964) also used ALTEMÜLLER's method of preparation. He investigated the frequency of pores ranging from 5000 μm–20 μm using ODÉN's (1957) system of classification:

>200 μm	Coarse pore
200–20 μm	Medium pore
20– 2 μm	Fine pore
2–0,2 μm	Micro pore
<0.2 μm	Ultra pore

It should be mentioned that no detailed description of the preparation of thin sections has been given in the pedological articles referred to in the preceding text, except in the papers of KUBIENA, BREWER & HALDANE, EMERSON, ALTEMÜLLER and GEYGER. These authors used dried samples in the embedding procedure. Since drying changes the natural microstructure, their investigations have a restricted value as regards the concept of natural soil structure.

Experimental investigations of thin microtome-cut sections (8–10 μm) of fresh- and brackish water clays from Sweden, prepared according to MITCHELL, showed that these clays are characterized by aggregate formation (PUSCH, 1964). Typically, the clays contain pores and fissures of varying size.

Because of the limited resolving power of light microscopes, the great majority of the particles belonging to the clay fraction cannot be identified but the spatial arrangement and orientation of silt particles and larger microstructural units, such as clay particle aggregates, can be investigated. Hence, valuable information about clay microstructure may be obtained by using such instruments, provided that the original microstructure is not affected by the preparation technique. The interpretation of micrographs is generally complicated by the large thickness of the sections.

Aggregation patterns, internal flow structures (domains) and interconnecting organic substance are some of the reported microstructural features observed in ordinary microscopes.

2.6 Electron microscopic investigations

When electron microscopy was introduced into soil science, clay particle arrangements could be studied in detail for the first time. Electron microscopic studies of soil material sedimented from suspensions have given some indications of the main features of clay microstructure. Already the characteristic statistical distribution of the size of particles belonging to the clay fraction suggests some kind of structural feature of natural clays. Thus, the preferential attachment of small particles leading to big units and their tendency to be connected to larger particles in the course of sedimentation implies a microstructural order of the type seen in Fig. 12 (PUSCH, 1962).

FOLLETT *et al.* (1965) working with shallow layers of glacial till from Scotland, showed that hydrogen peroxide treatment and washing gave clumps of material. After treatment with cold and hot sodium carbonate solutions, the material was effectively dispersed and a great many small electron-dense granules appeared, probably iron compounds which had been contained in the aggregates. It was supposed by the authors that in natural soils there is always an aggregation of primary particles with coatings of poorly ordered alumino-silicates.

FIG. 12 Hypothetic floc or aggregate structure with reference to the characteristic variation of particle size in the clay fraction (PUSCH, 1962). The largest particle may be assumed to have a maximum diameter of the order of 0.05—0.1 μm.

Electron micrographs of dry gels of halloysite were taken as a proof of the card-house structure of thixotropic gels by HOFMANN (1952). According to HOFMANN, a disturbance of the card-house structure, "die Gerüste" (Fig. 13), causes complete dispersion. The thixotropic strength regain at rest was assumed to be caused by the formation of a new card-house. This structure, caused by a renewed sticking of the particles, would be built up during the time-dependent movement and diffusion of the solid particles. HOFMANN claimed that thixotropic behaviour is related to anisometric particle shape.

In 1947, results were published from an exploratory investigation of clay structure based on an electron microscopic study of fractured pieces of moist clay (HAST, 1947). HAST applied amyl acetate in which cellulose nitrate had been dissolved, to clay surfaces exposed by fracture. After the evaporation of the solvent, the thin film of cellulose nitrate, which formed a cast of the clay surface, was peeled off. Beryllium or

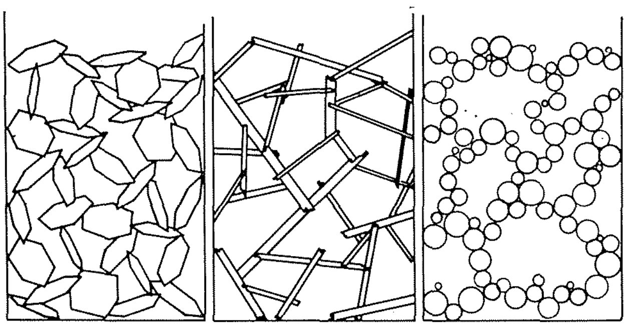


FIG. 13 Schematic illustration of particle arrangements in thixotropic gels at different particle shape according to HOFMANN (1952). a) Card-house of plates. b) Network of laths and tubes. c) Loose layering of spheres.

aluminum was applied under vacuum, the cellulose nitrate film was dissolved, and the remaining metal replica could be investigated in an electron microscope. The photographs obtained did, however, not reveal any fine details of the particle arrangement. Later, HAST applied the method to fractured pieces of freeze-dried clay samples.

During the last twenty years several investigations of clay structure based on transmission electron microscopy and replica technique, and using air-dried or freeze-dried samples have been carried out (WILLIAMS 1953, BATES & COMER 1955, BRINDLEY & COMER 1956, AYLMORE & QUIRK 1962, and others). Some of the best known reports were given by ROSENQVIST in 1958 and 1962. He stated, on the basis of a study of about a hundred stereoscopic micrographs of Scandinavian virgin marine clays, that the particle arrangements were of the "corner/plane cardhouse" type suggested by LAMBE and TAN. Domain structures were found only in dry crust clays, for which ROSENQVIST introduced the term "hypo-metamorphic". The specimens were prepared by the quick freezing of 2 cm thick plates immersed in liquid air. After drying at a pressure of about 10^{-3} mm Hg, the plates were broken mechanically and the fracture surfaces were covered with carbon while being rotated in an evaporator. On the carbon film a 0.1–0.2 μm thick aluminum film was evaporated and subsequently investigated in an electron microscope.

ROSCOE (1967) reported studies of fractured freeze-dried (and air-dried) kaolin clay specimens in which scanning microscopy was applied. Due to the low resolving power of such microscopes, the detailed arrangement of colloidal particles was not revealed.

Although the replica method applied to freeze-dried specimens makes possible a microstructural investigation, it has some great disadvantages which are also characteristic of scanning microscopy. Thus, these techniques only give a rough picture of the microstructure depending on the "topographic" variation of the fracture surface under investigation. Pore dimensions, for instance, cannot be adequately determined if the pores can be defined at all. There is also a serious disadvantage in using freeze-dried specimens although this preparation is superior to air-drying. Firstly, it is well known that clay specimens, independent of size, are cracked and therefore disturbed when immersed in low temperature media such as liquid air or nitrogen. Secondly, when the freeze-dried specimen is broken to reveal internal microstructural features, the exposed surface represents the failure zone caused by the breakage. Furthermore, ANDERSON & HOEKSTRA (1965) concluded from experiments with montmorillonitic clays that on lowering the temperature to less than $-5°$ C the clay lattices abruptly collapsed leading to significant particle movement and reorientation in freeze-thaw cycles. Similar changes in dense groups of particles in illitic clays may likewise occur.

A technique based on acrylate embedding and ultramicrotome sectioning has been applied by the author since 1963. Small specimens of undisturbed clay are treated with ethyl alcohol and butyl/methyl methacrylate (subsequently polymerized) by which the water is replaced by alcohol and the alcohol replaced by monomer through diffusion. The treated specimens are hard enough to permit sectioning in slices about 0.05 μm thick by means of a microtome equipped with a diamond knife (PUSCH, 1966a, 1968). A similar technique has been used by SCHELLMANN (1967) for an investigation of clay mineral ooides and by SMART (1967) for a study of remoulded kaolin clay.

The main advantage in studying such ultra-thin sections instead of the afore-mentioned thick sections is illustrated by Fig. 14, which shows a schematic microstructure. It is obvious that the very thin slice represents a "two-dimensional" section through the clay whereas a slice with a thickness of several microns may not reveal the majority of the smallest pores. Besides, such thick sections are not sufficiently penetrable by the electron radiation in ordinary electron microscopes.

The method of investigating ultra-thin sections in a transmission electron microscope has proved to be very useful since detailed microstructural features are preserved and can be revealed and defined so that a statistical treatment is possible.

Due to the high resolving power, electron microscopy is a suitable technique for the identification of the detailed arrangement of clay size particles. Various electron microscopic methods have been applied in the study of clay microstructure but the natural particle arrangement has been found to be seriously affected by certain procedures of specimen preparation. The technique based on acrylate embedding and ultra-microtome sectioning seems to be most suitable and it has been chosen for the author's tests which are reported in the subsequent text.

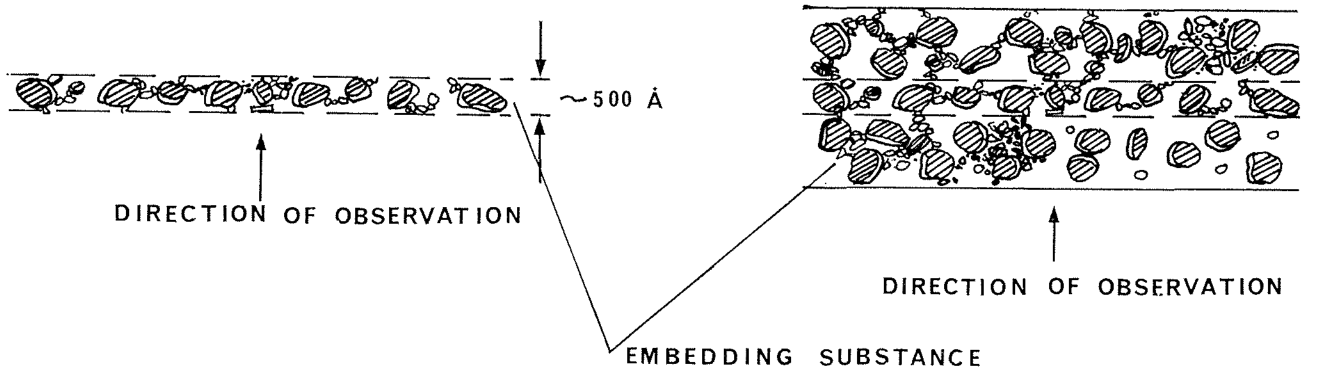


FIG. 14 Influence of section thickness on the microscopical study of thin sections.

TEST REPORT

3 INTRODUCTION

Three Quaternary clays were investigated with reference to the microstructure and to some of their important physical properties. The techniques of ultra-thin sectioning of plastic-treated clay and transmission electron microscopy were applied. The three clay types represent different geographical areas and geological ages as well as different problems in soil mechanics and foundation engineering.

3.1 Clay types

a) Skå-Edeby. Location on the island Svartsjölandet about 25 km west of Stockholm. The exact situation and the topographic characteristics of this research field have been reported by HANSBO (1960).

According to an investigation made by JÄRNEFORS (1956, 1957), the sediments, which have a total thickness of 10–15 m, were formed during Quaternary glacial and post-glacial times. The glacial as well as the post-glacial sediments were deposited in stagnant, oxygen-poor water. Thus, the clays are sulphide-rich and contain layers and lenses of iron sulphide throughout the whole profile. The post-glacial clay forms the upper 5 m layer at the test site. The ground water level is situated 0.5–0.8 m below the ground surface. The basal part of the glacial clay was deposited from about 7900 B.C. according to JÄRNEFORS. The fine-grained dark-grey clay layer which was found at about 5 m depth was probably formed in the Ancylus Lake, a fresh-water stage in the Baltic.

The salinity of the water during the formation of the post-glacial sediments was estimated on the basis of diatom analyses. This investigation showed that marine, marine-brackish and brackish-marine specimens dominated in the sample from 2 m depth. The estimated original salt content was 10–20‰. The abundance of *Nitzschia punctata* indicates that the water depth was small. Diatom analyses of samples from 5 and 8 m depth were not successful due to the small number of specimens, but the distinct varves of the glacial clay indicate that the original salt content was very low.

The present salinity—as concluded from electrical resistivity measurements of expelled pore water—was equal to or less than 5‰ for the samples from 2 and 5 m depth, which means that leakage has taken place. For samples from 7, 8, and 9 m depth the salinity was of the order of 5‰ which is probably higher than the original value. The major cations in expelled pore water from the lower part of the post-glacial clay layer, which was poor in electrolytes, were Fe (60 mg/l), Na (60 mg/l), K (15 mg/l), and minor amounts of Ca and Mg, as determined by a spectrophotometric technique. The analysis of the glacial clay showed that Na was most abundant and that considerable amounts of Mg, K and Ca were present as well, the electrolyte content probably being changed from the time of deposition.

Samples from 2, 5, 7, 8, 9, and 10 m were used for microstructural and geotechnical investigations. Only the fine-grained varves in the glacial clay samples were investigated.

b) Lilla Edet. Location near the Inland Paper Mill in the Göta River Valley, about 45 km north of Gothenburg (Fig. 15). According to investigations made by the Geological Survey of Sweden (SGU) the sediments, which have a total thickness of more than 20 m, were formed during Quaternary late-glacial and post-glacial times. Generally, the late-glacial clays are fine-grained and rich in iron sulphide. The sulphide occurs in distinct, 1–3 cm thick dark-grey layers similar to cyclic varves. Diatom analyses have shown that the late-glacial and post-glacial clays were mainly formed in marine environments. According to these earlier investigations by SGU, the late-glacial clay (Senglacial II) which forms the upper 7 m clay layer at SGU VII: 1 and the clay below 16 m depth at SGU VII: 2 were deposited under cold and temperate marine conditions while the post-glacial

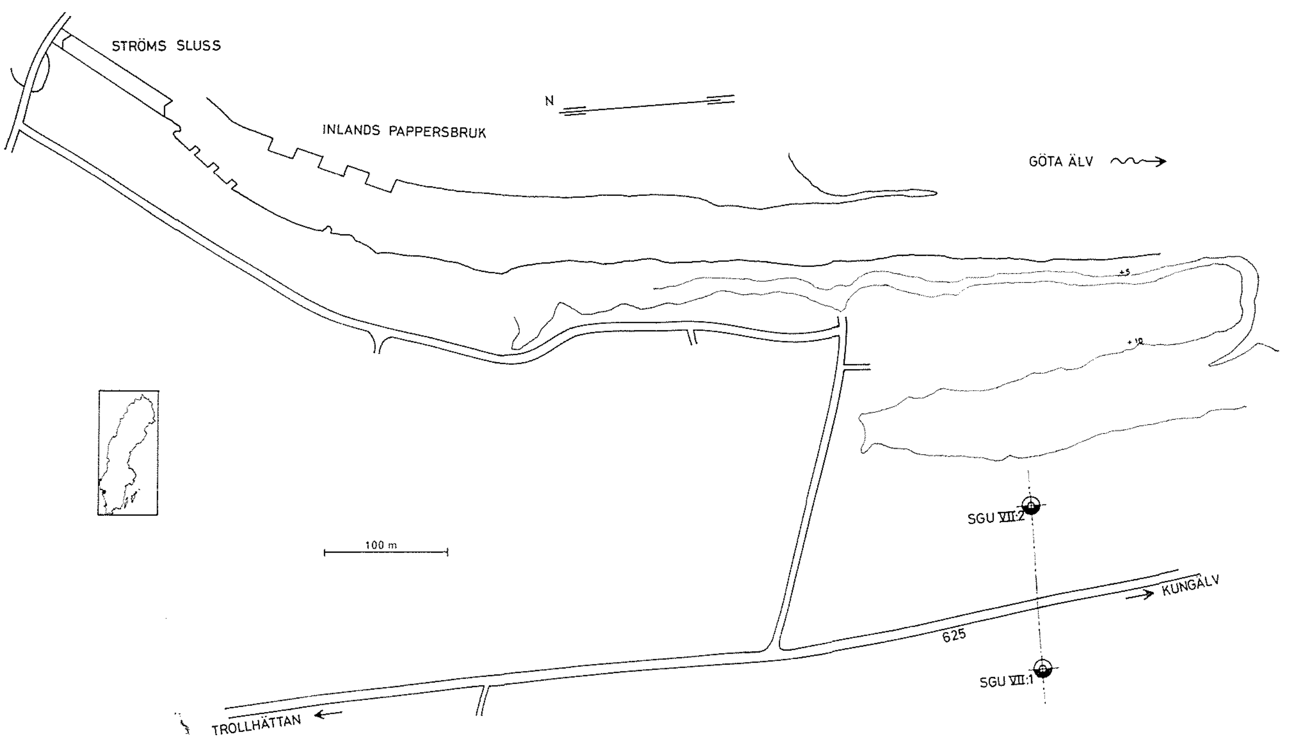


FIG. 15 Location of bore holes in Lilla Edet.

clays were formed under temperate marine conditions. No unanimous statement has been obtained concerning the age of the Lilla Edet sediments.

The groundwater level is situated 1.0–1.5 m below the ground surface. The sediments have been leached and according to investigations by the SGU the present salinity is equal to or less than about 5 ‰. A recent analysis of the cation content gave a strongly varying ionic composition. In the quick clay layers, where the lowest salt content values were found, the major cations were Na (90–330 mg/l), K (30–45 mg/l), Mg 20—70 mg/l), and Ca (5–140 mg/l).

It should be mentioned that the site in Lilla Edet was chosen for the author's investigation because of the occurrence of a quick-clay zone in the late-glacial clay in borehole SGU VII: 1. The same layer of late-glacial clay in SGU VII: 2 was not found to be quick. Samples from 3 and 6 m depth at SGU VII: 1 and from 3 and 19 m depth at SGU VII: 2 were used for microstructural and geotechnical investigations.

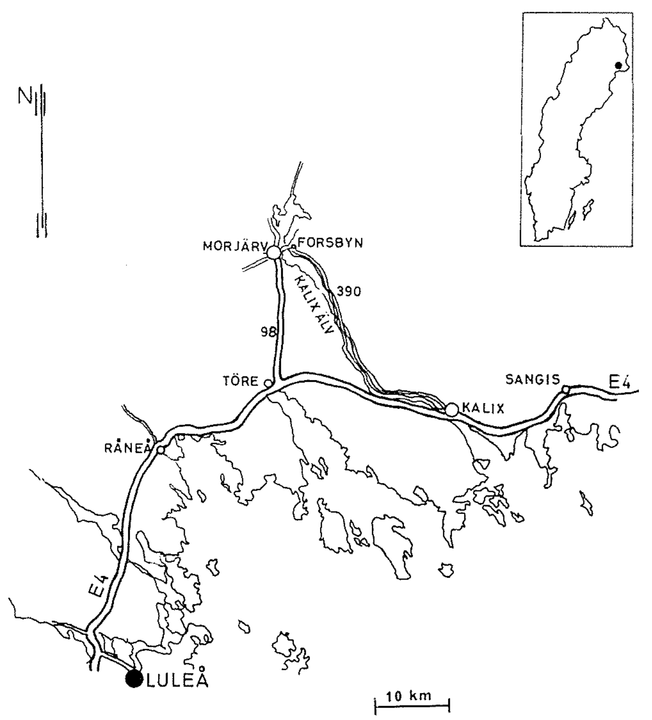


FIG. 16 Location of Road 390 (Morjärv—Kalix).

c) Morjärv. Location: Road 390, Section 17480, Forsbyn–Morjärv, about 50 km north-east of Luleå (Fig. 16).

The sediment consists of an almost 10 m thick layer of homogeneous black or greyish black clay or silt ("svartmocka"). The groundwater level is situated about 1 m below the ground surface. According to FROMM (1965) this type of organic sediment was formed during the Litorina time.

The salinity of the water during the formation of the Morjärv sediments was estimated by means of diatom analyses, which showed that marine, marine-brackish, and brackish-marine specimens were dominant. The estimated original salt content was 5-20‰ for samples from 6 m depth and 5-15‰ for samples from 4 and 9 m depth. The high content of specimens like *Coscinodiscus lacustris*, *Achnanthes hauckiana*, *Melosira monoliformis* and *Melosira nummuloides* indicates that the sediments were formed in or close to a river mouth at a shallow depth.

The present salinity was found to be equal to or less than about 5 ‰. The major cations in the profile were Na (240–490 mg/l), Ca (120–230 mg/l), Mg (110–230 mg/l), Mn (80–130 mg/l), K (60–100 mg/l), and Fe (25–110 mg/l).

Samples from 4, 6, and 9 m depth were used for microstructural and geotechnical investigations.

Summing up, the clays from Morjärv and from 2 m depth in Skå-Edeby are organic brackish (brackish-marine) deposits. The clay from 5 m depth in Skå-Edeby is a fresh-water deposit, relatively poor in organic substances, while the remaining Skå-Edeby profile represents somewhat organic, fresh-water (possibly brackish) deposits. The Lilla Edet clays are marine deposits, relatively poor in organic substance.

The extraction of all samples was made by using the Swedish 50 mm standard piston sampler. The laboratory investigations of the samples were made within one to two weeks after extraction.

The determination and interpretation of the diatom content were made by the Geological Survey of Sweden, under the supervision of Dr. U. Miller. The chemical analyses were carried out at the Swedish Geotechnical Institute and at the Department of Geology, Chalmers University of Technology.

3.2 Grain size distribution

The grain-size distributions were determined by sedimentation analyses according to the pipette method (AKROYD, 1957). The following preparation method was used:

1. 100 g of the natural material was dispersed in a solution of 20 ml hydrogen peroxide in 100 ml distilled water.

2. The sample was stored for one hour in an oven at 110° C.

3. After one hour at room temperature the sample was washed by centrifugation (20 minutes at 2000 r.p.m.). This process was repeated four times.

4. The washed sample was dispersed by 30 minutes treatment in an 80 kc/s 120 W ultrasonic apparatus.

5. The dispersed sample was transferred to the sedimentation cylinder. Sodium pyrophosphate was added to 0.005 M solution.

The results of the grain-size investigation are given in Table 1.

The size distribution of the clay size particles was investigated by electron microscopy. Droplets taken from the suspension containing particles with an equivalent diameter less than about 5 μm were transferred to 400 mesh copper grids covered with a carbon film prepared in a Hitachi evaporator. The specimens were shadowed by gold coating at an angle of about 25°.

A series of micrographs was taken of each sample with a Zeiss EM 9 electron microscope with a 25 μm aperture in the objective lens. The film used was a Gevaert Scientia Film 23 D 46, 70×70 mm. The electronic magnification of the micrographs, which were taken at random, was ×7000. A linear optical magnification of ×2 was also used.

The size parameter (maximum particle diameter) a and the shape parameter (ratio of maximum particle diameter and particle thickness) a/c were determined by direct measurement using paper copies of the micrographs (PUSCH, 1962, 1966b). All particles with a distinct outline and metal "shadow", which were present within a representative area of each micrograph, were measured.

Median and quartile values are given in Table 2, which also gives the 10th percentile values of a illustrating the content of "colloidal" particles. Fig. 17 shows a representative micrograph and histograms of one of the clays.

TABLE 1. Grain-size distribution in percent in terms of equivalent diameter.

Site	Depth m	Weight percentage of material finer than 0.04 mm	0.02 mm	0.01 mm	0.006 mm	0.002 mm
Skå-Edeby	2.0	88	81	75	70	59
	5.0	82	80	79	79	77
	7.0	92	86	79	73	52
	8.0	99	98	92	86	63
	9.0	96	90	83	78	56
	10.0	95	91	84	79	54
Lilla Edet	3.0	99	99	90	88	65–70
	6.0	99	99	96	90	74
	19.0	99	96	90	83	55
Morjärv	4.0	90	78	65	52	20
	6.0	86	70	52	41	19
	9.0	90	85	75	68	37

TABLE 2. Median values, quartiles and percentiles of *a* and *a*/*c* for clay particles.

Site	Depth m	*a* in μm M[1]	Q_1[2]	Q_3[3]	P_{10}[4]	S_K[5]	log S_0[6]	*a*/*c* M	Q_1	Q_3
Skå-Edeby	2.0	0.052	0.036	0.130	0.025	1.73	0.279	4.6	3.0	7.0
	5.0	0.037	0.026	0.083	0.021	1.58	0.252	4.0	2.9	5.9
	7.0	0.100	0.060	0.150	0.037	0.90	0.199	5.8	3.7	8.4
	8.0	0.099	0.060	0.184	0.043	1.13	0.243	7.6	5.1	12.0
	9.0	0.117	0.075	0.184	0.045	1.01	0.194	6.6	4.5	10.0
Lilla Edet	3.0–6.0	0.018	0.014	0.025	0.012	1.08	0.126	2.7	1.8	3.3
	19.0	0.017	0.013	0.022	0.010	0.99	0.114	1.8	1.5	2.3
Morjärv	4.0–9.0	0.045	0.027	0.083	0.019	1.11	0.242	4.6	2.8	7.8

1 Median value.
2 Lower quartile.
3 Upper quartile.
4 10th percentile (finest part of the cumulative curve).
5 Skewness (TRASK, 1932), $(Q_1 \cdot Q_3)/M^2$.
6 S_0=sorting coefficient $\sqrt{Q_3/Q_1}$; log S_0-values are directly comparable.

The clay particle analysis showed that the post-glacial clay at Skå-Edeby is more fine-grained than the glacial clay, which is explained by the fact that the post-glacial clay is originally glacial clay which has been eroded and redeposited. The stratum at 5 m depth, which is especially rich in clay, is the most fine-grained part of the Skå-Edeby profile.

The *a*-values of the Skå-Edeby and Morjärv clay fractions are of the same order, while the Lilla Edet clay is much more fine-grained. The content of colloidal particles (showing electron diffraction patterns) is also highest in the Lilla Edet clay samples as shown by the percentile values and by the skewness values. This means that a large number of extremely small particles were present in the settling clay suspensions during the formation of the Lilla Edet deposit. The microstructural investigation, which is presented in the subsequent text, showed that a considerable number of such particles exist as linking units in the microstructural network. Hence, they were not formed after deposition and they are not a product of the preparation of the clay suspensions. The log S_0-values illustrate that the Lilla Edet clay fraction is well sorted.

As in previously investigated clays of similar origin the *a*/*c*-value was found to be a function of the *a*-value (PUSCH, 1962). Thus, only the largest particles in the clay fraction have *a*/*c*-values exceeding 20–50. The majority of the smallest particles have an almost equidimensional shape.

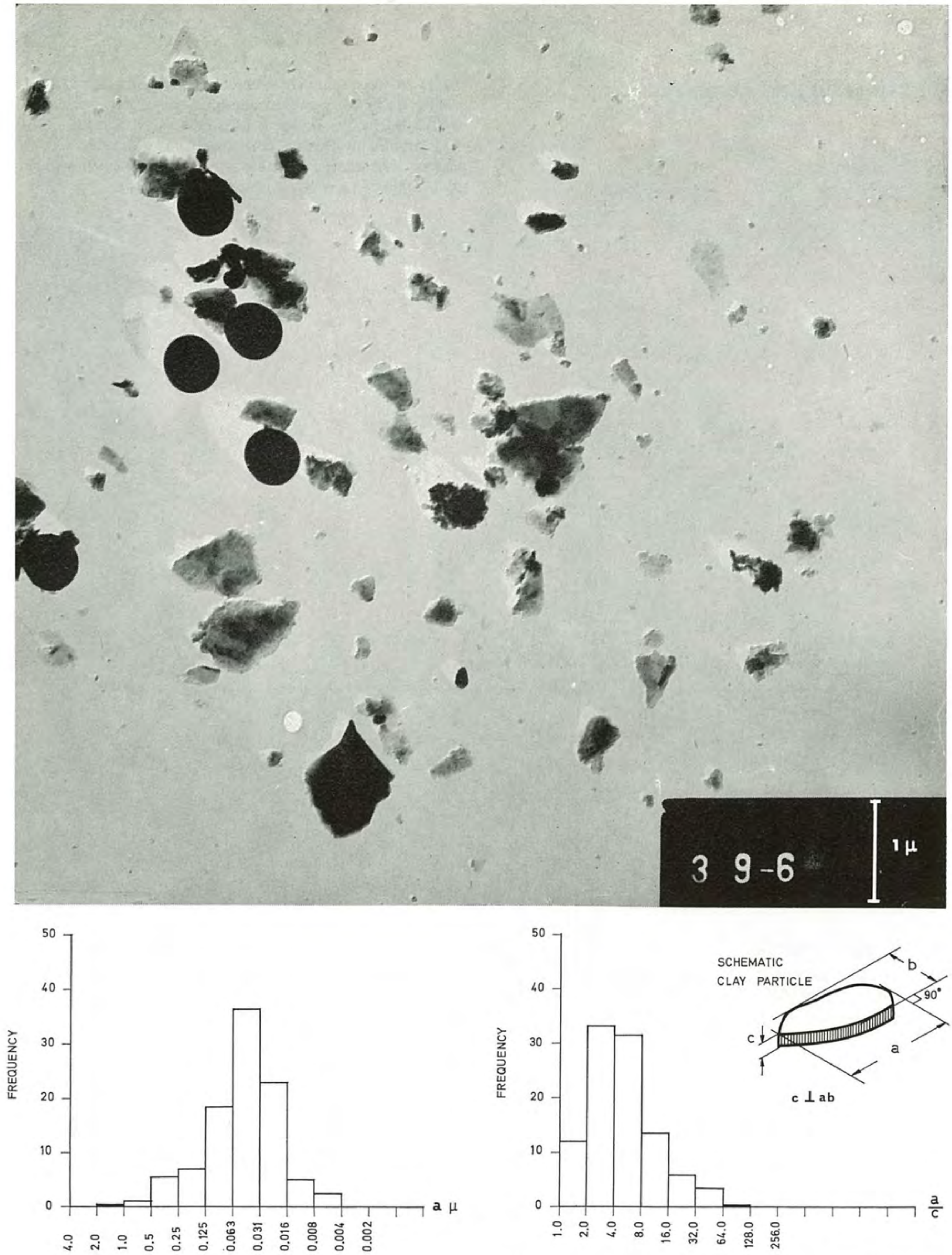


FIG. 17 Micrograph of ultrasonically dispersed Morjärv clay material from 6 m depth. The dark rounded bodies in the micrograph are latex particles used for checking the magnification. Histograms of particle size and shape parameters.

3.3 Mineralogical composition

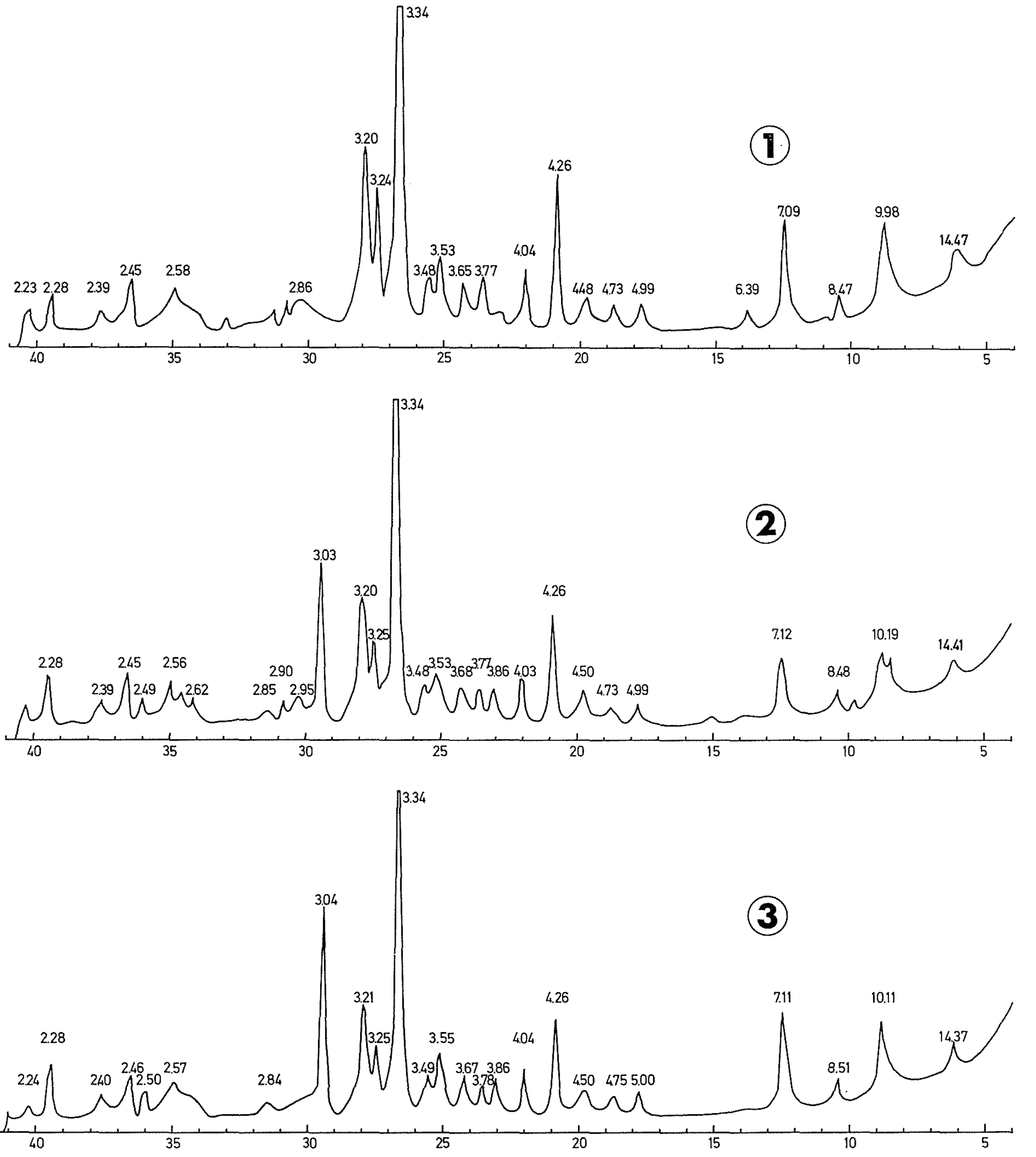
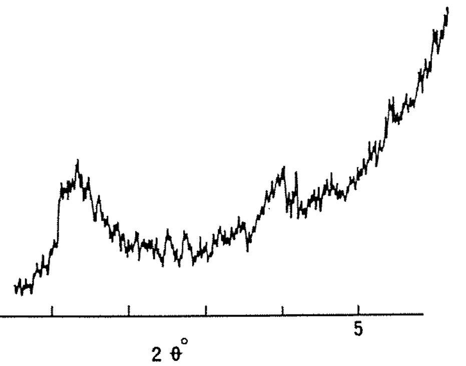


FIG. 18 Representative diffractometer records showing d-values. 1) Skå-Edeby 2 m depth. 2) Skå-Edeby 5 m depth. 3) Skå-Edeby 8 m depth. 4) Lilla Edet, representative of the soil profiles at SGU VII: 1 and :2. The detailed picture shows a characteristic 0.001 μm peak of a quick sample. 5) Morjärv 4 m depth. 6) Morjärv 9 m depth.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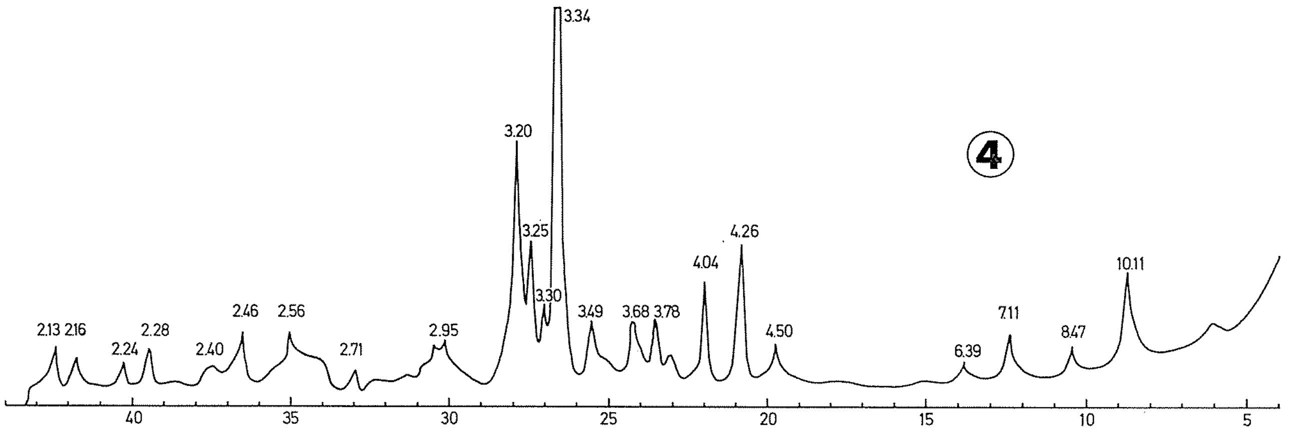
5
2 θ°


3.34
3.20
4
3.25
4.26
4.04
10.11
3.30
2.13 2.16
2.46
2.56
3.49
3.68 3.78
7.11
2.24
2.28
2.40
2.71
2.95
4.50
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6.39
40
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30
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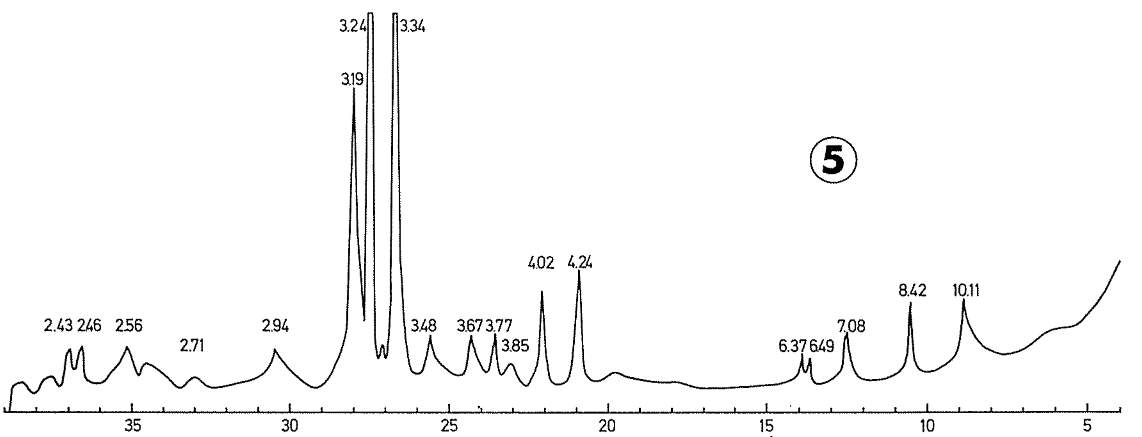
10
5


3.24
3.34
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4.02
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10.11
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2.56
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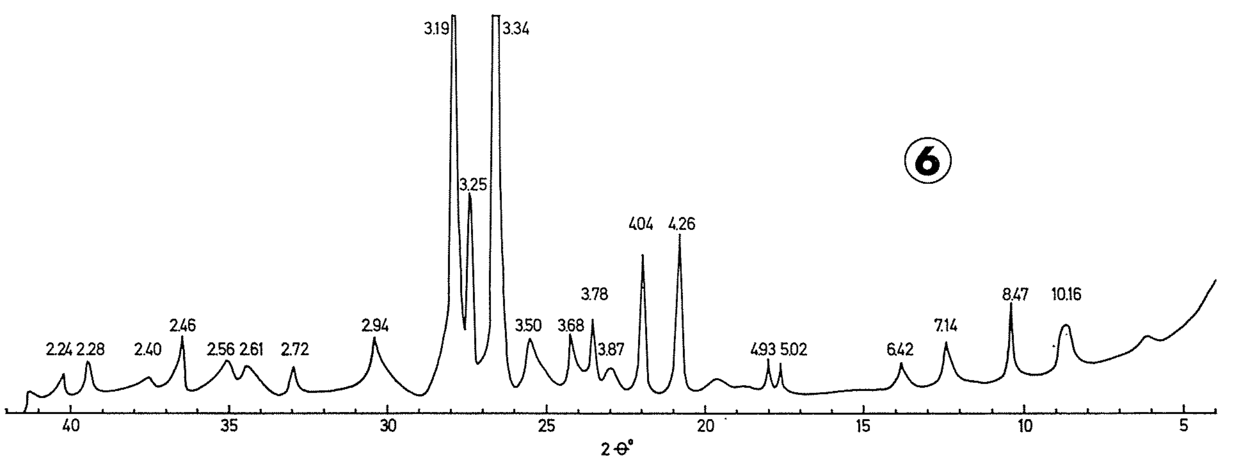
3.19
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2.40
2.56 2.61
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3.87
4.93 5.02
6.42
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2 θ°

X-ray diffraction technique and determination of the cation exchange capacity (C.E.C.) have been used to get a general idea of the mineralogical compositions. The preparation of samples for X-ray investigations was made according to the following method:

1. 10 g of clay with natural water content was immersed in a solution of 5 ml hydrogen peroxide in 100 ml distilled water. The suspension was dispersed ultrasonically.

2. Sedimentation for 24 hours. Decantation and addition of 200 ml diammonium oxalate. Ultrasonic treatment for 10 minutes.

3. Sedimentation for 48 hours. Decantation and addition of 300 ml distilled water and of hydrochloric acid to pH=3.

4. Sedimentation and drying at 105° C.

5. Grinding in an agate mortar.

The X-ray investigation was made with a 40 kV Philips diffractometer. The goniometer radius was 170 mm and the scanning speed ½–1° (2 Θ) per minute. CuK_α radiation and a Ni filter were used. Fig. 18 shows some representative diffractograms. Specimens with well-oriented particles as well as with fairly randomly arranged particles were investigated. Dried specimens as well as specimens treated with hydrochloric acid or glycol were used, the maximum equivalent particle diameter being about 10 μm.

The Skå-Edeby clay samples from 2 m depth contained quartz, feldspars, illite, kaolinite, chlorite, hornblende, and rock-forming micas. The three first-mentioned minerals, which formed about 75 % of the total amount, were present in fairly equal amounts. The main minerals in the samples from 5 m depth were carbonate minerals (mainly calcite), illite, quartz, kaolinite, and feldspars, the three first-mentioned minerals being dominant. The irregular, strongly asymmetric 0.001 μm[1]) peak was taken as an indication of slight interlayer hydration of the illite. By glycol treatment very weak peak shifts toward the 0.0017 μm region occurred, indicating the presence of swelling minerals. Hydrochloric acid treatment and heating to 600° C showed that a small amount of chlorite was present. The high area/height ratio of the 0.0007 μm peak was taken as indicative of poorly crystallized kaolinite or small particle size. The samples from 7—10 m depth had a mineral composition which was similar to that of the 5 m layer, but calcite and chlorite were more abundant. Separate studies of the clay fraction showed that illite formed more than 50 % of the total amount throughout the profile.

The Lilla Edet profiles, being similar, are represented by a diffractogram of the normally sensitive clay from 19 m depth in Fig. 18. The investigated sediments were found to be rich in rock flour, especially feldspars. The main minerals were feldspars, quartz, illite, kaolinite, hornblende and rock-forming micas. For samples from shallow depths, generally representing quick clay, the 0.001 μm peak was very broad and asymmetric toward the low angle region, which indicates interlayer hydration. Illite was the dominant clay mineral in the clay fraction which also contained considerable amounts of quartz and feldspars.

The Morjärv samples contained a high amount of feldspars, quartz, and hornblende. Illite, kaolinite, and rock-forming micas were moderately abundant. In the clay fraction the content of illite was roughly estimated to at least 50 % of the total amount. The broad, in some tests strongly asymmetric 0.001 μm peak was taken as an indication of slight interlayer hydration of the illite and not as a consequence of small particle size. This is because the average particle size *a*, according to the microscopic investigations, is larger for the

[1]) The crystallographic *d*-values are given in μm units instead of the conventional Å units, following the general recommendations given by the Swedish Standards Institution (1 μm = 10^4 Å).

TABLE 3. Cation exchange capacity (C. E. C.).

Site	Depth m	C.E.C. meq (100 g)$^{-1}$	Remarks
Skå-Edeby	2	39	
	2	43	Diammonium oxalate treated
	5	35	
	5	33	Diammonium oxalate treated
	8	39	
Lilla Edet	3	29	
	6	25	
Morjärv	4	40	
	6	40	
	6	40	Diammonium oxalate treated
	9	41	
	9	34	Diammonium oxalate treated

Morjärv clay than for the Lilla Edet material which showed fairly symmetric 0.001 μm peaks for certain samples.

C.E.C.-values were determined by a method recommended by ROSENQVIST (1962 *b*). This method is based on saturating the soil sample with sodium acetate after hydrogen peroxide treatment, followed by exchanging calcium for sodium by calcium chloride treatment and by the exchange of sodium for calcium at pH=7. The calcium concentration in the leachate was then determined by titration. In the case of clays rich in iron sulphide, parallel tests were run on samples treated with diammonium oxalate to dissolve cementing iron compounds. The C.E.C.-values for the clay fraction were all within the interval 25–43 meq $(100\ g)^{-1}$ which supports the conclusion from the X-ray investigations that illite was the dominant clay mineral (Table 3). This is in reasonable agreement with the activity (Table 4) except for the Morjärv samples from 4 and 6 m depth. These samples had a_c-values higher than 3 which indicates a high content of swelling minerals according to SKEMPTON (1953). This is definitely contradicted by the X-ray and C.E.C. investigations.

4 GEOTECHNICAL PROPERTIES

4.1 Test programme

The investigation of the mechanical properties of the clays included determination of the preconsolidation pressure, compressibility and consolidation coefficient from oedometer tests. Also, the stress/strain behaviour in unconfined compression tests was observed and finally the shear strength and sensitivity were determined by using the Swedish fall-cone test. The most important geotechnical data are collected in Table 4.

Oedometer tests on trimmed specimens with 30 mm diameter and 15 mm height were made with load application perpendicular to (vertical) and parallel to (horizontal) the sedimentation plane. Tests on specimens with 50 mm diameter and 20 mm height were also made.

4.2 Preconsolidation pressure and compressibility

The preconsolidation pressure was evaluated by applying CASAGRANDE's method. The compressibility (compression index ε_2) was expressed and calculated as the deformation along the virgin curve due to double pressure. There was a negligible difference between the preconsolidation pressures at vertical and horizontal compression of the Skå-Edeby samples (Fig. 19 a). Also, the compression index values were almost the same. It should be noticed that the curves indicate a slight disturbance, especially the ones for horizontal compression. For the late-glacial Lilla Edet clay from 6 m depth, there was practically no difference between the preconsolidation pressures at vertical and horizontal compression. The tests of Lilla Edet clay from 19 m depth indicated that the compression index for horizontal compression was only about 80 % of the

TABLE 4. Geotechnical data for the investigated clays.

Site	Depth m	γ g/cm³	Shear strength Unc. compr. test N/cm²	Shear strength Cone test N/cm²	S_t (cone test)	w %	w_L %	w_P %	a_c	Ignition loss %	Description
Skå-Edeby	2.0	1.43	1.7	1.5	9	106	120	40	1.36	7.5	Green-grey muddy clay
	5.0	1.49	1.0	0.9	11	105	98	29	0.90	4.6	Grey clay
	7.0	1.59	1.1	1.1	21	73	58	22	0.69	5.5	Brown-grey varved clay
	8.0	1.61	1.0	1.0	18	70	56	22	0.54	5.6	Brown-grey varved clay
	9.0	1.62	1.2	1.2	20	67	55	22	0.59	5.1	Brown-grey varved clay
	10.0	1.61	1.0	1.1	16	65	52	22	0.56	5.1	Brown-grey varved clay
Lilla Edet	3.0[1]	1.48	1.3	1.6	60	97	68	30	0.58	3.5	Grey clay
	3.0[2]	1.48	1.0	1.2	42	97	73	30	0.61	3.4	Grey clay
	6.0[1]	1.50	1.7	1.6	147	91	58	29	0.39	3.0	Grey clay
	19.0[2]	1.64	2.6	2.4	14	58	54	23	0.56	4.0	Dark-grey clay
Morjärv	4.0	1.25	1.0	1.3	26	198	157	79	3.90	8.8	Black muddy clay
	6.0	1.39	1.2	1.3	27	128	113	52	3.21	5.3	Black muddy clay
	9.0	1.43	1.5	1.3	27	100	92	42	1.35	5.0	Black muddy clay

[1] Bore hole close to SGU VII: 1.
[2] Bore hole close to SGU VII: 2.

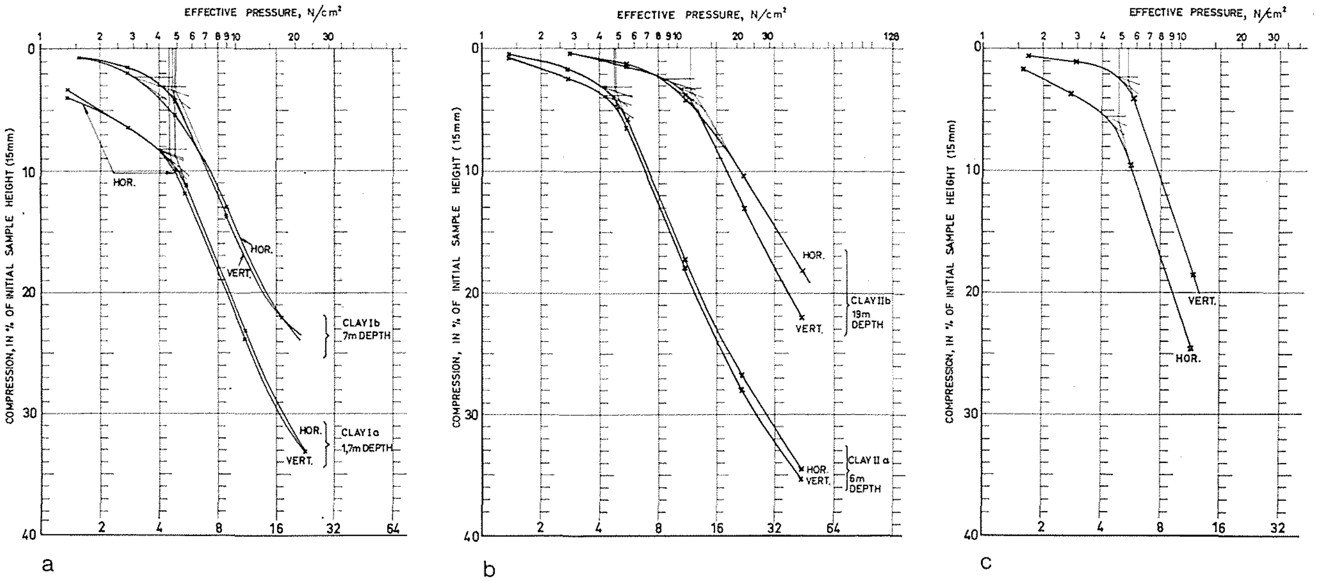


FIG. 19 Compression diagrams for a) Skå-Edeby clay (1.7 m—also representative of samples from 2 m depth—and 7 m depth), b) Lilla Edet clay (6 and 19 m depth), c) Morjärv clay (9 m depth). The preconsolidation pressures are marked in the diagrams.

index value for the perpendicular direction, whereas the preconsolidation pressures were of the same order (Fig. 19 b). Also in this case the curves indicate a slight disturbance which may explain, at least to some degree, the difference between the compression index values.

The Morjärv clay samples from 4 and 6 m depth had anisotropic compression properties but almost the same preconsolidation pressure at vertical and horizontal compression. The compression index values of samples from 9 m depth were the same for vertical and horizontal compression, whereas the preconsolidation pressure in the horizontal direction was only about 90 % of the corresponding pressure in the vertical direction (Fig. 19 c). As in the previous cases, the curve for horizontal compression indicates a slight disturbance. Thus the real difference between the preconsolidation pressures may be less than is indicated by the determined values.

The evaluated compression and consolidation data are collected in Table 5.

4.3 Consolidation properties

The coefficient of consolidation was calculated on the basis of the time value for 50 % consolidation using TAYLOR's method of graphical plotting. Where the specimens were compressed vertically the coefficients (c_{v_v}) were generally smaller than those of the horizontally compressed specimens (c_{v_h}). The c_{v_v}-values of the Skå-Edeby clay, which were of the same order as those reported previously by HANSBO (1960), correspond to 10–50 % of the c_{v_v}-values of the Lilla Edet and Morjärv clays, which indicates a slower consolidation rate for the Skå-Edeby clay.

The consolidation coefficient was also used for estimation of the coefficient of permeability using Eq. (1):

$$k = c_v \cdot m_v \cdot \gamma_w \qquad (1)$$

where k = DARCY's coefficient of permeability
m_v = coefficient of volume compressibility
γ_w = density of pore water

TABLE 5. Compression data.

Site	Depth m	Diam. mm	$\varepsilon_{2vert.}$ %	$\varepsilon_{2hor.}$ %	$\sigma_{0\,vert.}$ [1] N/cm²	$\sigma_{0\,hor.}$ [2] N/cm²	σ_0 [3] N/cm²	$c_{v_v} \cdot 10^4$ [4] cm²/sec	$c_{v_h} \cdot 10^4$ [5] cm²/sec	$k_v \cdot 10^8$ [6] cm/sec	$k_h \cdot 10^8$ [7] cm/sec
Skå-Edeby	2.0	30	12	12	4.5	4.8	1.5	0.7	0.5	7.9	5.3
	5.0	50	12	—	3.3–3.9	—	3.2	0.55–0.90	—	7.8–13.0	—
	7.0	30	11–12	11	4.7	4.9	4.2	0.95	1.6	9.1	15.0
	7.0	50	10–12	—	4.0–5.3	—	4.2	0.22–1.15	—	1.9–9.7	—
	8.0	50	9–12	—	3.2–5.1	—	4.8	0.37–0.72	—	3.3–6.6	—
	9.0	50	11	—	5.2	—	5.6	1.2	—	10.0	—
	10.0	50	10	—	5.6	—	6.2	1.1	—	8.5	—
Lilla Edet	3.0	30	13–14	—	7.8	—	2.8	5.9	—	53.0	—
	6.0	30	11–12	11–12	4.7	4.8	3.8	3.7	3.8	32.0	32.0
	19.0	30	10	8	11.8	11.8	14.2	4.0	11.0	7.5	40.0
Morjärv	4.0	30	12	10	2.5	2.2	2.1	4.0	3.5	91.0	90.0
	6.0	30	12–13	10	3.8	3.4	2.8	2.0	5.7	28.0	87.0
	9.0	30	14–15	15	5.3	4.8	4.0	2.0	2.5	19.0	30.0

[1] Preconsolidation pressure in vertical direction acc. to oedom. tests.
[2] Preconsolidation pressure in horizontal direction acc. to oedom. tests.
[3] Existing overburden pressure.
[4] Consolidation coefficient calculated from vertically loaded specimen.
[5] Consolidation coefficient calculated from horizontally loaded specimen.
[6] Coefficient of permeability of vertically loaded specimen.
[7] Coefficient of permeability of horizontally loaded specimen.

The coefficient of permeability was found to be much smaller for the Skå-Edeby specimens (generally less than 10^{-7} cm/sec) than for the other clays investigated. The average value for vertical pore water flow was $3.1 \cdot 10^{-7}$ cm/sec for the Lilla Edet clay and $4.6 \cdot 10^{-7}$ cm/sec for the Morjärv clay. In the two last-mentioned soil profiles the permeability decreased with increasing depth. The permeability for horizontal flow was of the same order as for vertical flow in certain samples from Skå-Edeby and Lilla Edet, but in the majority of the samples the permeability for horizontal flow was higher (Table 5). TERZAGHI's theory of consolidation, which formed the basis of the calculation of the c_v-values, may not give the true time dependence of the pore water dissipation in certain cases (HANSBO, 1960). Therefore, the calculated k-values may be somewhat uncertain. However, for the Skå-Edeby material where deviations from theory should be most obvious, the k-values were found to be of the same order as the values from precision permeability tests reported by HANSBO (1960).

4.4 Axial strain at unconfined compression

Unconfined compression tests of certain samples were made by using an automatically recording apparatus (cf. KALLSTENIUS, 1963). The pressure was increased at a constant rate of 0.3 N/cm²/minute. Due to the design of the apparatus, which automatically records the compression, the curves formed circular arcs after the failure conditions had been reached (Fig. 20). The Skå-Edeby samples from 2 m depth were considerably deformed at pressures less than the ultimate pressure, the failure being successively developed. The deformation, which is illustrated by the distance between the horizontal straight line through A and the branch of the curve in Fig. 20, was much less for the samples from 5 and 8 m depth where the organic content was smaller. At failure these latter curves were sharply bent which indicates an instant collapse of the microstructure in the failure zone (brittle failure). The

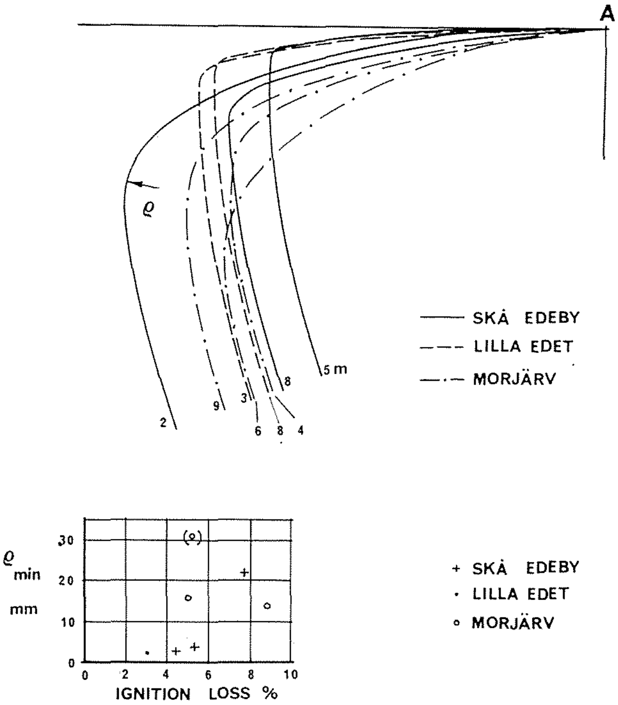


FIG. 20 Stress/strain relationship determined by the SGI unconfined compression apparatus. The diagram shows the relationship between ignition loss and ϱ. The ϱ-values refer to the recorded curves.

deformation at sub-failure pressures was small for the Lilla Edet material. At failure the structure collapse was complete and instant in the failure zone. As in the case of the upper clay layer in Skå-Edeby, the deformation of the organic Morjärv samples was large at sub-failure pressures. Also, the failure was successively developed. As shown by the diagram in Fig. 20 there seems to be a correlation between organic content and successively developed failure as illustrated by the (minimum) radius of curvature ϱ. The close similarity between the shear strength values at the unconfined compression tests and those at the cone tests as well as of field vane tests indicates that the compressed samples, except for the Morjärv sample from 6 m depth, were not noticeably disturbed at the extraction.

4.5 Shear strength and sensitivity

The overconsolidated top layers at Skå-Edeby and Lilla Edet have a shear strength of the order of 1.2–1.6 N/cm^2 according to the cone penetration tests. The remaining clay layers, except the Lilla Edet sediments, are soft and normally consolidated and have a shear strength of the order of 0.9 to 1.3 N/cm^2. The Lilla Edet clay layers at 6 and 19 m depth, which are slightly overconsolidated, have average shear strength values of 1.6 to 2.4 N/cm^2 respectively.

According to Swedish terminology, the investigated clay layers in Skå-Edeby and at 19 m depth in Lilla Edet have a normal sensitivity. The clay layers at 3 m depth and 6 m depth at SGU VII:1 in Lilla Edet are quick, whereas the layer at 3 m depth at SGU VII:2 in Lilla Edet and the layers in Morjärv may be called highly sensitive.

5 ELECTRON-OPTICAL INVESTIGATIONS

5.1 Specimen preparation

The pore water in small specimens of undisturbed clay was replaced by acrylate plastic by means of a diffusion process. The following preparation was used:

1. A prismatic specimen with a basal area of approximately 1/8–1/2 cm^2 and a length of approximately 1/2–1 cm was cut from a clay sample with a thin steel wire so that the orientation could be related to the clay layers *in situ.*

2. The specimen was placed in a solution of 50 % ethyl alcohol in water for 30 min, in 70 % alcohol for 5 min and finally in 90 % and 99.5 % alcohol for 5 min each.

3. The specimen was placed in a monomer mixture consisting of 85 % butyl methacrylate and 15 % methyl methacrylate for 45 min. This process was repeated once.

4. The specimen was placed for 90 min in a solution consisting of 98 % monomer and 2 % 2,4-dichlorbenzoylperoxide (EWM) catalyst and finally transferred to a gelatine or plastic capsule which was filled with monomer and catalyst.

5. After polymerization by incubating the sample for 15 h in an oven at 60° C, the capsule was removed. The sample was then trimmed and cut so that the original orientation of the thin sections obtained in the microtome could be defined. This was effected by giving the sections a rectangular shape.

For the largest specimens, the time of treatment in the various baths had to be doubled.

The trimming and cutting operation, which was made by using an LKB Ultrotome 4801 A/4202 A, has been described and commented on previously (PUSCH, 1968). In the standard procedure, the thin sections were taken parallel to the vertical plane *in situ.*

The microtome was equipped with a diamond knife, the collecting trough of which being filled with 10 % acetone solution in water. The sections, which had a thickness of about 0.05 μm, were placed on carbon-coated 150 mesh grids for investigation in a Siemens Elmiskop I. The microscope, which was equipped with a platinum-iridium aperture of 50 μm in the objective lens, was operated at 80 kV.

Ocular inspection was made using a wide range of magnifications and micrographs were taken at random. The electronic magnification during exposure was ×5,000–10,000 depending on the size of the object. The plates used were Ilford "Special lantern".

With the applied preparation procedure the majority of the thin sections obtained from pure clay could be used. When the diamond cutting edge reached coarser particles harder than the embedding plastic substance, these particles were in most cases pushed up in front of the edge, which caused distortion of the clay structure in the vicinity of the coarse particles. By observing the cutting process through the eye-piece of the microtome, it was possible, however, to select those sections which appeared to be intact.

The important problem of the influence of the chemical treatment and the shearing operations on the microstructure has been investigated by the author (PUSCH, 1968). The applied procedure for obtaining ultra-thin sections seems to be satisfactorily reliable for the preservation of the original microstructure.

5.2 Statistical description of clay microstructure

Clay microstructure is suitably analysed using simple statistical methods. The total pore area P in percent of the total area T of the studied thin section and the average size of the pores are characteristic microstructural parameters (cf. PUSCH, 1968). The dimensions of the pores are suitably defined in accordance with the concepts used for clay particles but in microstructural analysis only the longest intercept (a_p) is measured. Since the pore image represents a cross section without reference to the real extension and orientation of the pore, the dimension a_p only describes the sectioned part of the pore. Pores extending outside the micrograph edges are measured as if they have the truncated shape.

All the measurements were based on drawn images of the micrographs. The drawings, in which no discrete particles were depicted, show black areas for the sectioned clay particle matrix with no observable pore space. Depending on particle size and arrangement, this matrix has a varying density which is not illustrated by the even black areas. This means that the true porosity cannot be judged from the drawings. Although the sections are extremely thin they contain a certain number of very small pores which are embedded in the clay sections and are thus not revealed. The observed frequency of the smallest pores is therefore probably not representative. The under-representation of small pores also results from the fact that poreswith an a_p-value less than 0.031 μm could not be identified with certainty. However, due to their small dimensions they act more or less as the surrounding minerals in the natural sediment on account of the high water viscosity and may therefore be disregarded.

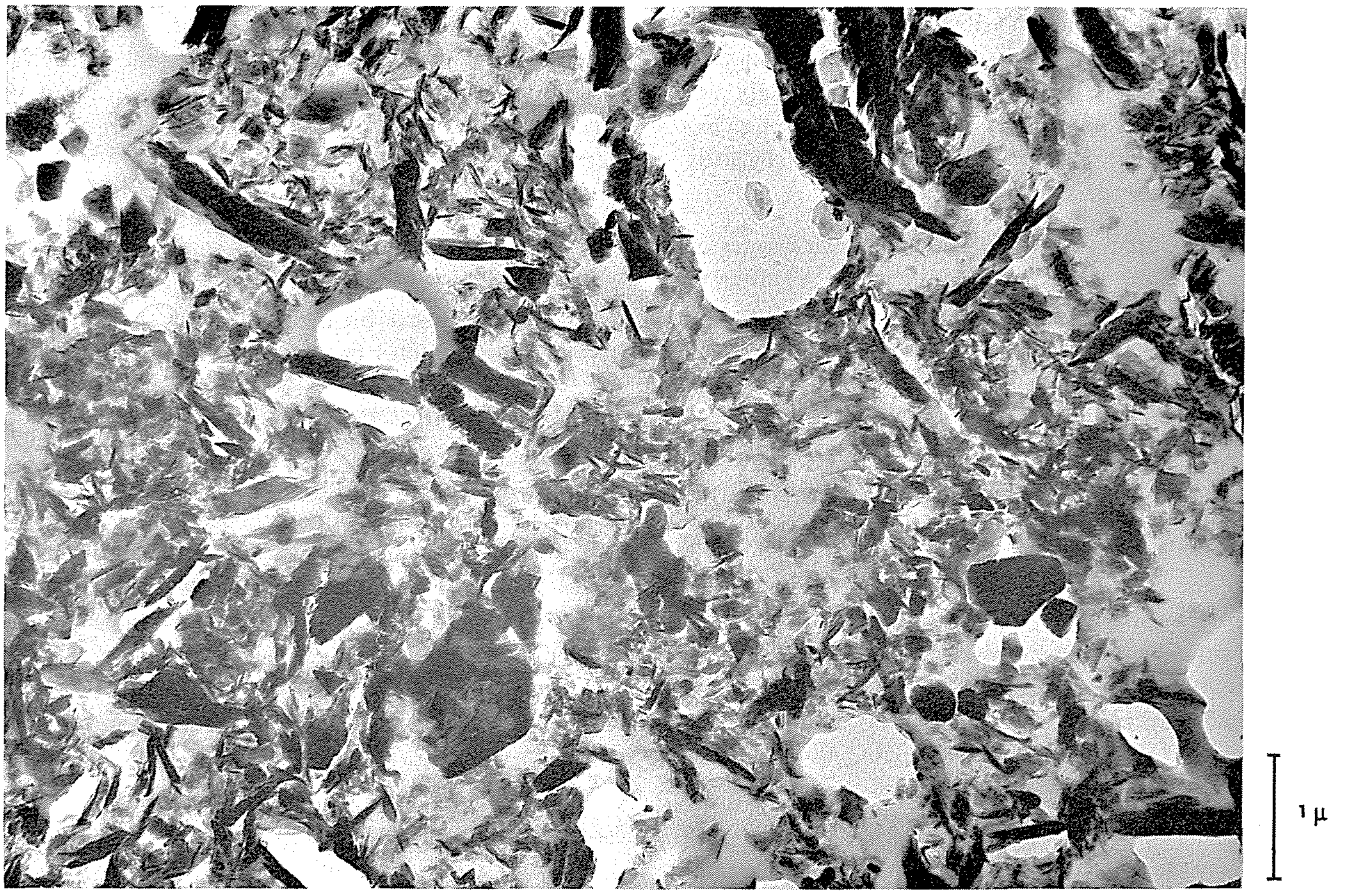
A series of micrographs of the investigated clays is illustrated in Figs. 21–26. These figures also show histograms of a_p. The class intervals agree in the main with the ones used by the author in the graphical representation of clay particle size and shape.

The statistical treatment of the images drawn from the micrographs gave the values in Table 6, each sample being represented by 5 micrographs on average. The content of the coarsest pores ("micro-pores" and "fine pores" according to ODÉN's classification) is illustrated by the 95th percentile. The majority of the measurable pores in all the investigated clays belongs to the "ultrapore" fraction (<0.2 μm) and to the finer part of the "micro-pore" fraction (2–0.2 μm) as defined by ODÉN.

TABLE 6. The structural parameter P/T and median values, quartiles and percentile P_{95} of the pore size a_p.

Site	Depth m	P/T %	a_p in μm M	Q_1	Q_3	P_{95}[3]	S_K	log S_0
Skå-Edeby	2.0	17.4	0.21	0.14	0.34	0.70	1.08	0.192
	5.0	9.4	0.11	0.08	0.20	0.50	1.32	0.199
	7.0	13.8	0.13	0.10	0.19	0.35	1.12	0.139
	8.0	21.5	0.24	0.18	0.40	0.80	1.25	0.173
	9.0	24.9	0.20	0.14	0.31	0.70	1.09	0.172
	10.0	11.3	0.19	0.11	0.32	0.70	0.98	0.232
Lilla Edet	3.0[1]	37.5	0.15	0.06	0.25	1.10	0.67	0.310
	3.0[2]	48.4	0.14	0.09	0.25	1.30	1.15	0.221
	6.0[1]	49.3	0.13	0.09	0.25	0.95	1.33	0.221
	19.0[2]	39.7	0.14	0.10	0.28	1.00	1.43	0.223
Morjärv	4.0	57.0	0.21	0.14	0.43	1.50	1.37	0.243
	6.0	45.2	0.33	0.22	0.55	2.50	1.11	0.199
	9.0	56.2	0.35	0.25	0.60	2.00	1.22	0.190

[1] Bore hole close to SGU VII: 1
[2] Bore hole close to SGU VII: 2
[3] 95th percentile (coarsest part of the cumulative curve).



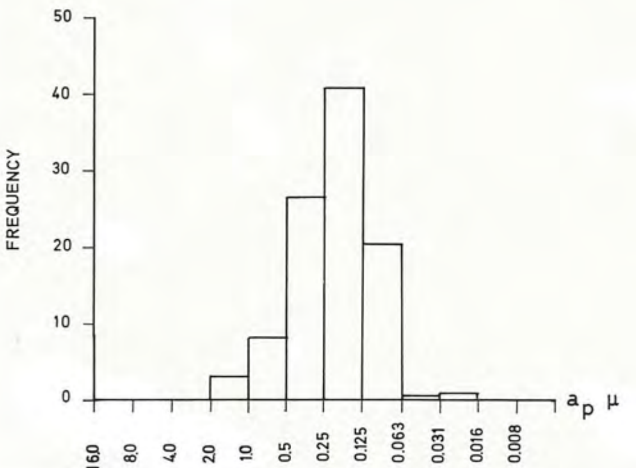
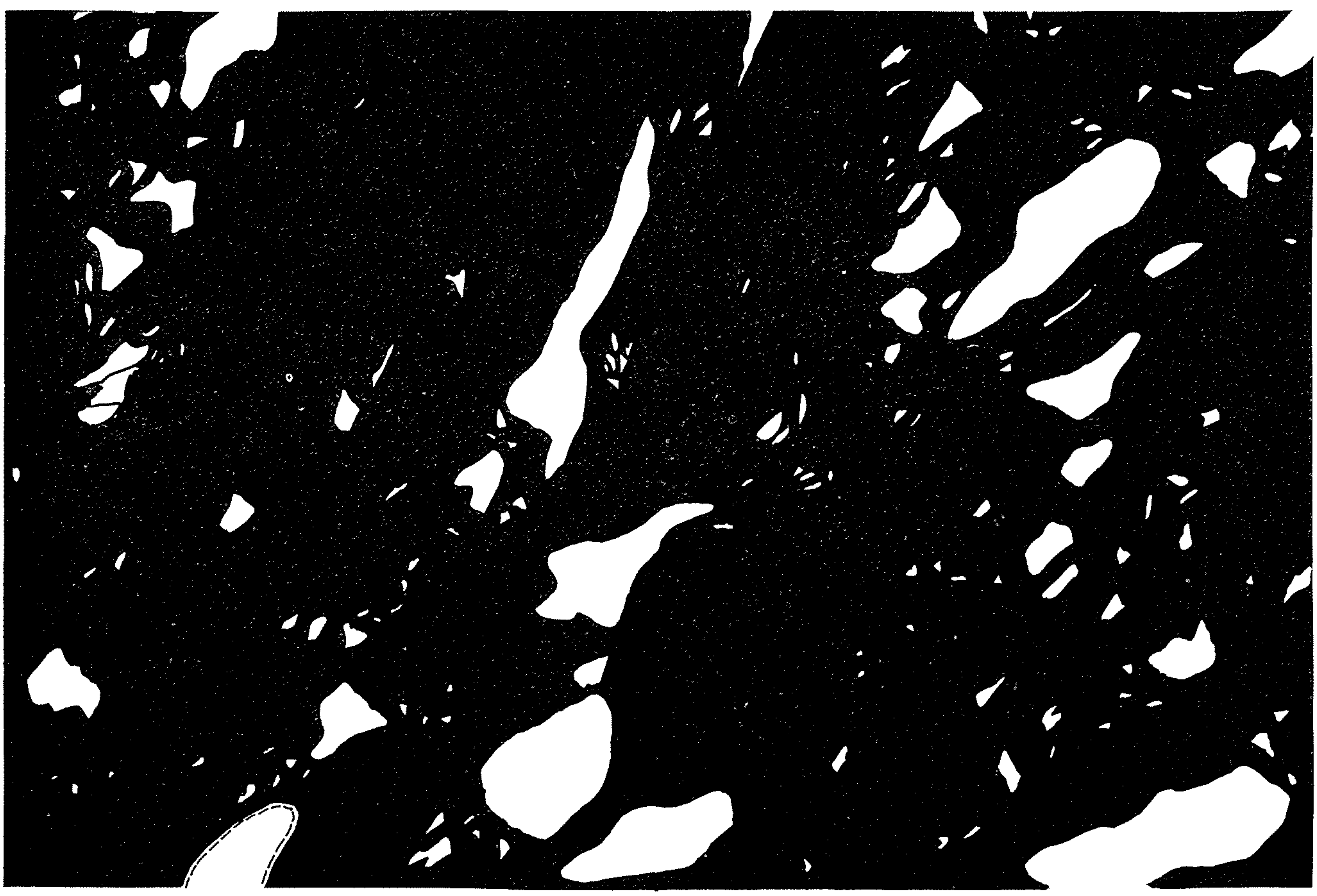


FIG. 21 Electron micrographs of Skå-Edeby clay from 2 m depth. The dark parts represent mineral and organic substances while the light-grey parts represent pore space. White areas are natural or artificial holes in the sections. The left, lower picture is a schematic drawing of the pore system of a representative part of the micrograph above. The histogram shows the size distribution of the (maximum) pore diameter.


1 μ



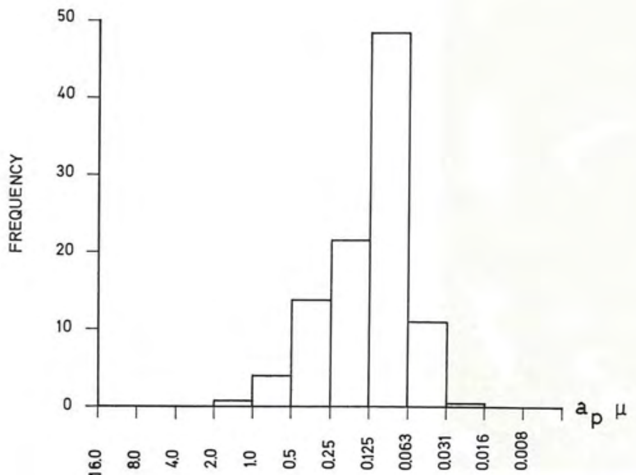
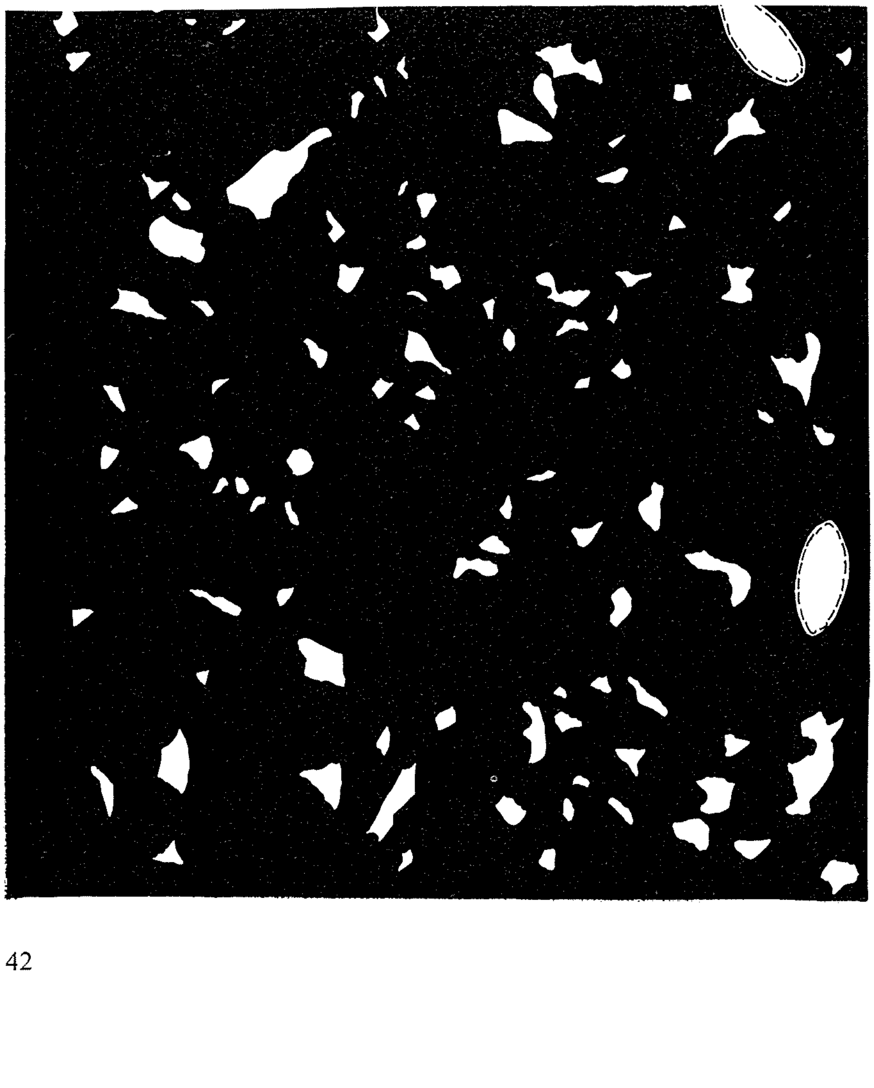


FIG. 22 Electron micrographs of Skå-Edeby clay from 5 m depth. Notice the fairly uniform particle distribution. Artificially produced holes are marked with broken lines in the schematic drawing.


1 μ



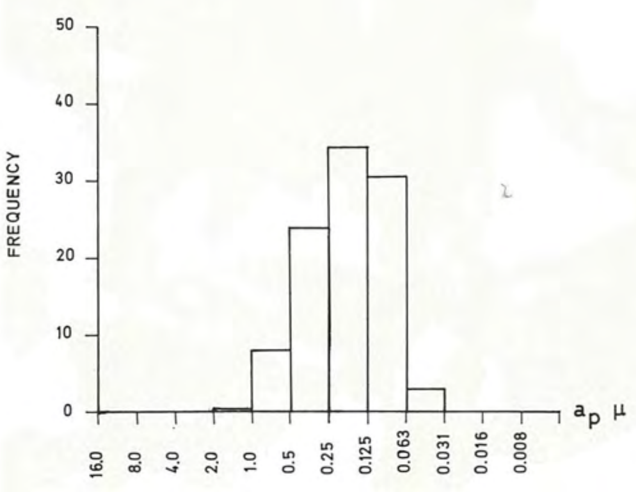
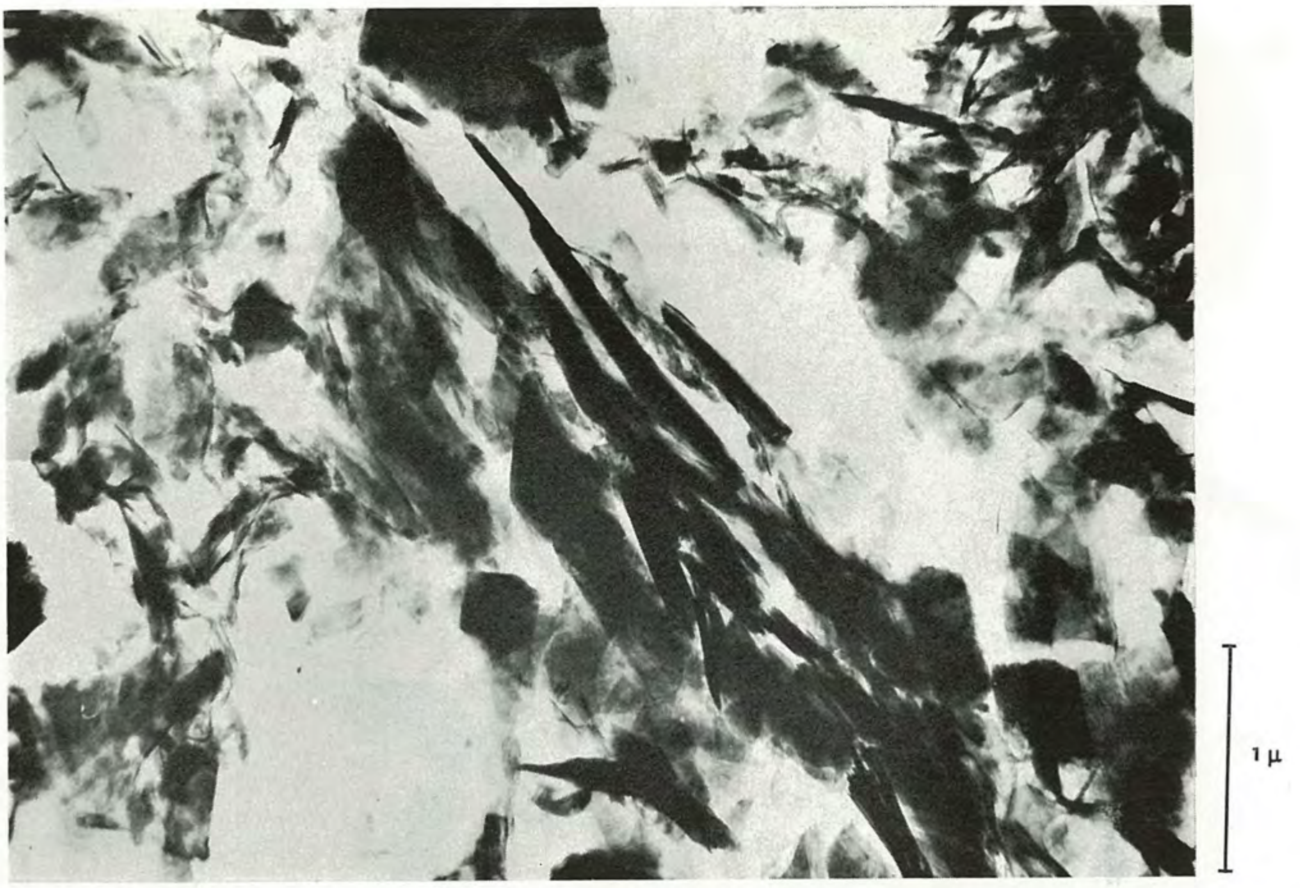


FIG. 23 Electron micrographs of Skå-Edeby clay from 10 m depth. Notice the tendency of larger clay particles to be oriented (parallel with the plane of sedimentation) in the left micrograph. The micrograph to the right illustrates a typical network of linked aggregates.

0.5 µ



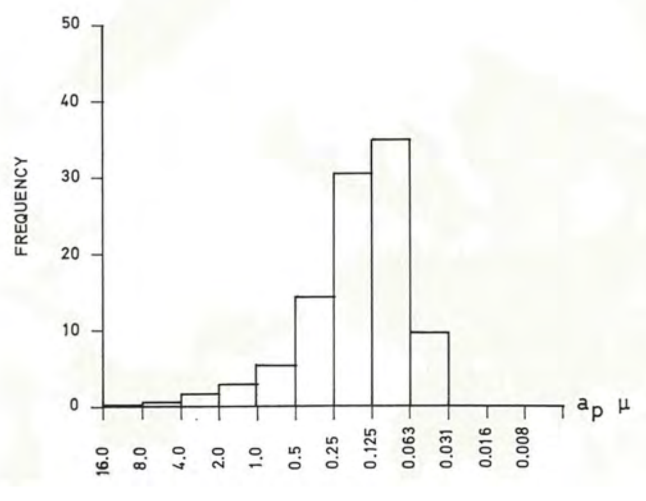
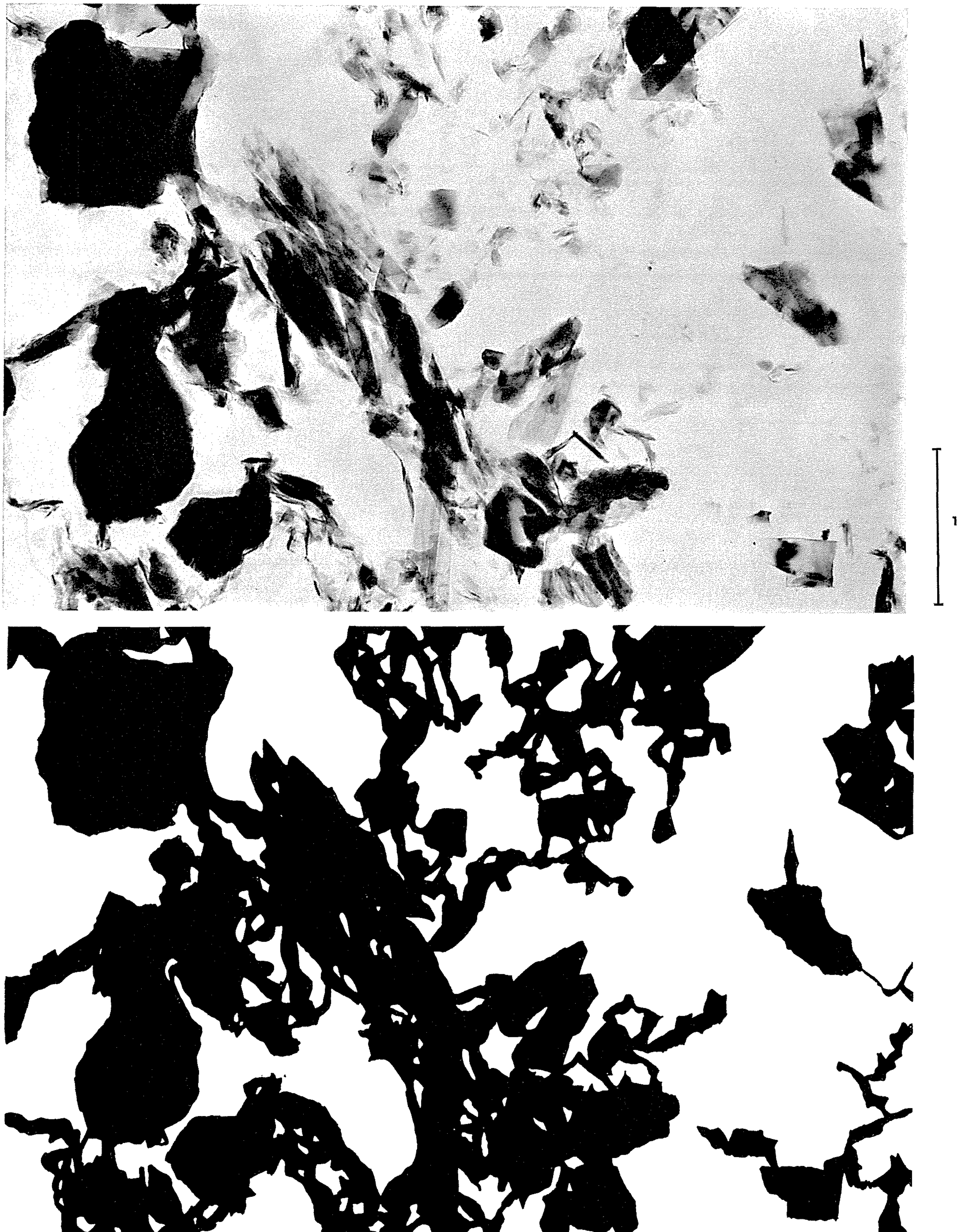
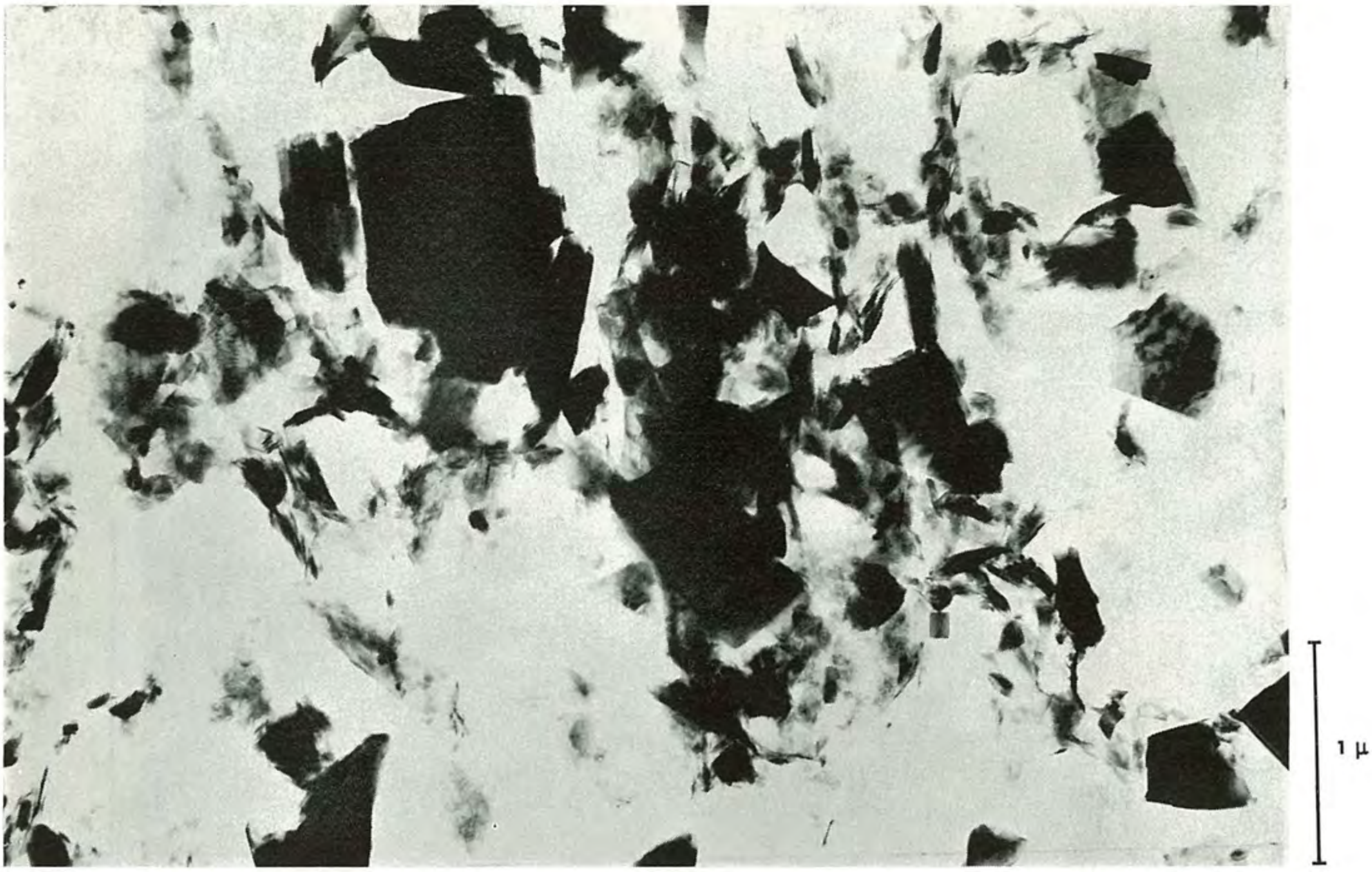


FIG. 24 Electron micrographs of Lilla Edet clay from 3 m depth. Notice the very open particle arrangement characterized by large, dense aggregates and large pores.


1 µ



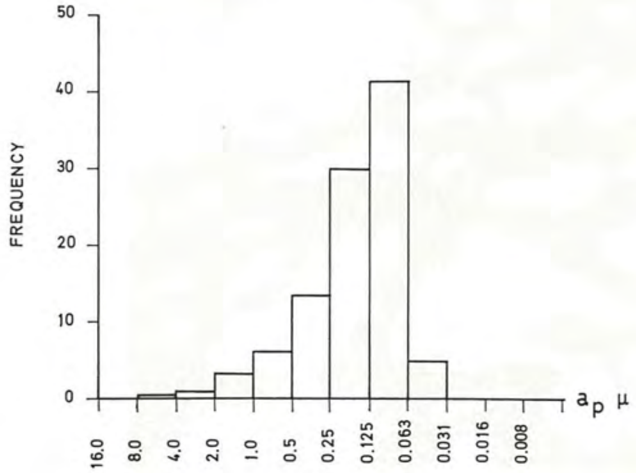
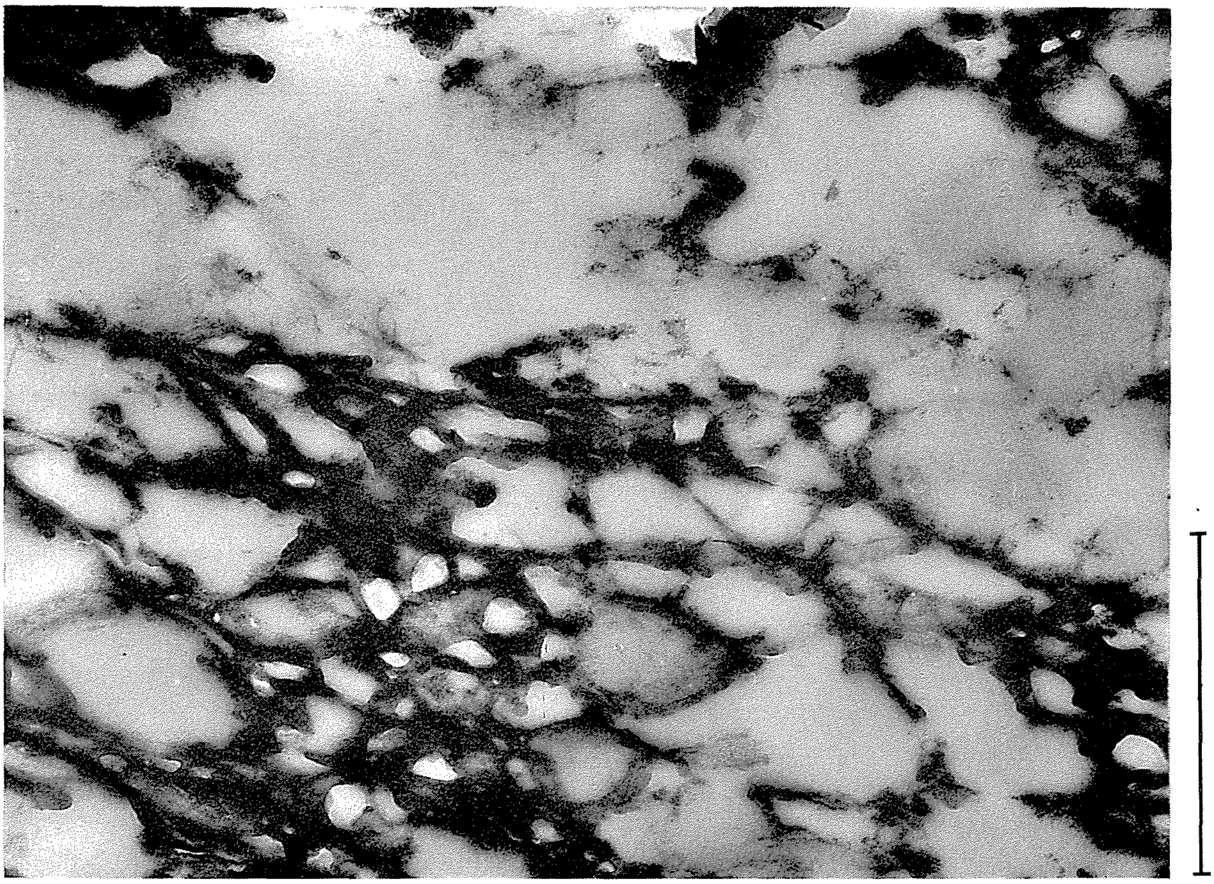
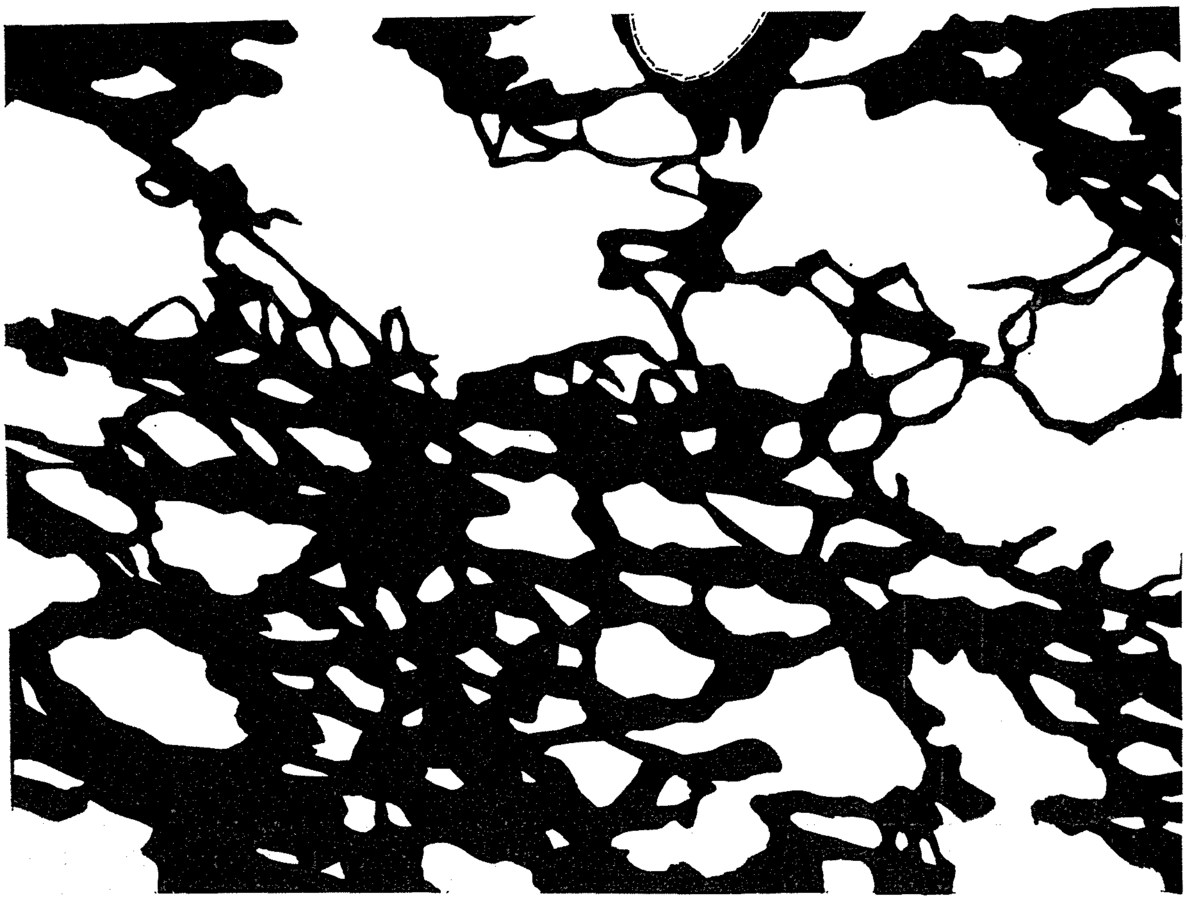
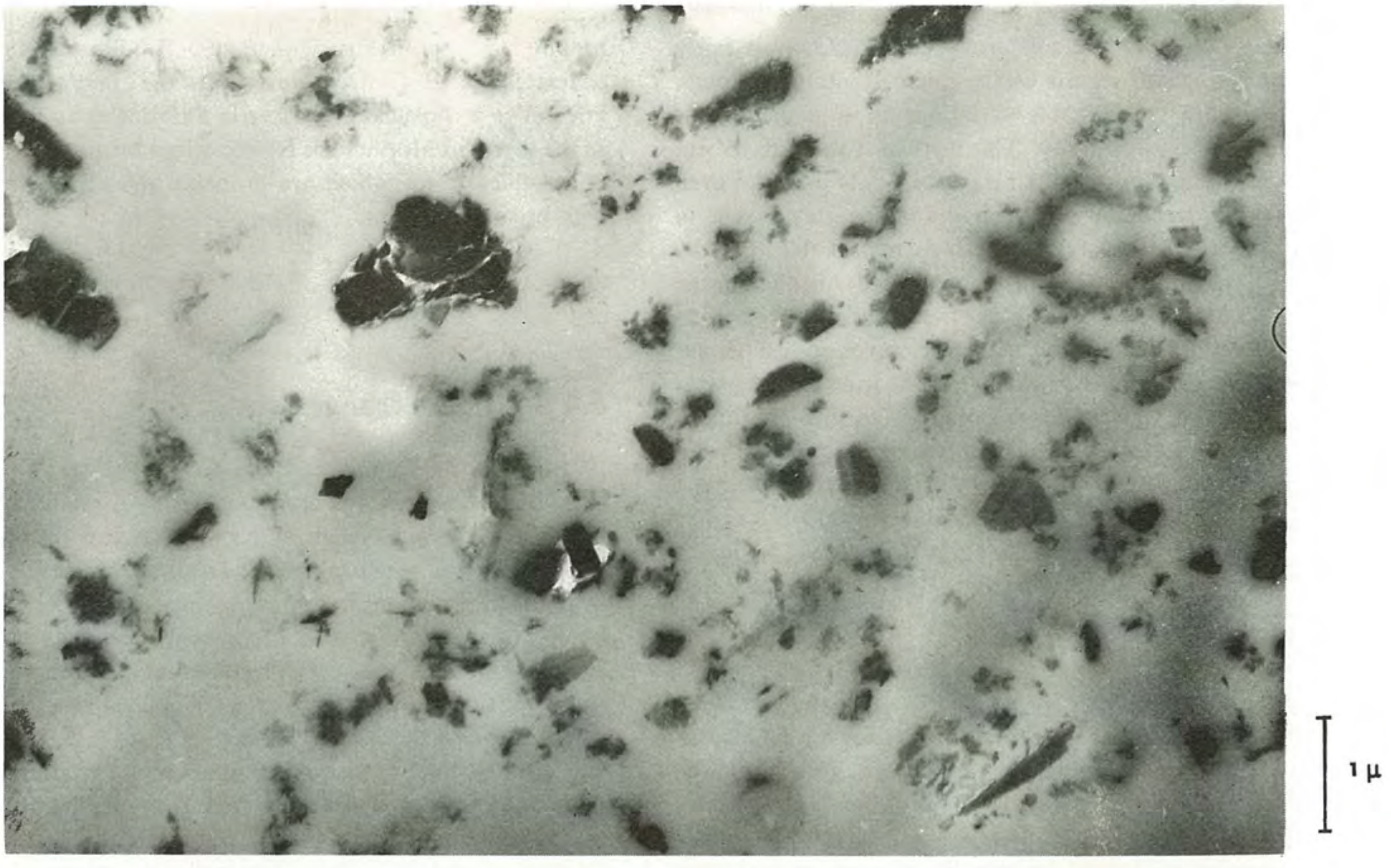


FIG. 25 Electron micrographs of quick Lilla Edet clay from 6 m depth. Similar general patterns were shown by the normally sensitive Lilla Edet clay from 19 m depth.


1 μ





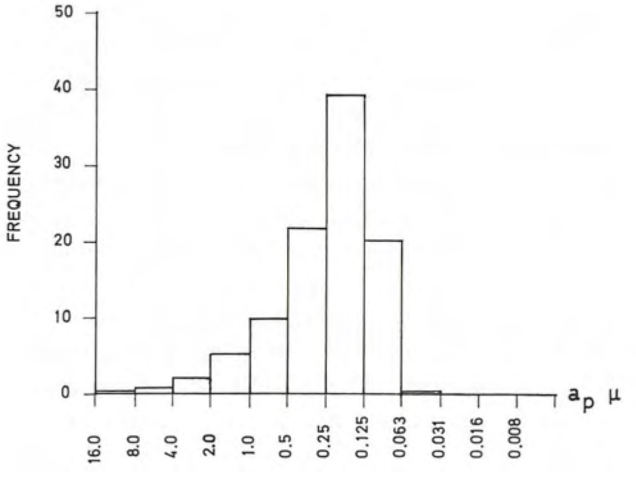


FIG. 26 Electron micrographs of Morjärv clay from 4 m depth. Notice the deficiency of contacts between the aggregates in the right micrograph. This deficiency is only apparent since consecutive sections show continuous links between most aggregates.

The median values of the pore size indicate that the clay matrix of the soil profile in Skå-Edeby is fairly uniform with regard to the microstructure, although there are considerable variations in organic content and original salinity. The median values (0.18 μm on average) are in the range 0.11–0.24 μm. An even smaller variation was found for the clay layers in Lilla Edet (0.13–0.15 μm). It is remarkable that the average pore size is of the same order in the marine Lilla Edet clay as in the fresh and brackish Skå-Edeby clay. The P/T-values are, however, quite different, which is due to the fact that the marine clay contains a number of very large pores which give the major contribution to the P/T value. This is revealed by the 95th percentile values. The Morjärv clay has the most open particle network of the investigated clays. The high median, quartile and 95th percentile values of the pore size a_p and the high P/T-values indicate that the microstructure is characterized by very large pores.

The log S_0-values of a_p, which describe the sorting of the pore size, are of the same order for all the investigated specimens with a few exceptions. This is also the case for the skewness values. Thus, the microstructure of the investigated clays basically forms a standard pattern, but special features like the existence of large pores in marine clay occur. It should be mentioned that certain quick marine clays are characterized by an extremely porous particle network, which can be described as a system of negative tactoids (Karlsson & Pusch, 1967). Such clays have an extraordinarily high water content and give a large amount of excess water by remoulding.

Various microstructural features have been studied like gas voids, general particle arrangement, particle orientation and type of contact of adjacent particles. They will all be considered in the following text.

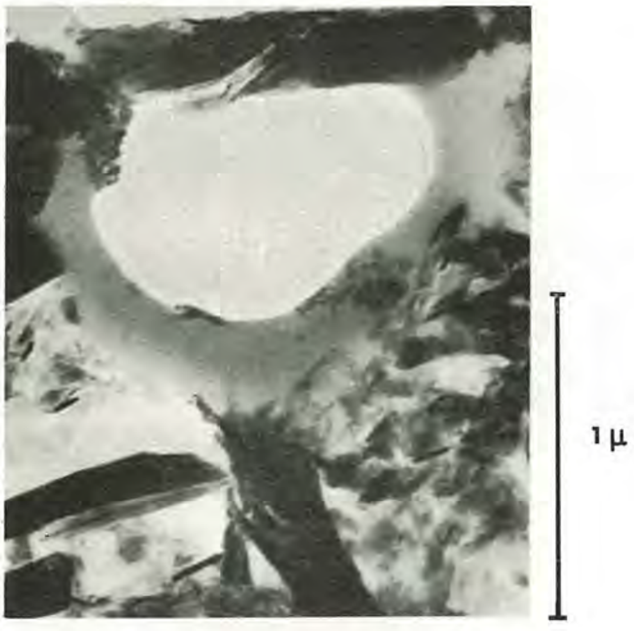
5.3. Gas voids

The investigation of the size, shape and arrangement of micropores showed that the ultra-thin sections contained small isolated voids not filled by plastic substance. These voids were probably present as gas-filled pores already in the natural sediment. They have a smooth outline by which they are easily distinguished from irregularly shaped distorted parts of the sections caused by the microtome knife. A typical void of the rounded type with a smooth outline is shown in Fig. 27 a. Figure 27 b illustrates a characteristic distortion caused by the microtome knife. The postglacial clay layer in Skå-Edeby as well as the layers in Morjärv are characterized by a number of gas-filled pores which are probably related to the high organic contents.

5.4 Particle arrangement

The Skå-Edeby clay is characterized by a relatively uniform, dispersed system of particles as can be inferred from the small a_p- and P/T-values. A close examination of the micrographs revealed a number of free particles but most of the clay platelets were collected in aggregates connected by links and groups of small particles. Most aggregates contained one or a few large particles. The specimens from 2, 7, 8, 9, and 10 m depth showed similar microstructural patterns. The fresh-water clay from 5 m depth with the highest content of colloidal clay particles, had the smallest a_p- and P/T-values. Hence, the particles at this depth are more uniformly distributed (but still aggregated) than in the rest of the profile.

The Lilla Edet clay consists of dense, large aggregates separated by a pore system which contains large voids. The micrographs showed that the linkage of groups or chains of small particles in and between the aggregates or between larger particles is a very characteristic feature. Domain-like bodies were found especially in the quick Lilla Edet clay (Fig. 28). It may be a question of partly split micaceous particles affected by weathering processes which caused a partial or complete separation of the individual crystallographic sheets. Weathering processes are known to involve a reduction of the potassium content in inner-layer positions by which expanding zones are formed as a first process in the transition mica→illite→mixed layer minerals (Jackson *et al.* 1952, Jackson 1965 and Jörgensen, 1965). The assumption of such crystallographic changes is in accordance with the interpretation of the X-ray diffractograms of the Lilla Edet clay. However, in most cases it is impossible to decide whether the domain-like bodies are split particles, sheared zones of particles or aggregates deposited as such. Domain-like bodies were also present in the other clays but to a much smaller extent.



a

b

FIG. 27 a) Gas void. The well rounded outline is probably diagnostic. b) Distortion by the microtome in the cutting process.

The very large aggregates of the Morjärv clay, which are fairly open, seem to be secondary formations of coupled small aggregates (Fig. 29). A characteristic feature is the presence of fibrous aggregates of presumably organic origin in which clay particles are embedded. Such aggregates are depicted in Fig. 30 which also shows some well rounded voids which probably contained gas in the natural state.

5.5 Particle orientation

Preferred orientation over large parts of the particle network was not found in the investigated specimens from the postglacial Skå-Edeby clay and from the Lilla Edet and Morjärv clays. In the glacial Skå-Edeby clays from 7 to 10 m depth preferred orientation parallel to the horizontal plane *in situ* was observed but only for particles with an *a*-value higher than about 0.5 μm. The determination and description of particle orientation was made by using DAPPLE's and ROMINGER's (1945) statistical method. It involves measurement of the angle between the direction of particle elongation and an arbitrarily chosen reference direction, which for instance may be taken as the plane of sedimentation. By plotting the values on a histogram a modal value is obtained if there is a preferred orientation. By experience it is generally possible to identify a preferred orientation directly by ocular inspection of micrographs. The fact that preferred orientation was not found in the Lilla Edet clay from 19 m depth indicates that a consolidation pressure of about 12 N/cm^2 is not sufficient to cause microstructural anisotropy in a marine sediment rich in clay.

5.6 Particle contacts

A question of great interest is the mode in which adjacent particles are arranged. The micrographs offer certain possibilities of investigating this although the detailed particle contacts are obscured by the limited resolving power of the microscope (about 0.0005 μm) and by the structural pattern of the

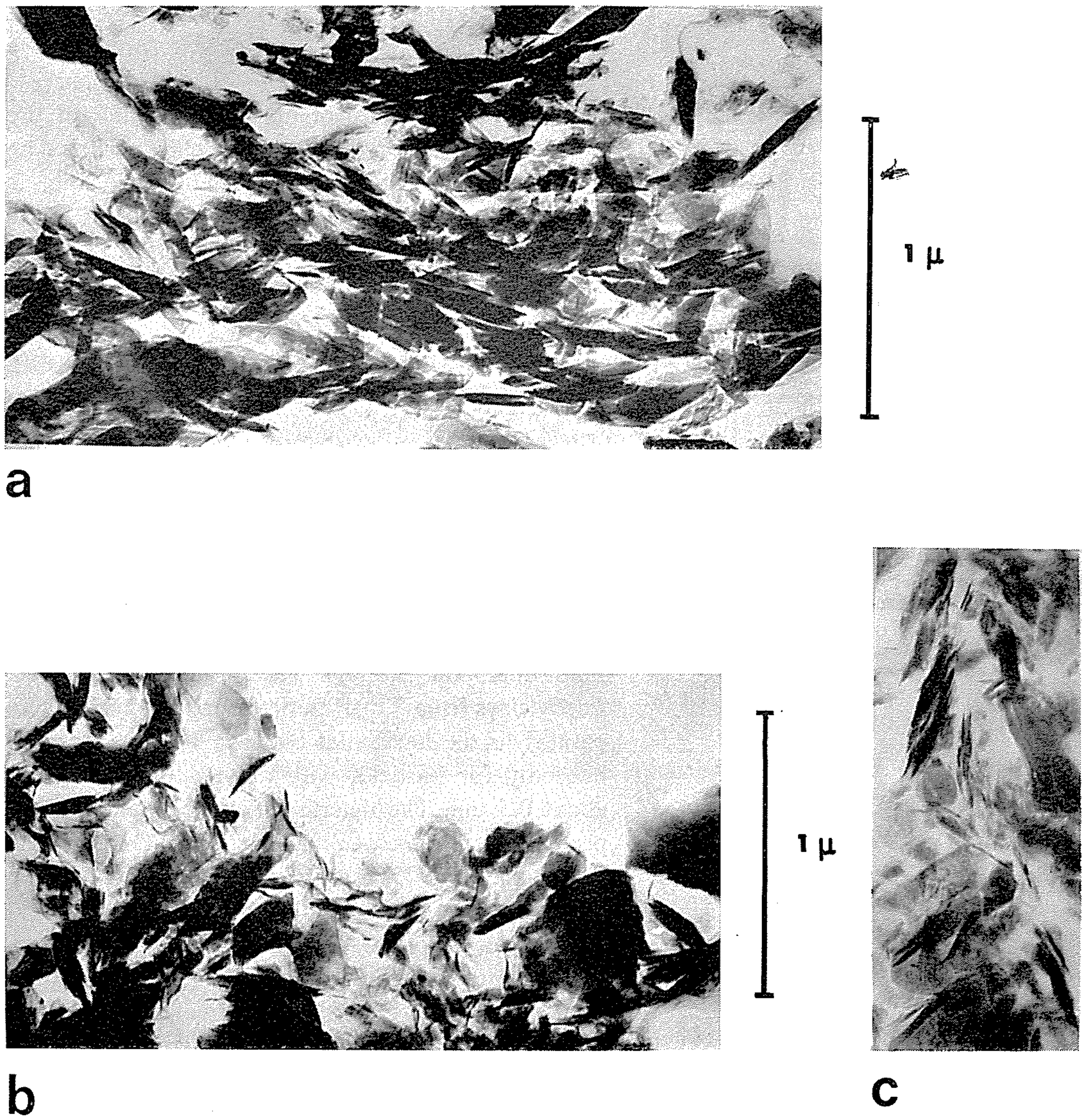


FIG. 28 Domain-like formations. a and b) Quick Lilla Edet clay. c) Partly split particles alternatively oriented particles in Skå-Edeby clay.

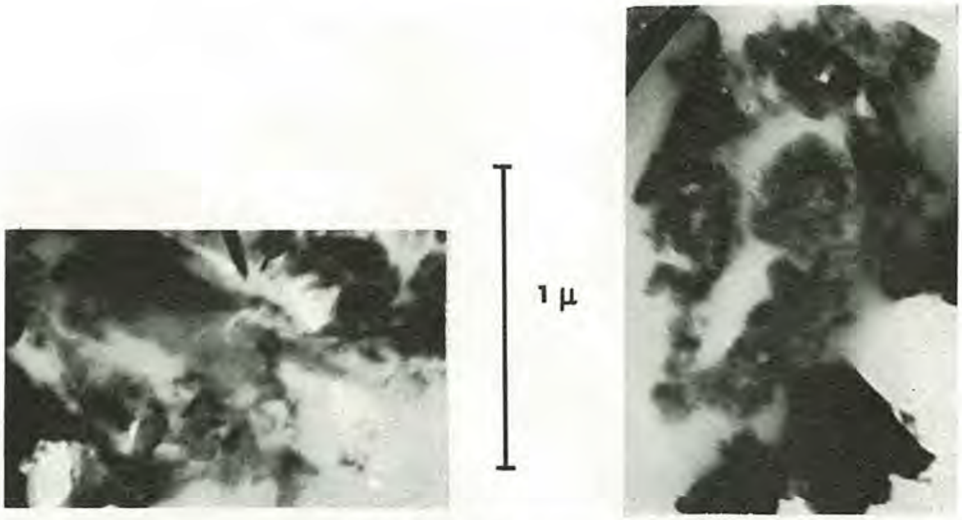


FIG. 29 Particle aggregates of varying density. The large aggregates contain microaggregates separated by very small pores. Morjärv clay from 6 m depth.

photographic emulsion of the photographic plates and paper. The identification of the mutual position of small particles is especially difficult but larger particles—still well within the clay fraction—often show a definite arrangement. The majority of the observed contacts were of the edge-to-edge type (Fig. 31) but edge-to-face and face-to-face modes were also identified. The minimum distance between closely located clay particles was found to be less than 0.002 μm. Since the clay preparation may involve minute movements of adjacent particles, the interpretation of such microstructural details is uncertain.

5.7 Discussion

The large changes in electrolyte contents in the pore water, which have occurred since the deposition and which have led to a fairly equal present salinity of all the investigated clays, have not resulted in identical microstructural patterns. It can be assumed that the original clay particle association has been preserved and that the observed microstructural features reflect the circumstances under which the clays were formed. Hence, clays deposited in highly saline water form larger aggregates than clays formed in fresh or brackish water, P/T being a characteristic measure of the degree of aggregation. However, this parameter is not a simple function of the salinity of the water in which the deposition took place. Thus, the brackish Morjärv clay shows higher P/T-values than the marine Lilla Edet clay, which is probably due to the higher organic content in the Morjärv clay. The organic substance may have created strong structural bonds at a high void ratio. The concentration of the settling suspension, the size distribution and the mineral type as well as the sedimentation rate may also be of importance when the microstructural pattern is formed.

It can be concluded that the most typical feature of all the investigated clays is that they are all built up by aggregates connected by links of particles. This heterogeneity means that simplified microstructural models of a particle network consisting of regularly arranged particles with uniform size and shape are not valid. The particle contacts in the investigated clays seem to be of three types, edge-to-edge, edge-to-face, and face-to-face, which is in agreement with VAN OLPHEN's statement. In fact, the simultaneous occurrence of the three modes of association is compatible with the fact that differential particle movements may have occurred during consolidation. In the author's opinion the mode of association is therefore not a characteristic microstructural feature. Instead, the degree of aggregation and the linking of particle

FIG. 30 Fibrous material of a presumed organic origin.
Morjärv clay from 6 m depth.

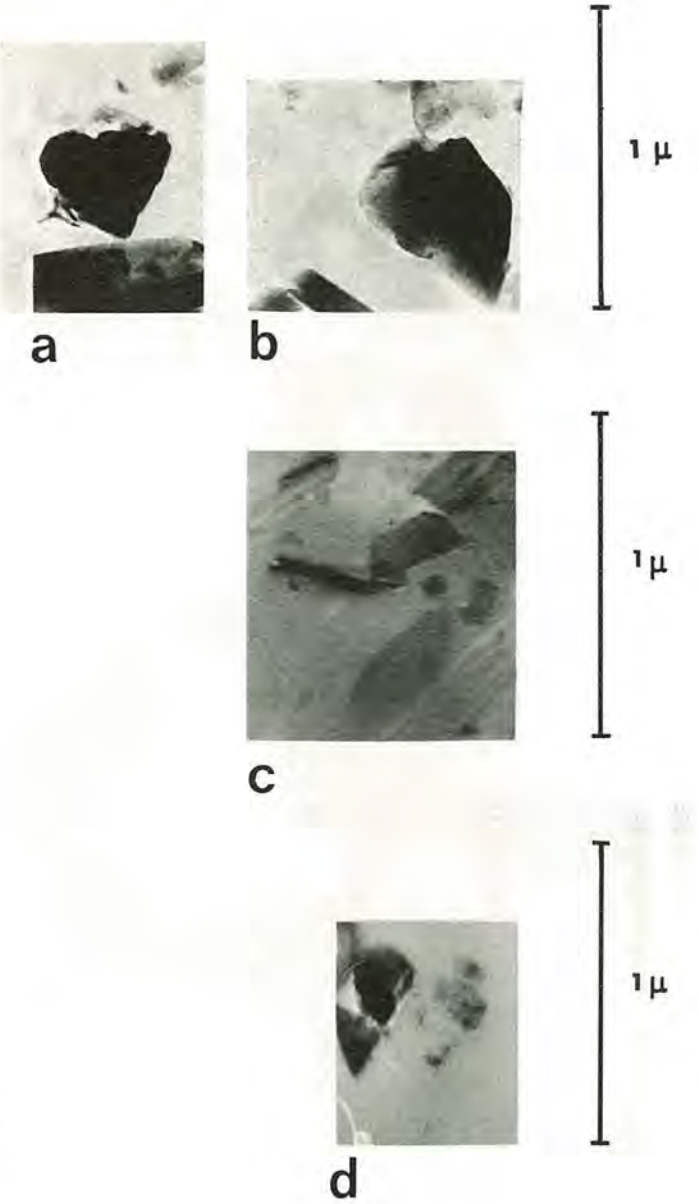


FIG. 31 Particle contacts. a and b) Edge-to-edge contacts in Lilla Edet clay. c) Edge-to-edge and edge-to-face contacts in Skå-Edeby clay. d) Edge-to-edge and edge-to-face contacts in Morjärv clay.

groups between larger aggregates or particles should be considered to be most important.

LAMBE's hypothesis of flocculation with a parallel particle arrangement at increasing water salinity is not valid for the investigated marine clays as regards the general microstructural pattern. The conditions within larger, dense aggregates are more difficult to interpret but no clear tendency towards a parallel arrangement can be seen except for the domain-like bodies.

MITCHELL's conclusion that preferred orientation is mainly found in non-marine clays is strongly supported by the author's investigation. It should be stressed, however, that preferred orientation in such clays is only found for relatively large particles, the fine-grained matrix being characterized by random particle orientation.

On the basis of the microstructural studies, the schematic pictures of a marine clay and a fresh water clay shown in Fig. 32 are suggested. Attention has to be paid to the varying density, rigidity and deformation properties of the aggregates and of the linking system of particles. These properties should be dependent on the average particle distance, which can also be expressed as a water content of these microstructural units. The particle distance within the aggregates and the distance between the aggregates may serve as characteristic structural parameters from

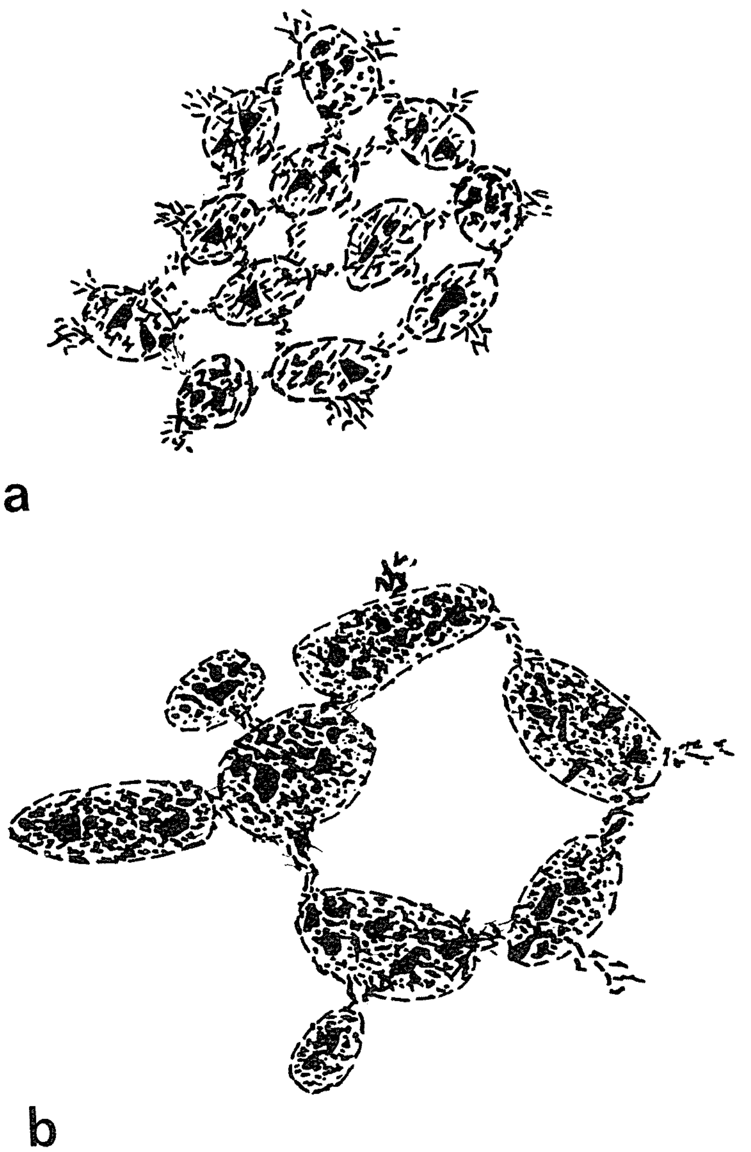


FIG. 32 Schematic clay particle arrangement. a) Clay deposited in fresh water having relatively porous aggregates and small voids. b) Marine clay with large, dense aggregates separated by large voids.

a rheological point of view. The a_p-value can be used as a measure of the average distance between the aggregates while the average particle distance within the aggregates has to be measured directly from the micrographs. This is illustrated by Fig. 33, which shows aggregates of an open type and detailed schematic drawings of the same aggregates. The drawings, which depict all clearly identified particles within a certain element, show that the average distance between the centers of gravity of particles situated on opposite sides of the intra-aggregate voids is of the order of 0.02 μm in the Skå-Edeby clays, while it is about 0.01–0.015 μm in the Lilla Edet and Morjärv clays. The majority of the aggregates in all the investigated clays are even denser and therefore characterized by even smaller particle distances. The observed difference between the Skå-Edeby clay and the other clays, meaning that the Skå-Edeby aggregates are less dense, explains why the average water content of the clays in bulk is of a similar order despite the different microstructural patterns and parameter values. At an average water content of 60–100 % of the investigated clays, the mean water content in the majority of the aggregates may well be of the order of 10–30 %. The distance between adjacent particles—interpreted as the distance between their centers of gravity—depends on clay particle size and shape as well as on the type and amount of interstitial organic substance and ions. The small intra-aggregate particle distance in the Lilla Edet clays is due to the small particle size and to the generally observed close arrangement of particles within aggregates and links of marine clays.

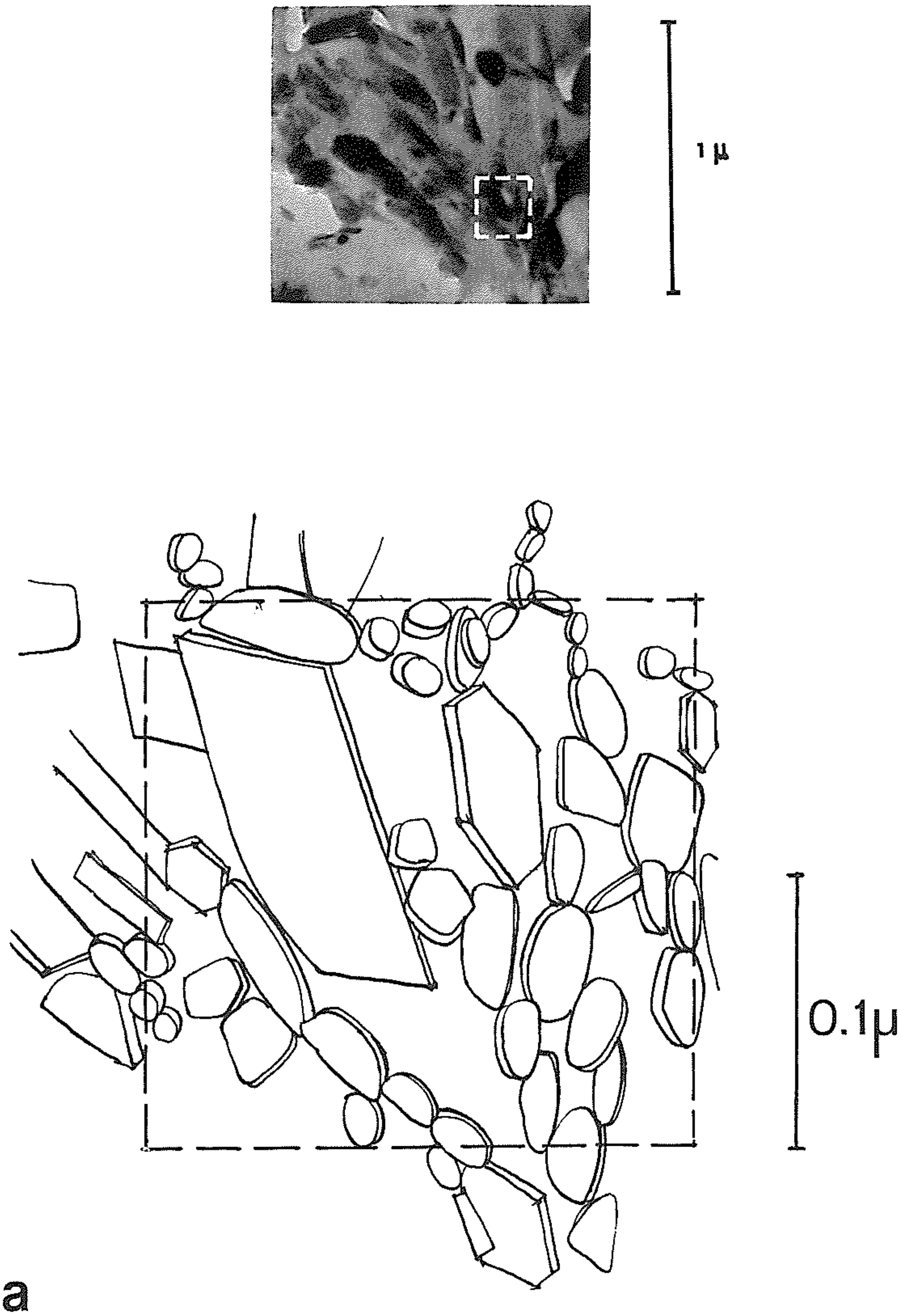
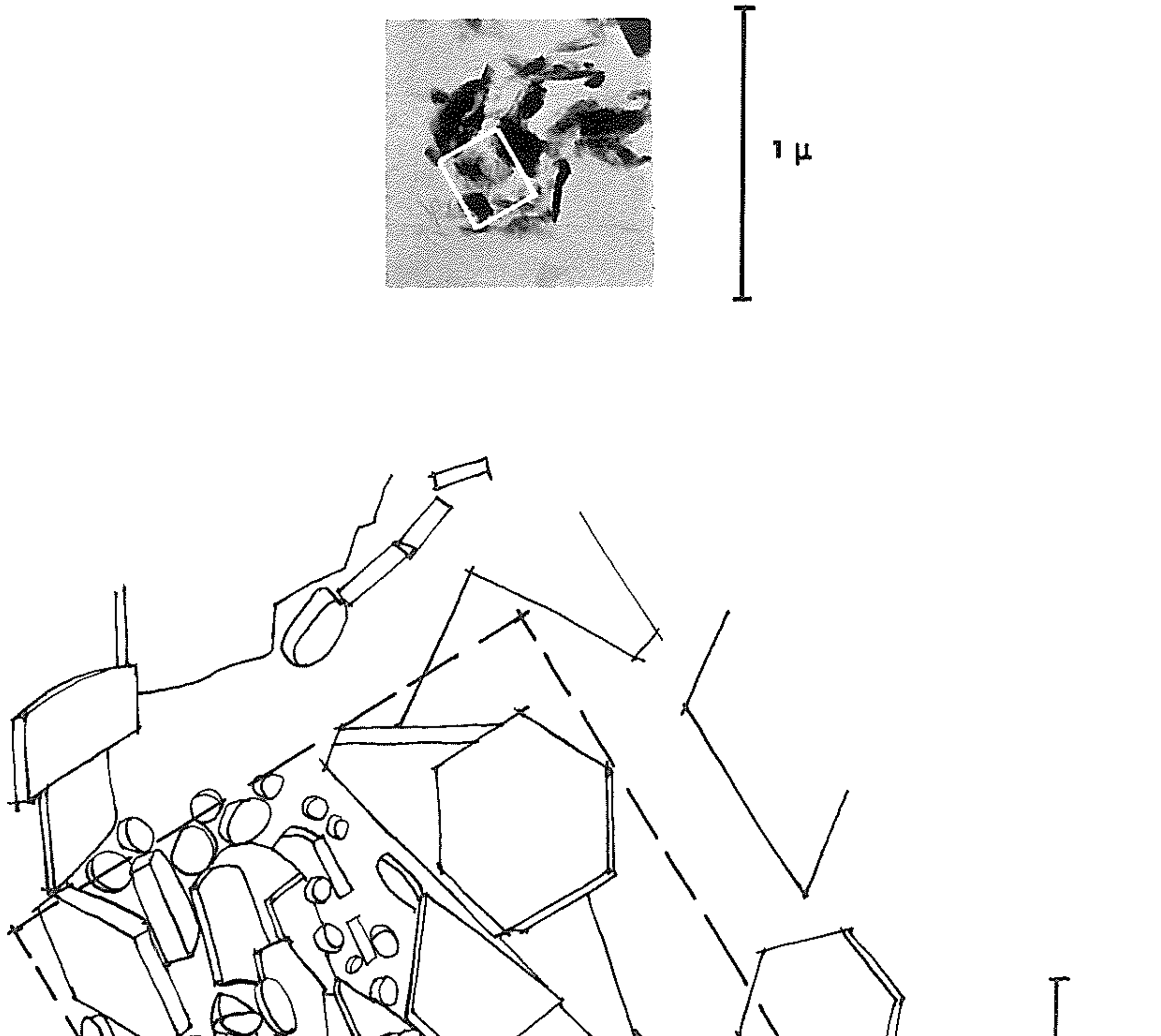


FIG. 33 Particle arrangement in clay aggregates. a) Skå-Edeby clay from 5 m depth. (Cont.) b) Lilla Edet clay from 6 m depth. c) Morjärv clay from 4 m depth. The particle arrangement in the small areas marked in the micrographs is reproduced schematically in the drawings.



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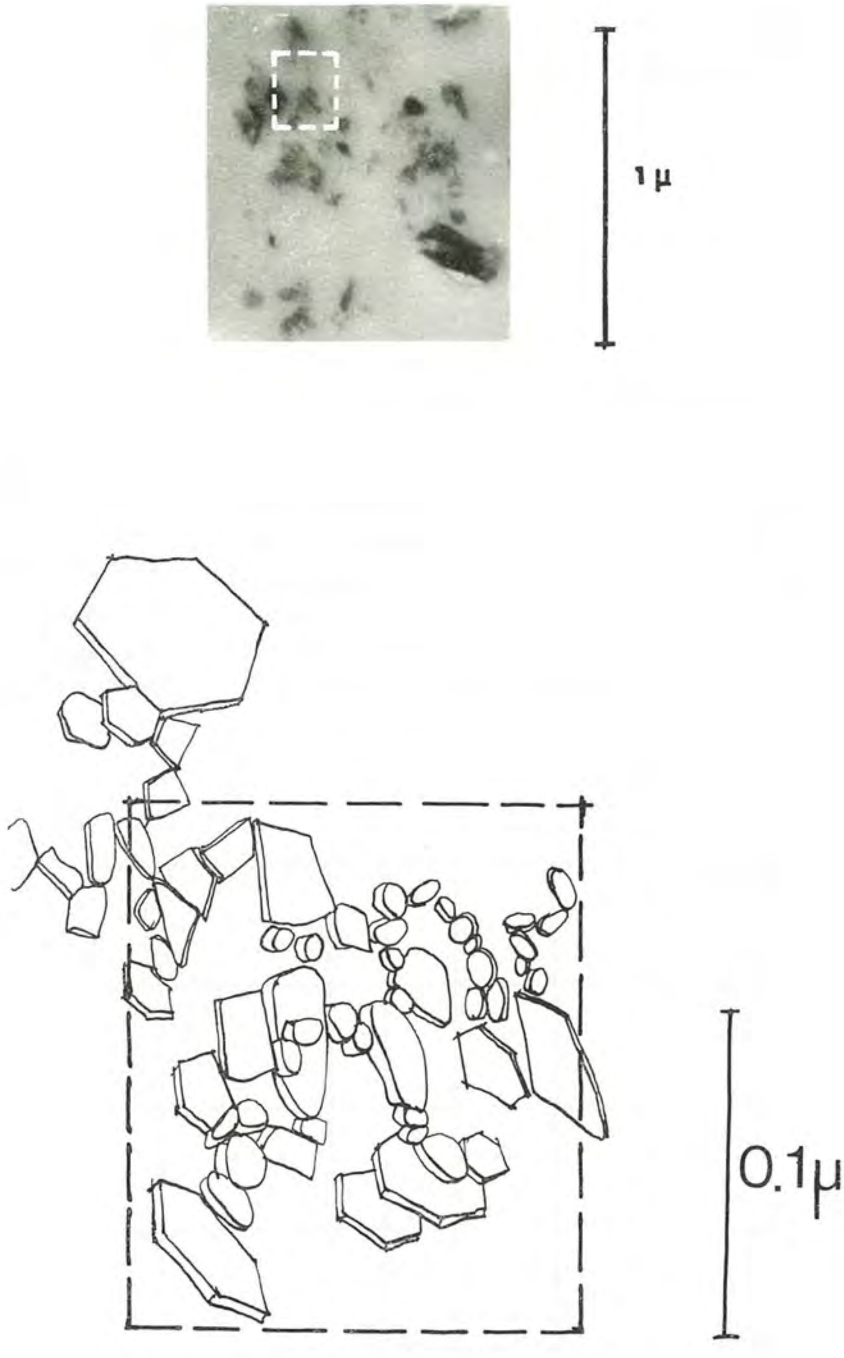


C

6 MICROSTRUCTURAL FEATURES AND PHYSICAL PROPERTIES

6.1 Permeability

By plotting the DARCY coefficient k for vertical flow versus the structural parameter P/T it can be seen that the microstructural pattern influences the permeability of the investigated clays (Fig. 34). The open particle network of marine clays is more permeable than the less porous system of particles in clays deposited in water of low salinity. This means that the P/T-values are representative of clay volumes of at least several cubic centimeters, although the studied sections were few and extremely small. Thus, larger voids and fissures did not have any decisive influence on the permeability of the specimens investigated.

The experimentally observed tendency for the permeability to be higher in the horizontal than in the vertical direction is explained by a microstructural anisotropy

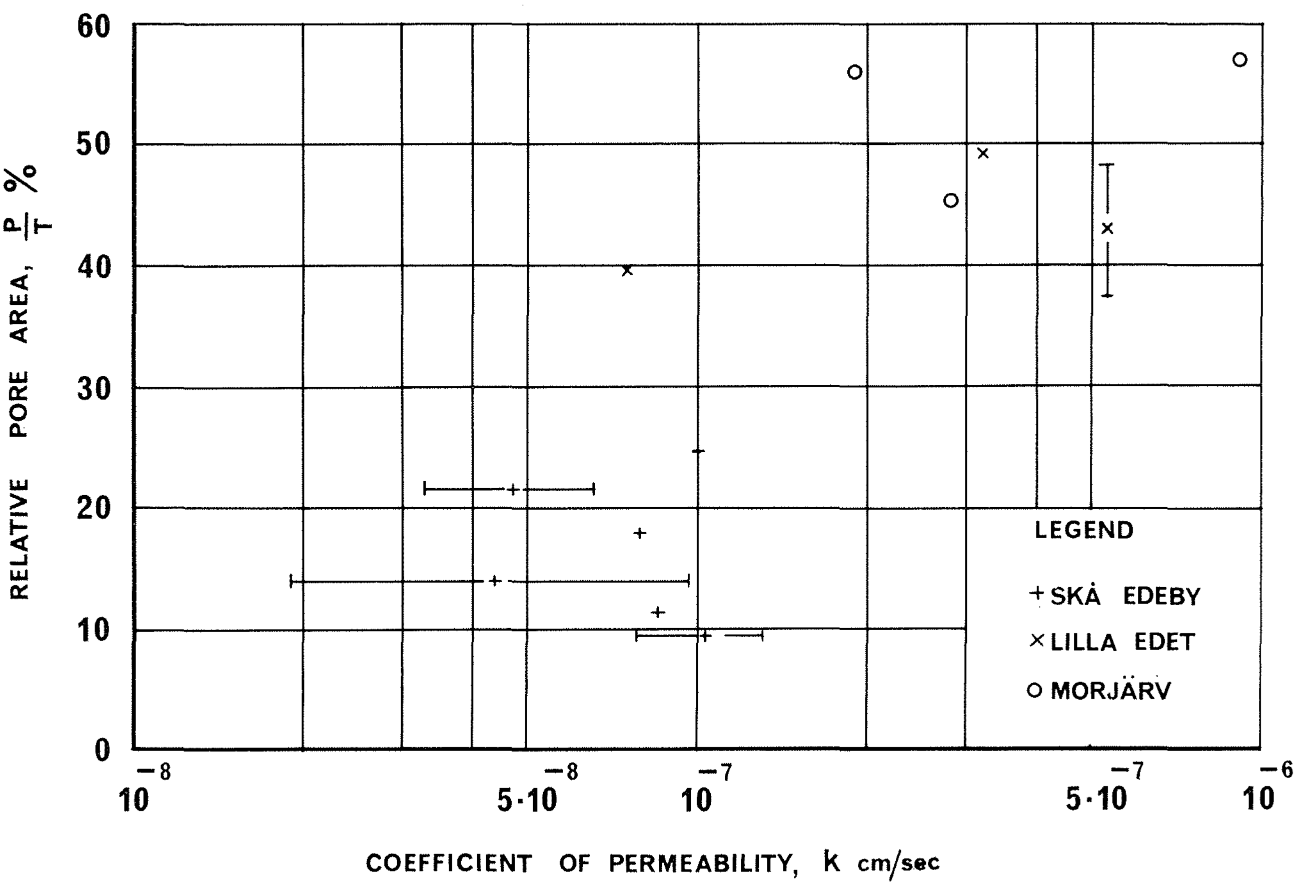


FIG. 34 The coefficient of permeability as a function of the microstructural parameter P/T.

caused by the orientation of large clay or silt particles or by the presence of very thin horizontal laminae of silt-sized particles.

According to HANSBO (1960) the pore water flow does not follow DARCY's law at small hydraulic gradients for layers from 5 to about 8 m depth at Skå-Edeby. For these layers HANSBO found that the rate of pore water flow was proportional to i^n where i is the gradient and n takes the value 1.0–1.6. When the hydraulic gradient exceeded a certain value there was a linear relationship between the flow rate and the hydraulic gradient. Probably the very small permeability at small hydraulic gradients is caused by the narrow passage-ways, as illustrated by the small P/T-values (9.4–13.8 %) and a_p-values (0.11–0.13 μm) for the clays at 5–7 m depths. Thus, for the clay from 2 m depth for which P/T=17.4 % and a_p=0.21 μm, HANSBO found no anomaly. The increased permeability at increasing hydraulic gradients in the clay with small pores may be due to microstructural changes or changes in the structure of the pore water. Pores may be enlarged and the structural order and viscosity of the adsorbed water may be reduced at a certain stress level.

Another phenomenon noticed by HANSBO may also be explained by the microstructural features. He found a tendency for gradual decrease of flow with time for a given gradient in his measurements. According to HANSBO this can be explained by a clogging effect by mobile particles. The micrographs of all the investigated clays support this hypothesis since they indicate the presence of loosely connected particles in the networks. This is especially characteristic of the marine Lilla Edet clay where considerable parts of the large aggregates are exposed to large pores. A number of peripheral particles have no linking function and can probably be moved by the flow. This is in fact supported by the results of percolation tests on artificially sedimented fresh- and salt-water deposited clays (PUSCH & ARNOLD, 1969). These tests showed a marked reduction of the permeability with time for the salt-water clay, while the fresh-water clay showed a much smaller permeability reduction. The time-dependent reduction of the permeability of the marine clays may also be the result of an improved state of order in the adsorbed water layers due to the diminishing number of ions. However, the resultant relatively small change of the permeable part of the characteristic large voids in the marine clays probably was of minor importance with regard to the permeability. It should be mentioned that electro-kinetic phenomena are associated with the percolation of clay systems. Thus, electric potentials are set up which counteract the flow and may be responsible for part of the time-dependent reduction of the permeability.

6.2 Deformation properties

6.2.1 Compressibility

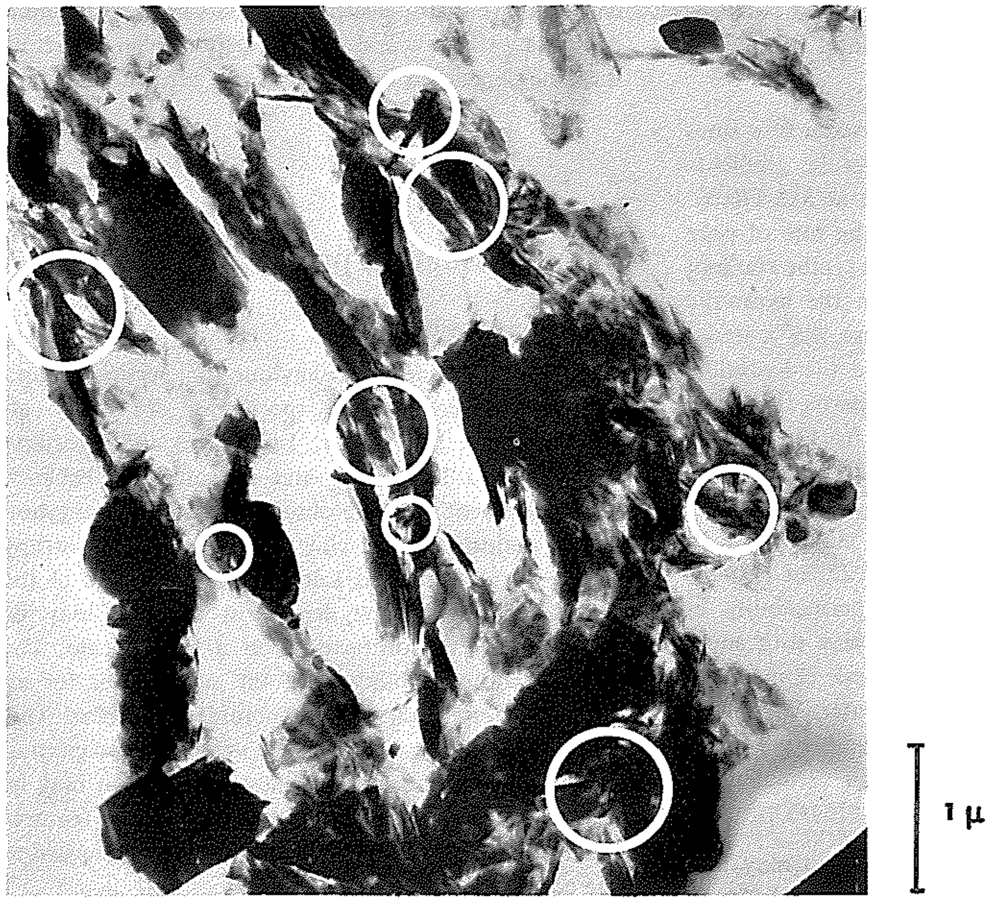
The almost identical behaviour of vertically and horizontally compressed specimens of nearly all samples from Skå-Edeby, Lilla Edet and Morjärv, as illustrated by the ε_{2v}–and ε_{2h}-values in Table 5, is in agreement with the observed random orientation of the majority of the clay particles. The anisotropy of the deepest situated Lilla Edet sample and of the samples from shallow depths in Morjärv may be explained by a stratification on a larger scale. The average compressibility is of the same order for all the clays despite the microstructural differences. This shows that factors like physico-chemical properties, organic content and particle size distribution may be more important than the microstructural features described by the parameters P/T and a_p, with regard to the compressibility.

The observed tendency for the highly organic clays to be successively and considerably deformed in the unconfined compression tests is compatible with the microstructural feature of inter-woven organic substance and clay particle network. The organic substance (especially of the fibrous kind) being easily deformed, may by stretched by the prolonged action of shear stress leading to discontinuities in the clay particle network and to considerable strength reduction at slow loading rates.

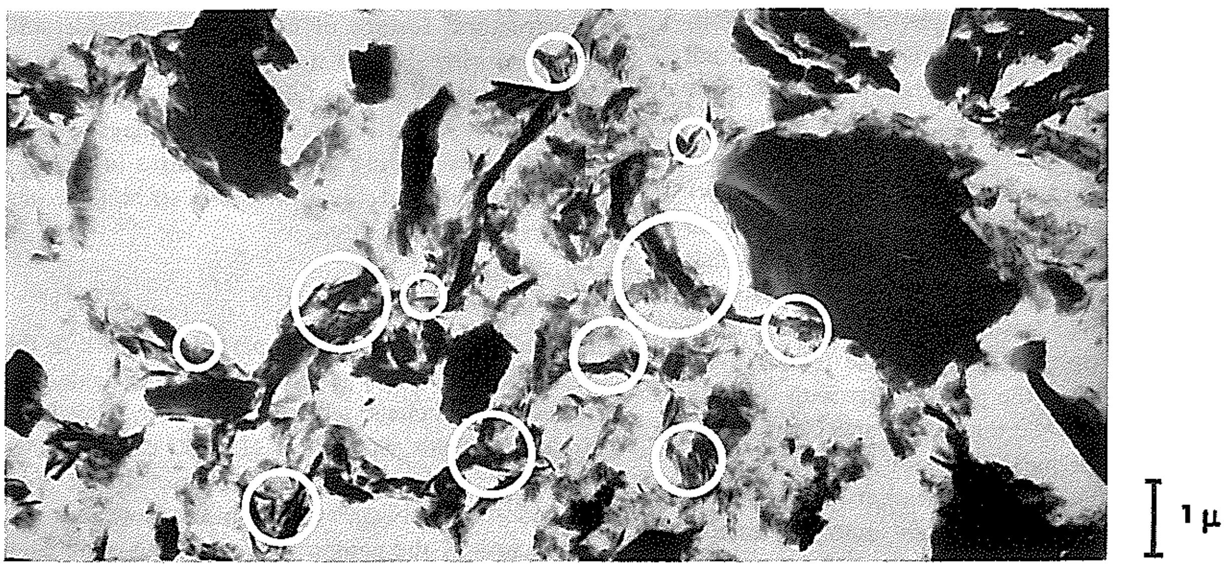
6.2.2 Consolidation processes

The detailed behaviour of the particle network in the compression of a clay specimen was investigated by a series of oedometer tests of Skå-Edeby clay from 2 and 8 m depth. Specimens of ⌀ 50 mm diameter were compressed to twice the preconsolidation pressure and to 128 N/cm². Small pieces cut from the central parts of the compressed specimens were used for microstructural analyses.

For the overconsolidated clay from 2 m depth the applied pressure, corresponding to twice the preconsolidation pressure, was 9 N/cm². The micrographs showed that the original microstructure was largely changed by internal deformations. Thus, the

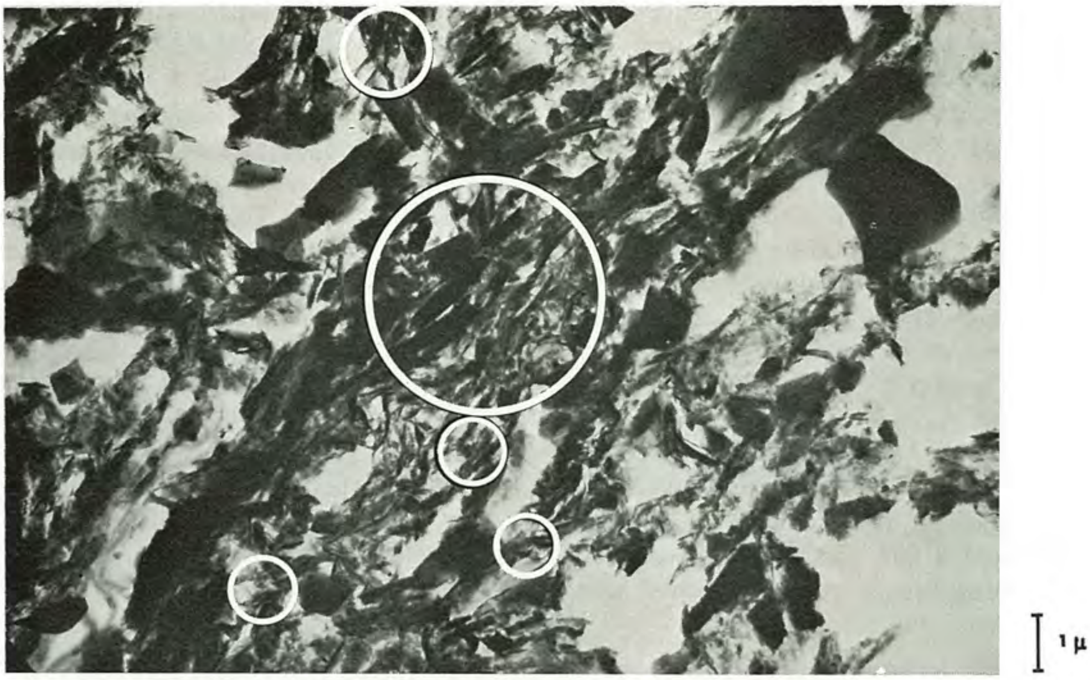


a



b

FIG. 35 Formation of domains (encircled) at double preconsolidation pressure. a) Skå-Edeby clay from 2 m depth. b) Skå-Edeby clay from 8 m depth.



a

b

FIG. 36 Formation of domains (encircled) and aggregate deformation at a consolidation pressure of 128 N/cm^2. a) Skå-Edeby clay from 2 m depth. b) Skå-Edeby clay from 8 m depth.

haphazardly arranged linking particles between the aggregates in the natural state were changed to oriented domain-like groups of particles in connection with the deformation, closing and opening of pores (Fig. 35 a). The same trend was found for the specimens from 8 m depth, the applied pressure being 8 N/cm² (Fig. 35 b). Compression to 128 N/cm² involved an intense distortion of the microstructural pattern. Thus, the number of domain-like particle groups was largely increased and the aggregates between which the oriented particles appeared were also deformed (Fig. 36 a and b). It was concluded that the groups of oriented particles were the sheared and deformed links originally connecting the aggregates. Certain domain-like groups of particles may have been formed by the microtome knife in the cutting operation but the undisturbed surroundings of the domains marked in the micrographs indicate that they are not artifacts.

Deformation processes during consolidation were also investigated by using a high voltage electron microscope in which internal particle movements in a drying drop of gel suspension were directly observed. The study was made by using the 1.5 MV electron microscope at the CNRS, Laboratoire d'Optique Electronique, Toulouse, France. The microscope, which was operated at 1000 kV, was equipped with a closed cell of the type shown in Fig. 37. The thin collodion-windows, which had a thickness of about 0.3 μm, were obtained from a 2 % solution of Parlodion [$C_{12}H_{16}$ $(ONO_2)_4O_4$, article No. 6552, Mallinckrodt Chemical Works, N.Y.] in butyl acetate. The collodion membranes were coated with SiO_2 by evaporation. The coating, which was about 0.07 μm thick, served as a protection against the humid atmosphere in the cell. Air pressure of about 1 torr existed in the cell which contained the drop of gel suspension under investigation. Immediately after the application of the cell with the drop of gel suspension in the microscope, micrographs were taken at one minute intervals. It was assumed, and this turned out to be the case, that the electronic radiation would cause a temperature increase and an evaporation, leading to internal deformations similar to those caused by mechanical compression. The gel was prepared by immersing 10 g Lilla Edet clay material in 100 ml distilled water. Hydrochloric acid was added to initiate flocculation, the concentration of the acid being extremely low.

Figure 38 a shows a representative part of the gel. The arrangement of particles was characterized by a system of aggregates connected by links or small groups of particles as in the case of the natural soft clays from Skå-Edeby, Lilla Edet and Morjärv. Fig. 38 b shows a schematic interpretation of a representative micrograph from one of the tests, the particle arrangement being characterized by a system of aggregates (No. 1–18) and small pores (drawn with full lines). The microstructural changes caused by the drying were investigated by comparing pictures taken at different time intervals. In the present case the structural changes caused by the drying could be measured by using one micrograph taken 6 minutes after the start of the investigation and one taken 2 minutes later. By comparing the micrographs, using the pores "A" as reference, the magnitude and direction of the movements could be determined. It was found that the deformation was mainly uniaxial in the direction of the arrows in Fig. 38 b within the investigated area. Clearly observable movements and deformations of aggregates and pores were marked by the dotted lines in the figure. Aggregate 14 was moved to the right without any measurable change of size or shape in connection with a decrease of the space of the inter-aggregate pores. This was also the case with aggregates 16 and 17, which were moved to the left without any measurable deformation of the aggregates, as can be concluded from the fact that the pores F, adjacent to aggregate 17, and G and H (locked in aggregate 17) were equally moved. The movement was accompanied by a compression of pore J. The structural changes were identified also in the original photographic plates. The investigation of the clay gel indicates that the characteristic microstructural pattern of a soft clay is established already in the stage when the settling clay particles are being deposited. It also indicates that the deformation of such a gel is characterized by a mutual movement of the aggregates in connection with a decrease in the interstitial pore space. The aggregates behave as fairly rigid bodies in comparison with the easily deformed link system.

It can be concluded from the microstructural investigation of the consolidation process that when the preconsolidation pressure is exceeded, the links break down to domains and the aggregates approach each other and find new positions of equilibrium. This is probably the main process during the period of primary consolidation. During this process, which may be rapid because of the high permeability of the porous system, the aggregates are also being compressed but their compression rate is low on account of their rigidity and low permeability. Compression of a laterally confined clay sample during the period of

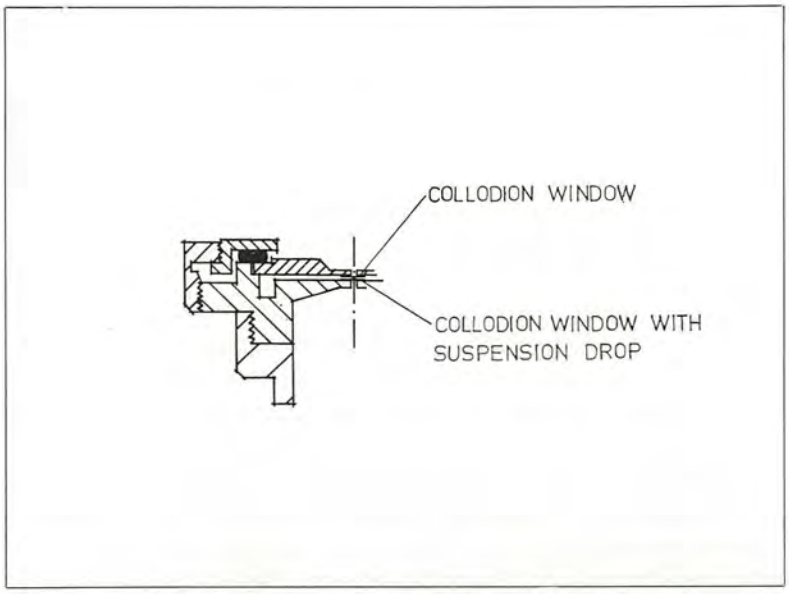


FIG. 37 Schematic section of the closed cell used for the electron optical investigation of dilute clay/water gels. The path of the electron radiation is indicated by the vertical broken line which is also the axis of symmetry of the partly shown cell section. Magnification about $\times 3$.

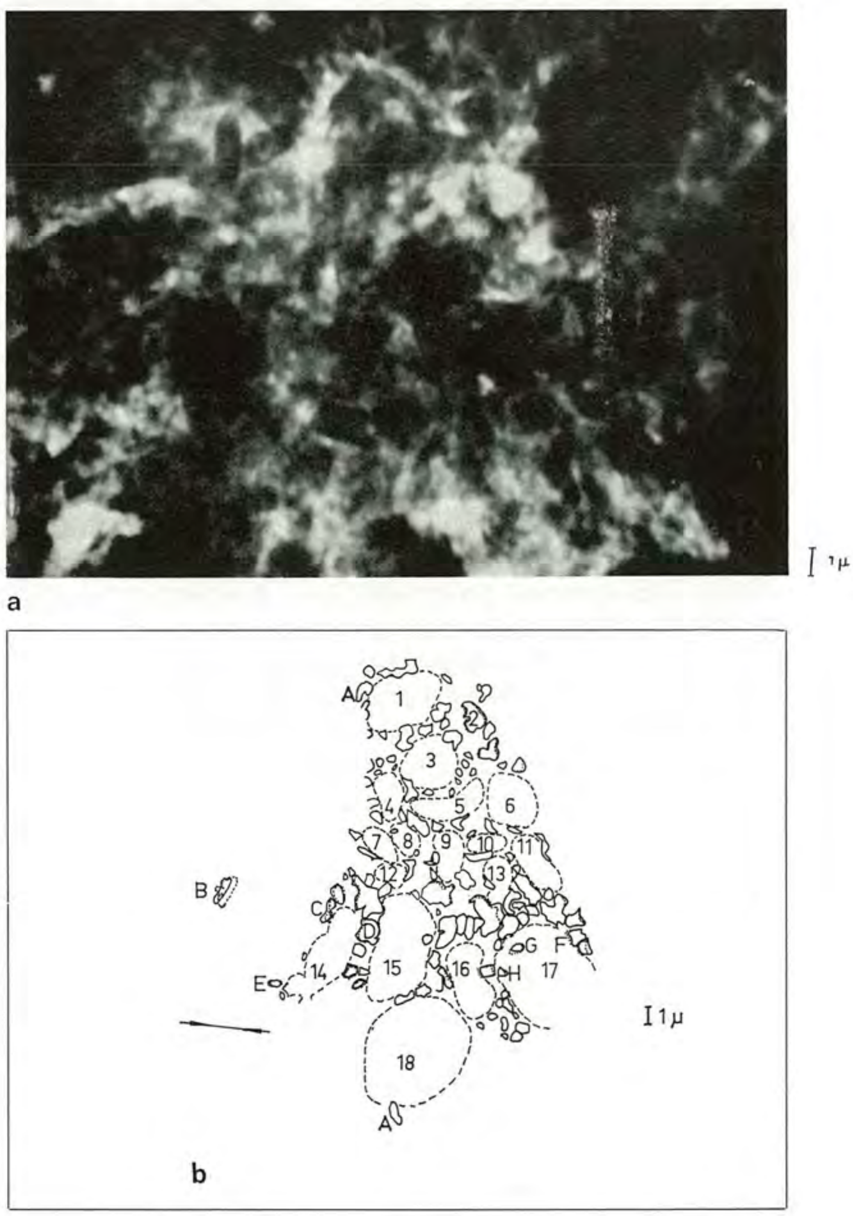


FIG. 38 a) Electron micrograph of a dilute clay/water gel. The arrangement of particles is characterized by a system of aggregates connected by links or small groups of particles. b) Schematic interpretation of microstructural changes at decreasing water content. Dotted lines show microstructural rearrangements.

secondary consolidation may well be dominated by a time-dependent deformation of the aggregates. The rate of deformation may be governed by the viscosity of the clay-water systems of the aggregates and of the domains and not by their permeability.

It may be asked why distorted links of the domain type are not the dominant microstructural features in soft normally consolidated clays. The reason may be that in such clays, which were only affected in nature by a slow loading rate caused by sedimentation, this rate was in phase with the delayed movement of aggregates and with link reformation, meaning that no state of broken links and temporary rigidity of the aggregates ever occurred. This hypothesis is supported by investigations of illitic clay rapidly consolidated in salt water by centrifugation, which showed that distorted links were numerous (PUSCH & ARNOLD, 1969). It is also supported by MEADE's statement that particle orientation recognized by X-ray studies is more obvious in rapidly compressed samples than in clay layers formed in nature.

6.3 Strength properties

6.3.1 Undrained shear strength

The undrained shear strength values, which are of the same order of magnitude for all the clays, are, except for the dry crusts, related to the overburden pressure. There is no simple relationship between these values and the microstructural parameters. Thus, it is seen that the Skå-Edeby clay, which has the smallest a_p- and P/T-values, has the smallest shear strength values despite the continuity and homogeneity of the particle network. This may be due to the observed greater interparticle distance in the links and aggregates in the Skå-Edeby clay than in the other clays.

6.3.2 Sensitivity

Very high sensitivity values were found only for the clays with the most open types of particle network as indicated by the P/T-values. Since the microstructural parameters are almost identical for the quick and normally sensitive clays from Lilla Edet it is obvious, however, that extreme sensitivity is not solely dependent on the microstructural constitution. Hence, physico-chemical processes, such as leaching (ROSENQVIST, 1955) or introduction of dispersing agents (SÖDERBLOM, 1966) may provoke a very high sensitivity although an open particle network may be a necessary prerequisite.

The sensitivity is highest for the layers with the most anisodiametric particles (the highest a/c-values) in the Skå-Edeby and Lilla Edet profiles. This relationship, which is consistent with earlier observations (PUSCH, 1962), can be explained by the open clay particle arrangement formed by the more anisodiametric particles or by a higher content of weathered particles. The weathering causes a partial splitting of particles by which the strength of the particles is reduced by remoulding. The splitting means that the a/c-value is higher in the weathered state.

6.3.3 Failure process

The detailed behaviour of the particle network under the influence of increased shear stresses was investigated by a series of consolidated, drained direct shear tests using the SGI (KJELLMAN) apparatus. Skå-Edeby clay samples from 2 and 8 m depth were tested at three consolidation pressures, σ_0, $2\sigma_0$ and 64 N/cm², σ_0 being the preconsolidation pressure. The specimens were sheared to failure, which corresponded to an angular deformation γ_f of 0.10 to 0.15 rad and, in separate tests, to the deformations $2/3\ \gamma_f$ and $\gamma_f/3$. The shear strength values at various pressures are shown in Fig. 39. Small pieces cut from the central parts of the sheared specimens were used for microstructural analyses. Since the influence of shearing on the microstructural pattern was similar for the samples from 2 and 8 m depth, only the tests of the first-mentioned clay layer will be reported and discussed.

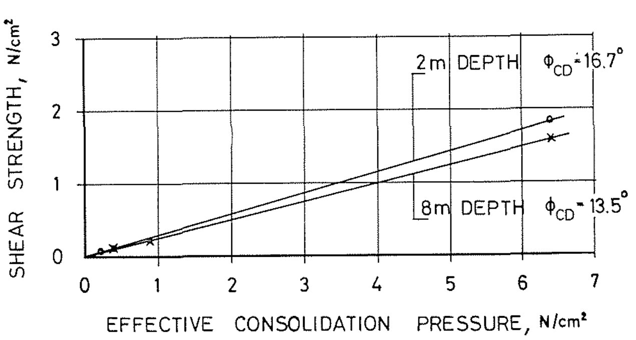


FIG. 39 Shear strength as a function of effective pressure at consolidated, drained direct shear tests. Skå-Edeby clays from 2 and 8 m depth.

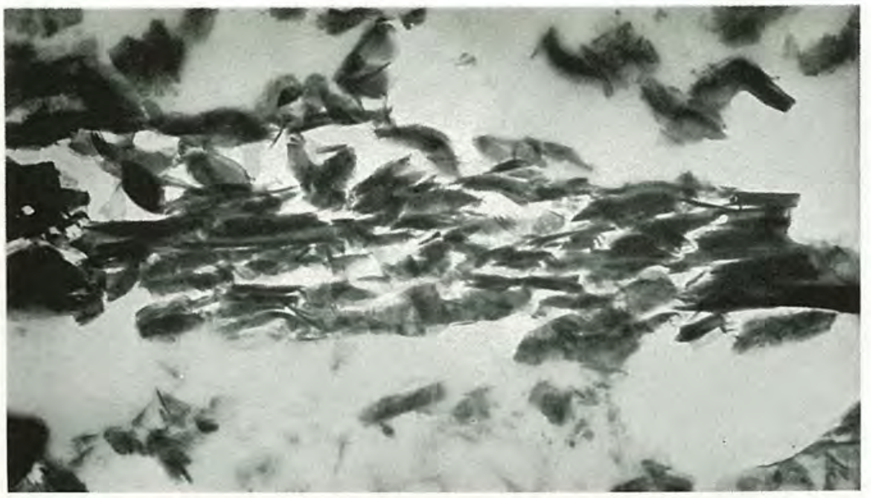


FIG. 40 Distortion of the microstructural network at failure. Micrograph of the central part of a specimen sheared at the preconsolidation pressure under drained conditions in the SGI shear apparatus. Skå-Edeby clay from 2 m depth.

For the clay from 2 m depth shearing to failure led to domain formation and particle orientation (Fig. 40) when the preconsolidation pressure was applied. Since the SGI shear device causes an angular deformation of the sample and not a definite shear zone as in the CASAGRANDE shear box, the particle orientation at failure is not very well expressed. It is dependent on the original position and orientation of the links as well as on the mutual movement of adjacent aggregates. At an angular deformation corresponding to $2/3\ \gamma_f$ the structural changes were less obvious but there was a tendency for the linking particles between aggregates and large particles to form domains (Fig. 41). At an angular deformation of $\gamma_f/3$ the influence of the shearing on the particle network was not detectable in the micrographs. They were identical

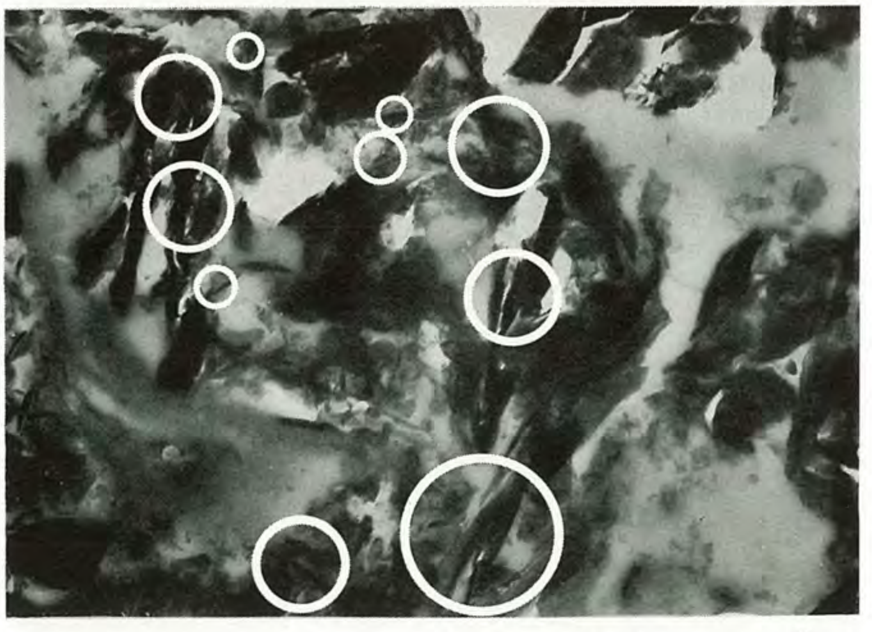


FIG. 41 Formation of domains (encircled) at an angular deformation of $\frac{2}{3}\gamma_f$ in the SGI shear apparatus. Skå-Edeby clay from 2 m depth sheared at the preconsolidation pressure.

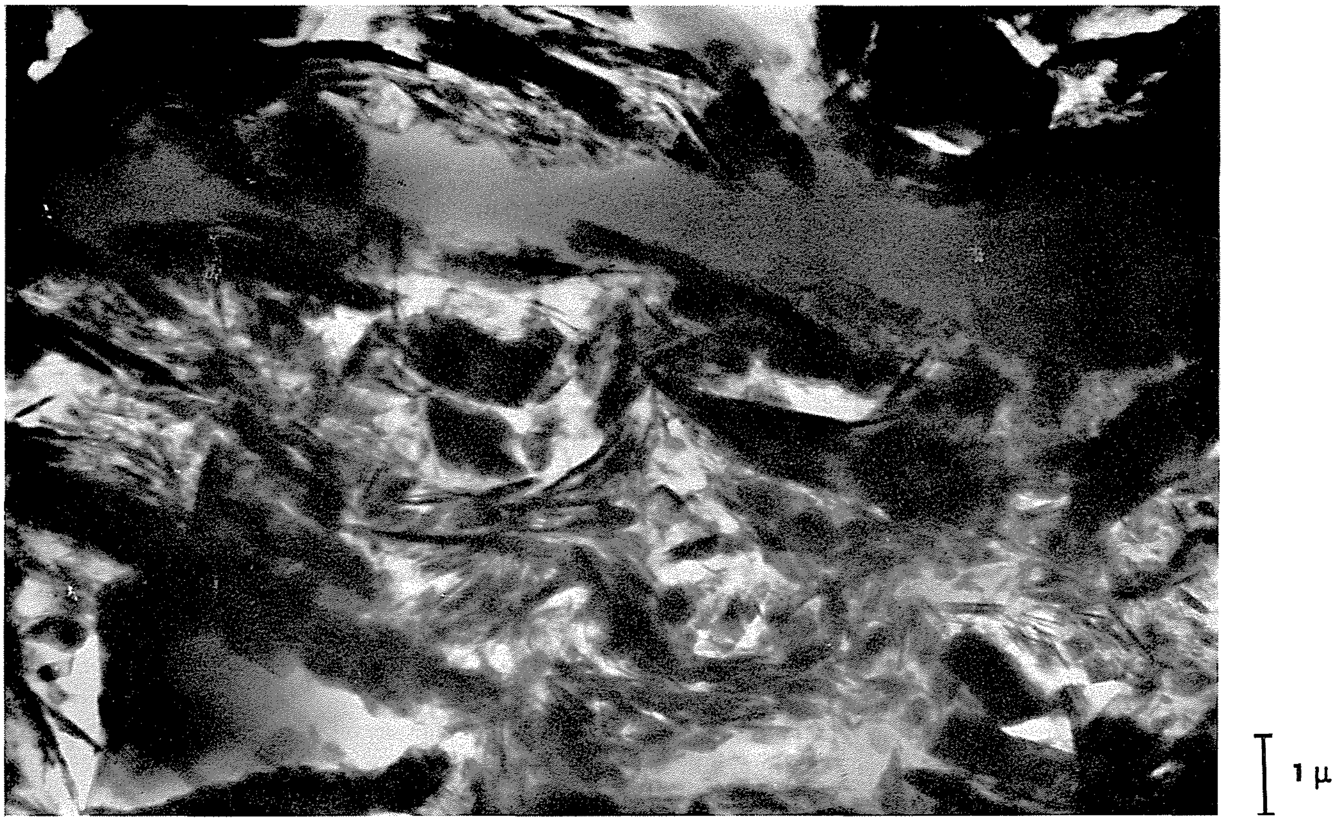


FIG. 42 Distortion of the microstructural network leading to domain formation and a general particle orientation at an angular deformation of $\frac{2}{3}\gamma_f$ in the SGI shear apparatus. Skå-Edeby clay from 2 m depth sheared at a consolidation pressure of 64 N/cm^2.

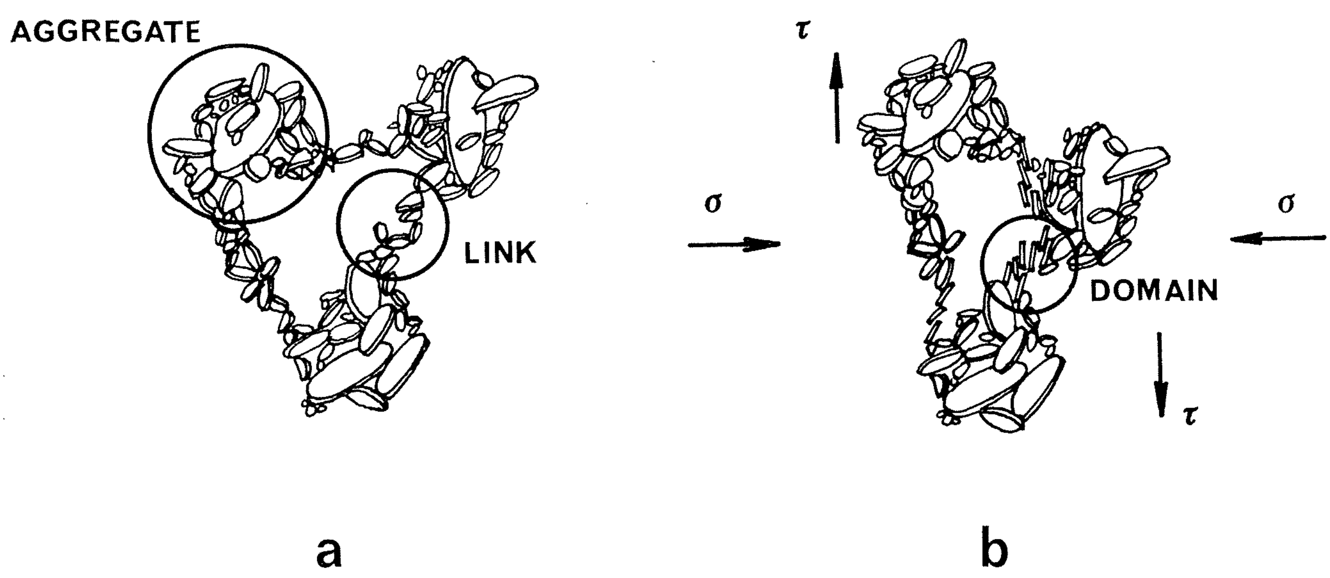


FIG. 43 Schematic picture of the failure process. a) Natural microstructural pattern. b) Breakdown of particle links resulting in domain formation (σ represents consolidation pressure and τ shear stress).

with the micrographs of the natural undisturbed clay material with reference to the frequency of domain-like groups of particles and to the structural parameters a_p and P/T.

At twice the preconsolidation pressure the same trend was found as for the series in which the preconsolidation pressure was applied but the structural changes were more obvious because of the increased consolidation pressure. At a pressure of 64 N/cm^2 shearing to failure gave a complete breakdown of the links as well as of the aggregates. At an angular deformation of $2/3\ \gamma_f$ the distortion was also considerable. Several aggregates were still intact but most links were broken down (Fig. 42). Structural changes at $\gamma_f/3$ were also observed but they were probably caused in the consolidation phase. As in the case of microstructural changes resulting from consolidation, the undisturbed surroundings of the domains, marked in the micrographs, indicate that they are not artifacts.

It is concluded that the mechanism of shearing of soft clays is that the aggregates, which form strong units, are mutually moved in connection with a failure of their links. The motion of the aggregates involves a large deformation of the connecting links, which results in a parallel orientation of the linking particles (Fig. 43). The failure mechanism may also furnish a phenomenological explanation of the concept "residual strength". This strength may correspond to the state where the links are inactive through large internal deformations. In this state the strength, which is reduced to 30–50 % of the peak shear strength value, may be caused by dilatancy as well as by viscous effects when the aggregates are approached during shearing. A further distortion in the form of remoulding brings about a breakdown of the aggregates leading to lower bulk strength values.

The appearance of domain-like bodies is in reasonable agreement with the hypothesis of TROLLOPE & CHAN for the microstructural behaviour at high shear stresses. It is also consistent with the microstructural changes at remoulding observed by EMERSON and with KORINA's and FAUSTOVA's observation of flow structures of oriented clay in pores and around grains in moraines. The author's description of microstructural changes is also in agreement with the hypothesis of TERZAGHI & PECK if coarse grains and rigid aggregates are assumed to behave in a similar way.

The observed domain formation under the influence of an increasing pressure in the rapid laboratory consolidation shows that determination of true shear strength parameters (HVORSLEV, 1937) from consolidated shear tests at different degrees of overconsolidation should not be applied to undisturbed clay since irreversible microstructural changes are caused in the consolidation phase.

It should be mentioned that the failure process observed in the Skå-Edeby clays did also take place in soft marine clay loaded axially to failure in unconfined compression tests (PUSCH, 1970).

6.4 Discussion

The consolidation as well as the shear processes seem to be governed by the aggregates. They give evidence of behaving as fairly rigid bodies up to a certain stress level which may be dependent on various physico-chemical phenomena. It is expected that the small interparticle distance in the aggregates gives rise to a highly ordered state of the water leading to a high cohesion of the aggregates. This hypothesis has been tested by the author in an exploratory investigation of the molecular behaviour of water at various water contents, using natural and organic-free specimens of the Skå-Edeby clay from 2 m depth. Similar investigations, mainly of montmorillonitic minerals and kaolinite, have been made by PICKETT & LEMCOE (1959), BLAINE (1961), GRAHAM *et al.* (1964), WU (1964) and others.

The basic idea is that flow is equivalent to stress relaxation following MAXWELL's concept for visco-elastic bodies. If the same definition is applied to a liquid, the problem of evaluating its flow properties in terms of viscosity is that of measuring molecular mobility. It can be made by determining the duration of molecular orientation in a magnetic field as in the nuclear magnetic resonance technique (NMR). For a review of the basic principles of NMR the reader is referred to BLOEMBERGEN *et al.* (1948) and ANDREW (1955). The application of NMR technique to clay/water problems has been described by JACOBSSON (1968) who used the same Varian DP 60 spectrometer equipment as the author (see also MYRBERG, 1968).

The resonance condition was achieved by keeping the oscillating radiofrequency magnetic field constant while slowly varying the main polarizing magnetic field through the resonance frequency. The voltage induced in the receiver coil formed the modulated RF-signal which was amplified and recorded. The main field was modulated at low frequency for lock-in amplification, so that the recorded signal was proportional to the derivative of the absorption curve. The temperature variation of the water cooling system as well as that of the spectrometer room were within $\pm 0.1°C$ during the recording of the spectrum. In most cases the field swept by the recorder was 2.5 G and the scanning time 25–50 min. The main magnetic field was kept at 3760 G throughout the study corresponding to a proton resonance frequency of 16.0 MHz.

Theoretically, in the case of statistically independent spins, the NMR absorption curve is true Gaussian as approximated by protons in free water. In clay/water systems a line broadening is produced by the coupling of the water to lattice OH-groups, and eventually to hydrophilic groups in organic adsorbed substances. The lattice protons, on the other hand, are strongly coupled to the clay lattice and give rise to a very broad signal. The line width of the absorption curve is essentially a function of the spin-spin coherence time T_2. In certain viscosity ranges the relaxation mechanism may, however, also influence the coherence time and therefore the line width. The interpretation of the absorption curve has to be made by separating the various contributions to the total absorption band: the very narrow band due to water molecules in secondary adsorption sites, the broad water band due to molecules in primary adsorption sites and the very broad main band due to lattice hydroxyls and eventual organic material present. For this purpose the recorded curve is expanded in a series of oscillator functions, the ground state of which represents a Gaussian distribution while the overtones describe the type and magnitude of the deviation from the statistical independence of the spins contributing to the spectral band. A typical proton resonance narrow band shape obtained for the Skå-Edeby clay is shown in Fig. 44.

The band shape analysis was carried out by use of a least square approximation in a computer program adapted and developed at the NMR Research Group, Royal Institute of Technology, Stockholm.

Natural material as well as material treated with hydrogen peroxide for elimination of the organic content were used. It was dispersed ultrasonically and freeze-dried and water was added by introducing water vapour in the dry material, the determination of the water content being made after the end of the NMR study. The coherence time T_2 has been plotted against the water content in Fig. 45. The plots of T_2 within the lower moisture range show that the molecular mobility of the water at clay mineral surfaces is much smaller than that of free water and that it increases with increasing distance from these surfaces. This is in agreement with results of similar investigations by PICKETT & LEMCOE, WU and others.

FIG. 44 Proton resonance wide line of natural Skå-Edeby clay from 2 m depth. Water content about 5 %.

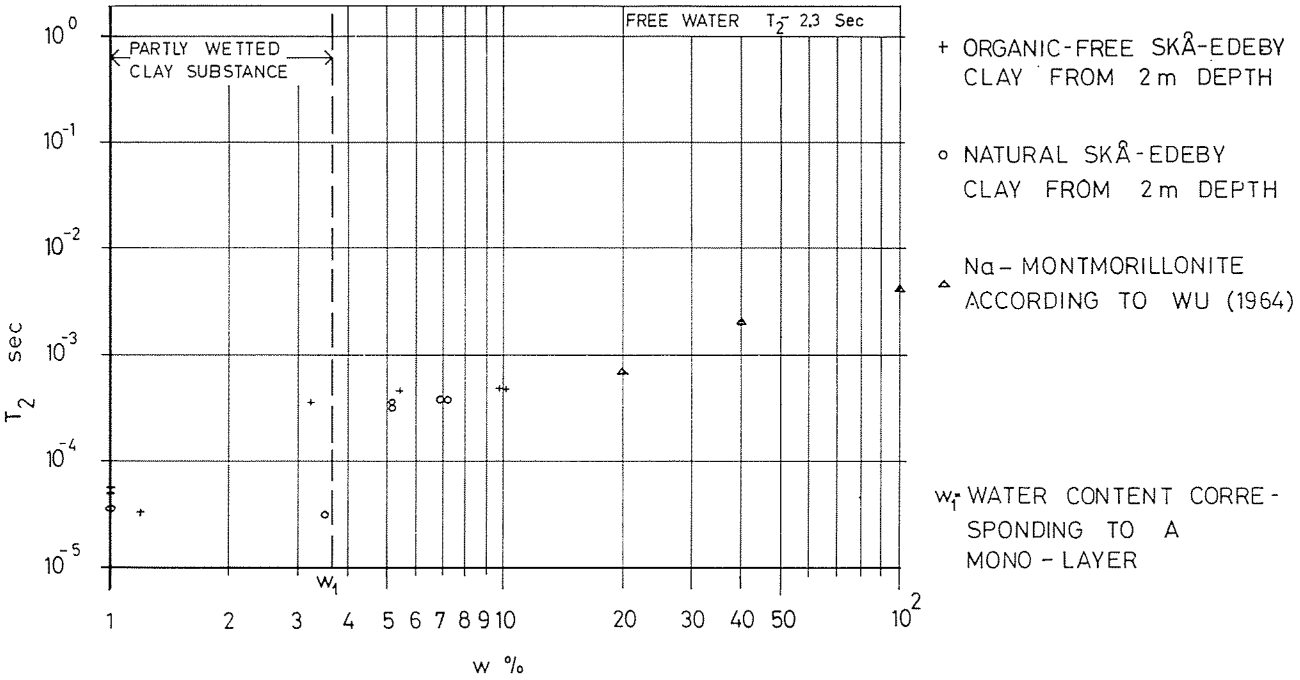


FIG. 45 Spin-spin coherence time T_2 as a function of the water content. Skå-Edeby clay from 2 m depth.

No attempt has been made to correlate T_2 with the viscosity of the adsorbed water since the influence on absolute values of paramagnetic contaminants is unknown. However, the increased viscosity of the adsorbed water on clay mineral surfaces as compared with free water may be illustrated by ROSENQVIST'S (1959) self-diffusion experiments with illite. He concluded that the average water viscosity is 153 centipoises at $w = 10$ % and 24 centipoises at $w = 30$ %. This means that the viscosity of the intra-aggregate water in the clays investigated by the author is very much higher than that of free water. Naturally the strong atomic bonds between the closely located minerals in the aggregates also contribute to their rigidity.

As can be seen from Fig. 45 the influence of the organic substance on molecular mobility was rather small, which indicates that it did not contribute largely to the fixation of water molecules. However, the property of water fixation of the organic substance may have been influenced by the clay preparation technique.

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