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## THE LOW-PLASTIC CLAY FROM GODTHÅB, GREENLAND

# AN X-RAY MINERALOGICAL INVESTIGATION

BY

EJNAR JENSEN

WITH 5 FIGURES AND 1 TABLE IN THE TEXT

KØBENHAVN
C. A. REITZELS FORLAG

BIANCO LUNOS BOGTRYKKERI A/S

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#### Abstract

A glacial sediment from Godthåb (Greenland) had very low plasticity and coherence in spite of its high content of particles smaller than  $2\,\mu$ . A sample was separated quantitatively into fractions of sand, silt and clay, and the mineralogical composition of each fraction was determined chiefly by X-ray powder methods. The composition of the sample was in fair agreement with that of the surrounding rocks. As a whole the sample was poor in quartz but rich in oligoclase, hornblende, biotite and vermiculite, the last mentioned presumably an alteration product of biotite. The low plasticity and coherence may be ascribed to the unusually large content of oligoclase in the clay-size fraction, to the sharp-edged irregular shapes of the coarse particles and to the exceptionally well-crystallized character of even the smallest clay particles.

#### INTRODUCTION

In 1962 The Danish Geotechnical Institute made an investigation of the foundation conditions in the vicinity of Godthåb in Greenland. The argillaceous material met with here, a fresh-water sediment probably settled originally in a glacial lake or inlet, had rather anomalous physical properties.

Estimated by convenient short-cut methods from its plasticity and its ability to cohere when rolled between the hands the material should be fairly coarse-grained. However, a particle-size analysis carried out for control in the laboratory showed on the contrary, that it was a rather fine-grained sediment. About one fourth of the material consisted of particles smaller than  $2\,\mu$ . (Ellen Louise Mertz and Niels Stokholm, private communication 1962.)

As the low plasticity and coherence might be due to the mineralogical composition, an attempt has been made to identify all the mineral components and to determine their percentages by quantitative methods. The sample analyzed was drawn at a depth of four meters from the sedimentary bed. It contains practically no organic matter but about 2 per cent of calcium carbonate, and the pH of the material suspended in distilled water was 7.65.

#### TECHNIQUE

In order to simplify the analysis the sample was first separated quantitatively according to particle size into a clay fraction with particles smaller than  $2\,\mu$ , a silt fraction with particles between  $2\,\mu$  and  $20\,\mu$  and a sand fraction with particles ranging from  $20\,\mu$  to ca. 1 mm, the largest particles observed in the material.¹) The fractionations were carried out by elutriation in 0.002 molar sodium pyrophosphate solutions using a special elutriator (Jensen and Møller Hansen 1961) and the results are shown in the upper parts of the table on page 11.

For particular purposes parts of the clay-size fraction were further separated into fractions above and below the  $0.6\,\mu$ -limit. At so low limits the elutriator works too slowly, and the separation was therefore carried out by centrifugation. By this method however separation was not quite complete, but it may be stated, that about fifty per cent by weight of the clay-size fraction consists of particles smaller than  $0.6\,\mu$ . Finally parts of the sand fraction were separated into a light and a heavy fraction by means of tetrabromo-ethane (specific gravity  $2.9\,\mathrm{g/ccm}$ ). The heavy fraction amounted to ca. 6 per cent of the total sand fraction. However the fractionation is not claimed to be complete.

The identification and quantitative determination of the various minerals have been based on X-ray powder methods. The powder diagrams were registered on films according to the Debye-Scherrer method as well as by a diffractometer. The two methods supplement each other. For identification purposes film recordings are convenient, except at very low diffraction angles where the diffractometer gives better results. For quantitative determinations the diffractometer method is also superior, and therefore all low-angle reflections and those used for quantitative determinations were registered with the diffractometer.

The photographic diagrams were taken with Co K $\alpha$ -radiation in circular cameras (diameter 57 mm and 191 mm). The samples were fixed to thin rods of glass with Canada balsam and rotated in a hydrogen atmosphere during the exposure (4 to 8 hours).

<sup>1)</sup> International Society of Soil Science classification system.

The diffractometer diagrams were registered with a General Electric apparatus (type XRD-3) using Cu K $\alpha$ -radiation, and the irradiated specimens were flat, about 1 cm high, 0.1 cm thick and 1 to 5 cm long. Instead of a simultaneous registration as in the photographic method the reflections are here registered one by one by means of a Geiger tube and recorded continuously on a chart as a series of peaks superposed on a more or less smooth background. From the charts (fig. 4) the relative reflection intensities can be read directly as a function of 2  $\theta$ , the double reflection angle. For practical reasons the peak positions on the charts in figure 1 and figure 2 are not given in angular units but in Ångström units of the interplanar spacings  $d = \lambda/2 \sin \theta$  (Bragg's law).

#### Identification of the minerals.

The photographic diagrams<sup>1</sup>) were very rich in lines, and as expected for such a material, many of the lines coincided more or less, a fact complicating the interpretations.

The diagrams were analysed by comparison with diagrams of pure minerals the presence of which might be expected. Beginning with the diagram of the sand fraction which was least complicated and with the component easiest to detect the corresponding series of lines were marked in turn on the film with ink. In this way quartz was easily detected. The felspar group studied next was represented almost exclusively by a plagioclase. Perhaps also a small amount of microcline was present. This could not be identified with certainty, nor could the possibility be excluded. On the other hand the scarcity of microcline and other potassium felspars was confirmed by the following micro-chemical test. A few hundred grains of the sand fraction were etched in hydrofluoric acid vapor and then placed in a solution of sodium cobaltinitrite. By this treatment the grains of potassium felspars will be stained yellow (see for instance Toyborg Jensen and Krogh Andersen, 1955) but here only a few grains reacted.

Assuming the plagioclase to be one of the low-temperature modifications, which seems most likely here, an estimate of its composition can be obtained from the almost linear relation existing between the angular difference  $2\,\theta_{111}$ – $2\,\theta_{1\bar{1}1}$  and the ratio anorthite/(albite + anorthite) within the range from zero to about 30 per cent of anorthite (SMITH 1956). In the present case the angular difference agreed with a content of 23 per cent of anorthite, and the plagioclase has been classified therefore as an oligoclase.

<sup>1)</sup> The films are not shown, because most of the fine details would be lost during the reproduction.

The third mineral identified was a hornblende. Only the five most distinct lines were visible, but detection was facilitated by comparison with a diagram of the heavy mineral fraction of the sand, in which hornblende was predominant. Having marked the lines belonging to the above mentioned components, only a few very weak lines were unexplained, and the coincident lines were given double marks in agreement with the fact that they appeared relatively stronger or broader than the corresponding single lines in the diagrams of the pure minerals.

Once detected in the sand fraction, quartz, oligoclase and hornblende could be detected also in the silt and clay fractions, but the two fine-grained fractions likewise contained 2 or 3 types of clay minerals (phyllosilicates).

In the diagram of the clay fraction (calcium-saturated) the four innermost reflections corresponded to interplanar spacings of 15 Å, 10 Å, 8.3 Å and ca. 7 Å respectively. The two first are certainly 1.-order basal reflections of clay minerals, the third at 8.3 Å is due to the horn-blende already detected, the fourth reflection at ca. 7 Å may be partly a 1.-order partly a 2.-order basal reflection of clay minerals. The identifications have been based largely on these basal reflections, and how they reacted on specific chemical and phycical pretreatments of the clay material as registered by the diffractometer.

Clay-mineral reflections are generally poor, but the basal reflections will be improved, if the platy crystals are oriented with their bases preferentially parallel with the irradiated surface of the sample. The samples used for these analyses were therefore suspended in water and allowed to settle by the force of gravitation on small plates of perspex which, after evaporation of the water at 25 to 30°C, were set up for measurements on the diffractometer.

Figure 1 shows diagrams of the clay fraction in which the sodium ions adsorbed during the particle-size fractionation have been exchanged for calcium ions by treatments with a dilute aqueous solution of calcium chloride at room temperature. In 1A the sample was given no further treatment. In 1B the sample was moistened with glycerol. In 1C the sample was heated for 8 hours at ca. 650° C, and in 1D the sample was boiled in concentrated hydrochloric acid for 5 minutes. From the three first diagrams (1D will be discussed later) the following conclusions have been drawn:

The position of the 10 Å peak is practically unaffected by glycerol, and by heat. This indicates that the reflection is due to a mica. The peak at 15 Å shifts to 18 Å by addition of glycerol. When heated it moves back to coincide with that of the mica at 10 Å. These observations prove, that it belongs to one of the swelling clay-mineral types,—either a montmorillonite or a vermiculite. Which of the two was determined

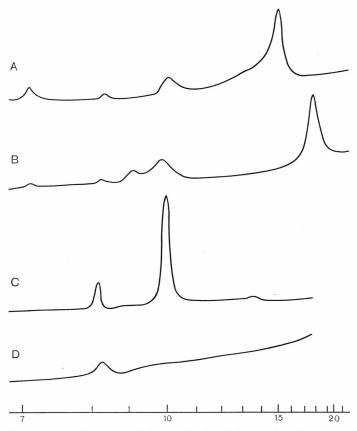


Fig. 1. Diffractometer diagrams of differently treated samples of the clay fraction. A: Calcium-saturated. B: Calcium-saturated and moistened with glycerol. C: Calcium-saturated and heated for 8 hours at 650°C. D: Boiled in 37 per cent hydrochloric acid for 5 minutes. Abscissa interplanar spacings in Ångström; ordinate relative intensity.

by further investigations of the lattice expansion in glycerol. In the case of montmorillonites this expansion is almost independent of the type of cations adsorbed. However that is not the case for the vermiculites. When saturated with calcium ions the first-order basal reflection is here about 15 Å, and by addition of glycerol it increases in some cases to 17–18 Å just as is the case with the montmorillonites. On the other hand, if a vermiculite is saturated with magnesium ions the first-order reflection lies as a rule at ca. 14 Å, and it is slightly or non-affected by glycerol (Walker 1961). The diagrams in figure 2 show the effect of glycerol on a magnesium-saturated sample of the clay fraction from Godthåb. The basal peak is now fixed at 14.2 Å. Therefore the conclusion was drawn that it belongs to a vermiculite.

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In figure 1A the second-order basal reflection of the calcium-saturated vermiculite can be seen at ca. 7 Å. As expected this peak was diplaced to about 9 Å by the glycerol (fig. 1B), but there is still a small peak left at ca. 7 Å. By heating to 650° C the 7 Å peak vanishes and a new, very small peak seems to appear at ca. 13.6 Å (fig. 1C). The simultaneous disappearance of the 7 Å peak and the development of a peak at 13.6 Å by heating may indicate that an iron-rich chlorite is

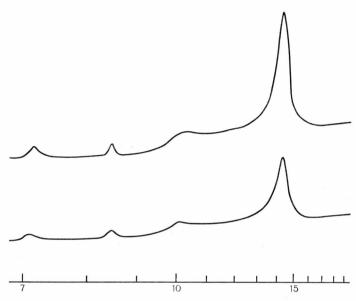


Fig. 2. Diffractometer diagrams of the magnesium-saturated clay fraction. Above: No further treatment. Below: Moistened with glycerol. Abscissa interplanar spacings in Ångström. Ordinate relative intensity.

present. On the other hand, the experimental evidence of the peak at 13.6 Å is somewhat questionable, and it is therefore possible that the 7 Å peak should be ascribed to a kaolinite rather than to a chlorite.

Some further information on mica and vermiculite were drawn from a powder photograph. On the ordinary film diagrams the many strong lines from the quartz and oligoclase obscured the much weaker lines from the mica and vermiculite. In order to obtain a more favourable ratio between the two mineral groups and thus weaken the influence of disturbing lines, the fraction with particles smaller than  $0.6\,\mu$  was used. The diagram of this fine-grained sample proved, that the mica was a biotite, i.e. a trioctahedral mica, and that the vermiculite was also trioctahedral.

Further evidence for this classification was obtained in another way. It has been generally accepted, that trioctahedral layer-lattice silicates are considerably less stable than those of the corresponding dioctahedral types. The mica and the vermiculite found here were very unstable. Boiling in concentrated hydrochloric acid for 5 minutes destroyed their crystalline structure completely as illustrated in fig. 1 D. Similarly, the acid-base titration curves in fig. 3 show that the two minerals are very sensitive to dilute bases. By a pure neutralization process the foreward and backward titration curves should be practically coincident. Here

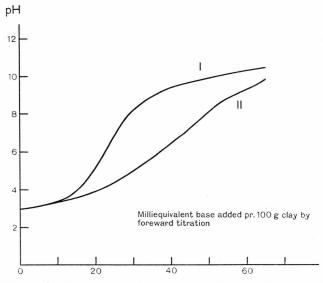


Fig. 3. Acid-base titration curves of the clay fraction. I.: Foreward titration with 0.1 normal sodium hydroxide. II.: Backward titration with 0.1 normal hydrochloric acid.

they diverge widely indicating that some material has broken down, and since quartz, oligoclase and hornblende are not affected appreciably by dilute acids or bases, it must be the mica and/or vermiculite which have decomposed.

Some trace minerals were detected by means of microscopic analyses of the heavy mineral fraction of the sand. A few hundred of the heavy grains were separated into groups under the microscope according to their appearance (shape, colour etc.) and identified by comparison with grains of pure heavy minerals. Besides the main components, hornblende and mica already detected, the heavy fraction contained a pyroxene and some few grains of garnet, epidote and magnetite. The results were checked as far as possible by examination of the x-ray diagram of the non-separated grains, and it was found that the pyroxene was probably a diopside.

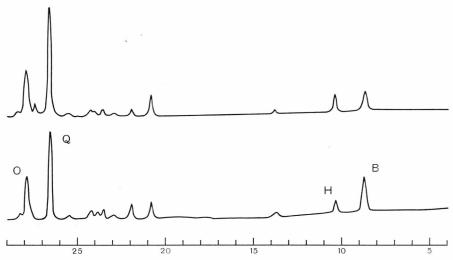


Fig. 4. Diffractometer diagrams of the sand fraction (above) and of a standard mixture containing 50 per cent oligoclase, 25 per cent quartz, 5 per cent hornblende and 20 per cent biotite (below). The peaks used for quantitative comparisons are indicated O, Q, H and B respectively for the four components. Abscissa 2  $\theta$ ; ordinate relative intensity.

#### Quantitative determinations.

In the diffractometer diagrams the peak areas above the back-ground line represent the integrated reflection intensities, and at suitable experimental conditions the area of a well defined peak will be proportional to the percentage of the corresponding mineral component in the sample. Hence, if we compare this area with the corresponding area from the diagram of a standard mixture containing a known percentage of the pure mineral, we can compute the content in the unknown sample.

One condition should be stated. Of the irradiated sample only the outermost ca. 0.1 mm layer contributes essentially to the reflecting power. Of the particles within this layer only a fraction of the order of  $10^{-3}$  are (by random orientation) oriented in such a way that their reflections can be caught by the Geiger-tube. Hence, the particles must be very small in order to obtain sufficient reflecting particles to represent the entire sample statistically, on an average not larger than 5 to  $10~\mu$  (Alexander et al. 1948). For the more coarse-grained materials investigated here (sand, silt and standard minerals) this was obtained by grinding in ethanol for an hour in a mechanical boron-carbide mortar.

The quantitative determinations were made as follows: Preliminary estimates of the contents were obtained by comparing the peak areas for the samples with those for the pure minerals. By these estimates it was possible to prepare mixtures with comparable contents of the pure

$Particle ext{-}size$	distribution	and	mineral	composition	of	sediment		
from Godthåb, Greenland.								

Fraction	Sand	Silt	Clay	Total				
Particle size range $(\mu)$	ca. 1000–20	20-2	2-0					
Per cent by weight	46.3	26.4	27.3	100				
Minerals in per cent by weight								
Quartz	30-35	20-24	4-6	ca. 20				
Oligoclase	45-50	40-45	25 - 30	ca. 40				
Microcline (?)	0-5	0-2	0	ca. 2				
Hornblende	5-10	4-6	3-5	ca. 5				
Diopside	1-2							
Garnet	trace							
Epidote	trace							
Magnetite	0.2							
Biotite	5-10	15 - 25	25 - 40	ca. 20				
Vermiculite	0	5-10	15-30	ca. 10				
Kaolinite or								
Iron-rich chlorite (?)	0	0	0-2					

minerals. Each particle fraction and each standard mixture was then measured under identical conditions and the contents recalculated. Two of these diagrams are shown in fig. 4. Each measurement was repeated once or twice in order to obtain rough estimates of the experimental accuracy. For the clay fraction both oriented and non-oriented samples were measured.

The content of diopside was calculated from its relative number of grains in the heavy mineral fraction, whereas the magnetite was separated quantitatively from a large sample of the sand by means of a permanent magnet and weighed. The contents of garnet and epidote could be stated only as traces.

The results of the analyses are listed in the table on page 11. The contents are given for each mineral and each particle-size fraction as a concentration range indicating the probable limits of accuracy. The approximate composition of the total sample shown in the last column has been calculated with due considerations to these limits and to the particle-size distribution given in the upper part of the table. It should be added that more exact values can be obtained, but then far more fundamental investigations would be required.

#### Discussions.

Two thirds of the sample consist of quartz, oligoclase and hornblende with oligoclase as the predominating component. The percentage of quartz decreases greatly with decreasing particle size as is normally the case for this mineral in sediments. The percentage of oligoclase and of hornblende decrease also with decreasing particle size but less, and the clay-size fraction is still very rich in oligoclase. Note that oligoclase is practically the only felspar present. It is possible that the sand and silt fractions contain some microline, but not more than one or two per cent by weight. Diopside, garnet, epidote and magnetite occur in small amounts. They have been analyzed only in the sand fraction, but they probably occur likewise in the silt fraction.

The last third of the sample consists of clay minerals, almost exclusively biotite and trioctahedral vermiculite. The contents of these minerals increase considerably with decreasing particle size (the vermiculite was found only in the silt and clay fractions). The clay fraction contains a third clay mineral. Due to the low concentration it could not be identified with certainty, but it is probably either a kaolinite or an iron-rich chlorite.

The crystalline structures of the biotite and the trioctahedral vermiculite are very similar, and it seems reasonable to assume that the latter is an alteration product of the former. Further studies on partly weathered pieces of the biotite (if available) might probably allow a definitive conclusion to be drawn. It should be added that Walker (1950) has detected such a biotite-vermiculite transformation in Scottish soils, and concluded that the weathering product of the "clay biotite" here is normally a vermiculite.

If the above assumption as to the origin of the vermiculite is correct then the original parent rocks must have been very rich in biotite. Now, in contrast to the rather worn particles characterizing the sands from Danish moraines, all particles in the Godthåb sediment were very irregular and sharp-edged. As the worn appearance of the former is most probably due to attrition during the long transport, it also seems reasonable to believe that the latter has not suffered from transport, but that it has been derived from rocks in the immediate vicinity of the bed.

According to Henning Sørensen (private communication, 1963) the gneisses from the vicinity of Godthåb are rich in plagioclase, hornblende and biotite. In addition they contain layers of amphibolite (composed of hornblende, diopside and plagioclase), mica schist (with muscovite, biotite, garnet and quartz) and small lenses of hornblendite, often fitted with biotite-rich border zones in which vermiculite has been found. In broad outline this composition agrees fairly well with that found for the sample analyzed. It is true that the content of biotite plus vermiculite in the sediment is high compared with that of the surrounding rocks, but this may be due to partial fractionations according to the shapes and sizes of the particles during the transport and settling of the material.

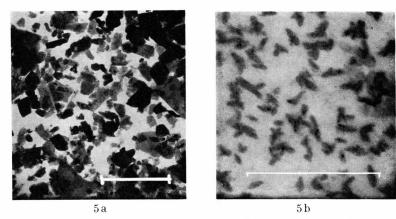


Fig. 5. Electron micrographs. a: Godthåb clay. b: Danish moraine clay (Brøndby-øster). Length of scales: One micron.

The low plasticity and coherence in spite of the large content of fine-grained material are probably due to several factors. No doubt the sharp-edged, irregular faces of the particles already mentioned make the whole material feel coarser.

Plasticity and coherence depend on the content of fine-grained material and, in the absence of organic matter as here, they depend especially on the platy clay-mineral particles. The content of these minerals within the clay-size fraction of the Godthåb sample is low, only about 50 per cent in contrast to the 70 to 90 per cent found in the clay fractions of Danish moraines, and consequently the entire sample will be relatively less plastic and less coherent. In this connection the well-crystallized character of the most fine-grained fraction may also be an important factor. Figure 5 a shows an electron micrograph of the fraction with particles smaller than  $0.6\,\mu$ . The opaque particles are without doubt oligoclase and/or quartz, the more or less transparent plates are probably biotite and/or vermiculite. The well-crystallized character is striking, especially if the picture is compared with that of a typical Danish clay shown in figure 5 b.

In many respects the Godthåb sediment resembles that of a powder obtained by mechanical grinding of rocks (rock flour, bergmjöl). Deposits of that type are found in other localities, e.g. in the northern parts of Scandinavia.

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