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**Development of clay characterization
methods for use in repository design
with application to a natural Ca
bentonite clay containing a redox
front**

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Clay Technology AB, Lund

December 1990

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DEVELOPMENT OF CLAY CHARACTERIZATION METHODS FOR USE
IN REPOSITORY DESIGN WITH APPLICATION TO A NATURAL CA
BENTONITE CLAY CONTAINING A REDOX FRONT

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This report concerns a study which was conducted for SKB. The conclusions and viewpoints presented in the report are those of the author(s) and do not necessarily coincide with those of the client.

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SUMMARY

Natural smectite clays in the form of "true" bentonites formed from volcanic ash, or resulting from in-situ weathering of rock, are available in very large quantities in southern Europe as well as in the rest of the world, and they are suitable for a number of sealing options in repositories, both as tightening component of sand/clay backfills and as highly efficient buffer for embedment of canisters, as well as for fracture sealing. The price and quality, in terms of smectite content and type of smectite, vary considerably and an optimum choice of clay for use in repositories has to be based on quantitative quality data. This requires characterization of the clay material for which a test scheme has been worked out. It comprises determination of the *granulometrical*, *chemical*, and *mineralogical compositions*, as well as of certain *physical properties*.

Recent research shows the importance of the type of smectite for the longevity of buffers in repository environment, beidellite being less favorable and saponite superior to montmorillonite, which is the most common smectite species. The test scheme hence includes means of distinguishing between various smectite minerals. The influence of accessory minerals on the chemical integrity of both the smectite and the canister material requires identification also of such minerals, for which the scheme is useful as well.

The report summarizes the various test procedures and gives data from application of the scheme to samples from a natural Ca bentonite containing a redox front. This study suggests that a significant part of the iron in the clay fraction is in the form of Fe^{2+} in octahedral positions of the montmorillonite of unoxidized natural clay and that it is converted to Fe^{3+} on oxidation. Part of the iron is probably in the

form of $\text{Fe}^{2+}\text{Fe}^{3+}$ hydroxy compounds that give the un-oxidized clay its bluish color, while they can be assumed to be transformed to yellowish FeOOH forms on oxidation.

1 INTRODUCTION

In 1987, a first attempt was made to put together a standard procedure for characterization and indexation of clay materials for use in repositories for final storage of radioactive wastes (1). Since the smectite content has currently been considered as the most important property of what is termed buffers and backfills, quantification of the content of this kind of minerals was focussed on in the previous study, in which special effort was made to investigate how accurately the smectite content could be determined by applying Reynold's XRD technique. The major outcome was that a slightly changed version of this technique could be recommended for standard testing but that it should be further refined and tested.

In a detailed characterization of natural bentonite deposits in Busachi, Sardinia, and Hamra, Sweden, it was found that rather large variations in mineral composition could be harboured by typical XRD diagrams of smectite-rich soil samples, which made it questionable whether true mixed-layer sequences can really be identified by Reynold's method (2). Further doubts have appeared later, a recent example being offered by Elen Roaldset at the fall meeting in Stockholm, 1990, of the Nordic society for Clay Research. At this meeting it was demonstrated that different investigators had arrived at smectite contents in the interval 60 - 90 % of one and the same clay material, and all this has led to the conclusion that the accuracy of even sophisticated XRD techniques is insufficient for detailed quantifi-

cation of the smectite content. Also, the involved work and cost is not in proportion to the accuracy.

A different policy for characterization of smectitic clays has therefore been proposed and it is based on combining four simple methods for quantification of the smectite content, and the content of accessory minerals of importance, as well as on the determination of major physical properties. XRD is still applied but in a simpler form, primarily to determine the major adsorbed cation, and to identify dominant accessory minerals. Chemical analyses of the clay fraction serve to indicate the major type of smectite, while the physical property data are used for comparison with the corresponding material data of standard smectite clays, primarily MX-80 Na bentonite and Ca/Mg Moosburg bentonite.

The present study summarizes the proposed laboratory test program and also application of the test program to Moosburg clay, with the additional aim of determining possible changes of the clay when a redox front passes through it. The detailed laboratory procedure is not given in this report, while it will appear in the comprehensive manual that will form part of the forthcoming "Clay Bible".

2 PROPOSED LABORATORY TEST PROGRAM

2.1 General

The program is primarily intended to be applied to potential candidate clay materials for classification purposes. Simpler, less comprehensive tests are sufficient for current quality checking of a large clay material shipment originating from a certain bentonite deposit and processed at a certain industrial plant.

2.2 Program for characterization of smectitic clays

A. *Granulometric composition*

Purpose:

The grain size distribution gives a good picture of the content of clay minerals since they only appear in the clay fraction (minus 2 μm). The compaction properties of the material can be estimated by the character of the entire grain size curve, from which one can conclude if the material is suitable for backfilling purposes.

Techniques:

Sieving, and sedimentation tests using the hydrometer or the Atterberg pipette techniques are recommended (SI standards). Automatic techniques, like the Coulter counter method, are applicable but calibration by use of one of the afore-mentioned techniques should be made.

B. *Chemical composition*

Purpose:

The chemical composition reveals the relationship between silica, aluminum, magnesium and iron, which helps to identify the dominant smectite type: i.e. montmorillonite, beidellite, saponite, or nontronite. It also provides a basis for identifying accessory minerals of importance, like calcite and sulphides.

Techniques:

Spectroscopic (ICP) analyses applying dissolution in LiBO_2 melt should be made as standard tests, while EDX tests, using transmission or scanning electron microscopy, are recommended for special purposes. Tests of the complete material, as well as of the clay fraction (minus $2\mu\text{m}$) are made.

Element analyses are made with respect to the presence of SiO_2 , Al_2O_3 , Fe_2O_3 , MgO , CaO , K_2O , Na_2O , S and LOI, the latter representing the ignition loss (LOI). All data are given in weight percent units.

A typical set of element data for montmorillonite corrected with respect to the LOI is:

SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	K_2O	Na_2O	S
65	20	6	5	1.8	1.8	0.3	0.1

Typical reference smectite formulae, excepting the adsorbed cations, are:

- * Montmorillonite: $(\text{OH})_4\text{Si}_8(\text{Al}_{3.34}\text{Mg}_{0.66})\text{O}_{20}$
- * Beidellite: $(\text{OH})_4(\text{Si}_6\text{Al}_2)\text{Al}_{4.44}\text{O}_{20}$
- * Saponite: $(\text{OH})_4(\text{Si}_{7.34}\text{Al}_{0.66})\text{Mg}_6\text{O}_{20}$
- * Nontronite: $(\text{OH})_4(\text{Si}_{7.34}\text{Al}_{0.66})\text{Fe}_4^{3+}\text{O}_{20}$

C. *Mineralogical composition*

Purpose:

The main purpose is to determine the content and type of smectite minerals as well as the nature and amount of accessory minerals of importance, of which K-feldspars, calcite and sulphide minerals are of particular interest.

Techniques:

X-ray diffraction analysis, and determination of the cation exchange capacity and the Atterberg consistency limits are made for standard characterization. Transmission electron microscopy of microstructurally intact soil in combination with EDX analyses is used for checking purposes.

XRD

Tank's identification scheme is applied to samples prepared both of the bulk material (finer than 74 μm) and of the clay fraction (3). Sedimented specimens are prepared, and heating to 250 and 600°C as well as glycol treatment are applied.

Major characteristics when applying CuK_α radiation (Ni filter) are:

* *Kaolinite*

Regular basal sequence of peaks of 7.1, 3.57, and 2.39 Å. Crystalline character lost on heating to 600°C

* *Chlorite*

First- and third-order reflections are 14.0 and 4.7 Å, respectively. Only partial dehydration at 600°C; strong solubility in warm dilute hydrochloric acid

* *Hydrous mica (illite)*

Sequence of basal reflections at 10, 5. and 3.3 Å; (020) reflection at d=4.5 Å

* *Smectites*

(001) peaks at about 12 to 15 Å in air-dry condition. 15 - 17 Å peaks on glycol treatment indicates Ca and Na as the respective major cation. Heating to 250°C yields collapse to 10 Å basal spacing.

Beidellite is distinguished from montmorillonite by not expanding to more than 14.1 Å on exposing Na-saturated clay to 100 % RH environment. On such treatment, montmorillonite gives a basal spacing of more than 16 Å

* *"Mixed-layer minerals"*

Identification of high (001) spacing and regular series of sharp higher order reflections indicate regularly interstratified minerals. Non-integral series of basal plane reflections indicate randomly interstratified minerals. Altered peak shape and intensity by cation saturation indicates segregated minerals; if layers

expand on glycol treatment and collapse on heating they are termed smectite

* *K-feldspars*

The following peaks are characteristic:

d-value (Å)	Intensity (100 max)
4.23-4.21	60
3.31-3.29	100
3.26-3.23	40-100
2.99	50
4.21-4.04	60-70 (K/Na)

* *Calcite*

The only easily identified reflection is the very strong characteristic peak for the d-distance 3.035 Å

* *Iron sulphide (pyrite)*

The following peaks are characteristic:

d-value (Å)	Intensity (100 max)
2.423	65
2.709	85
1.633	100

While this sort of interpretation of XRD spectra primarily serves to give a qualitative picture of the mineral assemblage, a rough semi-quantitative evaluation can be made by using certain peak height ratios. The uncertainty is considerable, however.

Cation Exchange Capacity

The cation exchange capacity should be determined only for the clay fraction. Chapman's method for determination of the total cation exchange capacity and of the amounts of adsorbed cations should be applied because of its relative simplicity. This technique involves exchange to NH_4^+ and repeated washing with ammonium acetate solution. The supernatants are saved for analysis of the initially adsorbed cations.

Washing with isopropyl alcohol to remove excess NH_4^+ ions is made and NaCl solution finally used for transfer to Na saturated conditions. The supernatant is analysed with respect to NH_4^+ for evaluation of the total cation exchange capacity.

Typical CEC-values, expressed in terms of meq/100 g are the following:

Material	CEC
Zeolites	300-100
Vermiculite	150-100
Montmorillonite	100 -70
Chlorite	47 - 4
Hydrous mica	40 -10
Kaolinite	15 - 3
Feldspar, quartz	1

Naturally, soils composed of two or several major clay minerals can give any CEC value in the range of about 10 to 100, which demonstrates that additional techniques must be applied to make identification possible.

Atterberg Consistency Limits

The hydration capacity of minerals is manifested by the three consistency limits: w_L - the liquid limit, w_P - the plastic limit, and w_S - the shrinkage limit. For estimation of the smectite content the liquid limit serves as a valuable indication. The following w_L -data can be taken to be representative:

Clay	w_L , %
Na montmorillonite (60-95 %)	250 - 600
Ca montmorillonite (60-95 %)	80 - 120

It is obvious from the fact that there is a considerable difference in w_L of sodium- and calcium saturated clay that variations in clay content (minus 2 μm fraction) makes estimation of the smectite content very uncertain. Hence, w_L data should only be used for this purpose if the clay fraction has been separated and tested, or if the type of adsorbed cation has been determined by XRD analysis.

Transmission Electron Microscopy

Transmission electron microscopy (TEM) of dispersed clay makes it possible to make a rough estimate of the relative amounts of kaolinite, hydrous mica and smectite based on the diagnostic morphology of these clay minerals (4). Applying TEM to suitably embedded clay, the general fabric can be identified and cementing agents revealed, and element analyses (EDX) performed (Fig.1). Such studies are very helpful in derivation of the genesis of the soil. Embedment is suitably made by stepwise saturation with ethylene alcohol and plastic monomers and polymerization at 60°C (4).

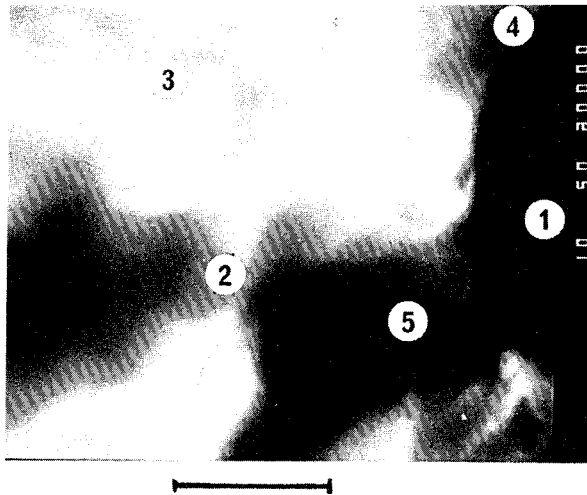


Fig.1. TEM picture of ultrathin (500 Å) section of Na montmorillonite clay with 100x100 Å² areas selected for element analyses. 1) dense silica object, 2) montmorillonite stacks, 3) soft montmorillonite gel consisting of thin stacks, 4) dense silica-rich object, 5) dense montmorillonite stack agglomerate

Bar represents 1 μm

Scanning Electron Microscopy

Scanning electron microscopy (SEM) of samples that are mechanically fractured after air-drying, freeze-drying or critical point drying and coated with carbon (or gold if element analyses are not going to be made) serves to illustrate the general microstructure with less tedious preparation than TEM requires. The resolution power (50-100 Å) is considerably lower than in TEM analyses but acceptable for many routine studies. EDX element analyses (spot or area) can readily be made. The procedure described in (5) is recommended for practical application.

Additional

A very detailed determination of the amounts of accessory minerals can be made by utilizing differences in specific gravity of the minerals. Thus, heavy minerals of particular interest, such as sulphides, can be determined by separation in liquids with different, suitable densities, using centrifuges. Such techniques are very tedious and not of practical use except for checking the homogeneity of smectitic buffers to which small amounts of graphite (sp.g. 2.09-2.23 g/cm³) have been added.

D. *Physical properties*

Purpose:

The hydraulic conductivity and swelling pressure as functions of the bulk density are very helpful in estimating the content of smectite minerals, and they give a direct measure of the usefulness of the material for buffer and backfill purposes. The heat conductivity and capacity are important as well and need to be tested for characterization of candidate materials.

Techniques:

Hydraulic conductivity and swelling tests, as well as rheological tests should be made by using water saturated material. Thermal properties may be of interest also at lower degrees of saturation but the testing procedure is very complicated and the evaluation difficult, and it is therefore recommended that they be determined using saturated material.

A standard preparation procedure must be applied in order to get reproducibility and for all the tests the following method is proposed:

1. Drying at 60°C for 3 days of 500 g samples
2. Careful crushing and sieving to yield a maximum grain size of 1 mm
3. Application of the air-dry material in a swelling pressure oedometer or in a shear box to the *dry densities* 1.1, and 1.6 g/cm³, with subsequent saturation with distilled water. For smectite-poor materials a backpressure has to be applied for water saturation. The height of the sample should be about 2 cm both for the hydraulic and for the creep testing
4. Homogenization for 1 week under confined conditions before testing starts

Hydraulic Conductivity

The hydraulic conductivity is determined by percolating the water saturated clay sample with distilled water (cf. Fig.2). The percolation should be conducted at constant, room temperature for at least 1 week at a water pressure that must not exceed 50 % of the swelling pressure in order to avoid consolidation of the sample.

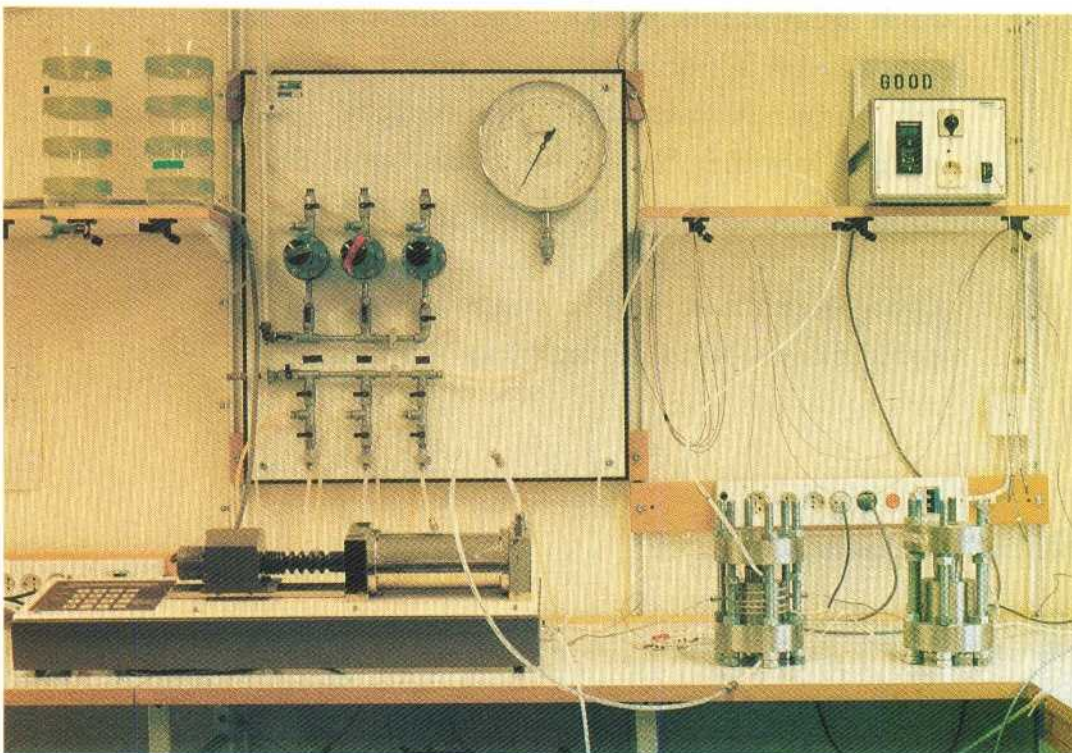
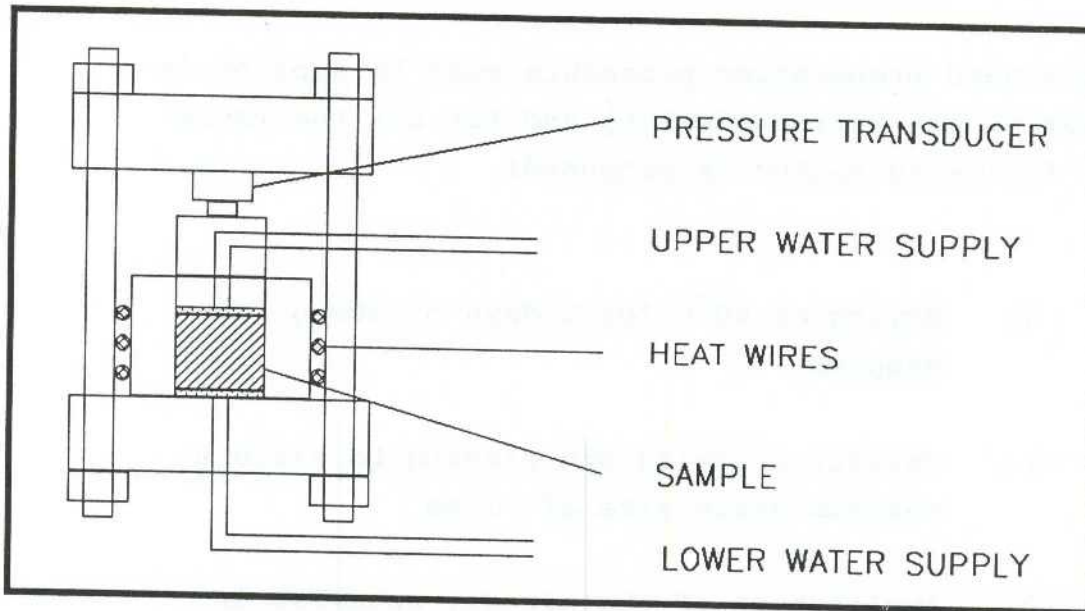


Fig.2 The swelling pressure oedometer (CT model) Oedometers seen to the right and precision flow meter (GDS), used for pressurizing also, to the left in the photo. Back-pressure system with manometer in the rear

Swelling Pressure

The signals from the pressure transducer are recorded in the course of the water saturation for the conductivity testing, by which the successive build-up of the swelling pressure is visualized. It is usually fully developed in less than one week. A typical graph is shown in Fig.3.

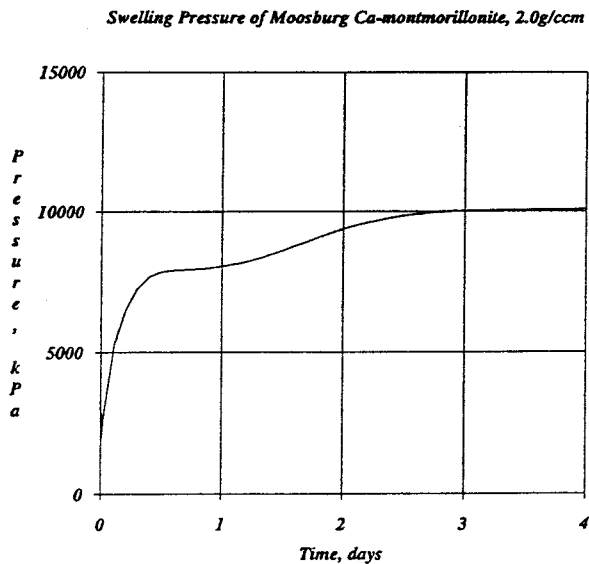


Fig.3 Typical build-up of swelling pressure in the course of water saturation of a smectite-rich sample prepared from air-dry bentonite powder

A simple checking of the swelling ability of the soil sample is made by reducing the normal load by 15 to 50 %. A very small expansion indicates a reduced expandability, which can be due to a low smectite content, or to cementation.

Creep Testing

All rheological tests must be made under specified stress conditions in order to yield parameter data that can be used in stress/strain calculations. Thus,

triaxial tests or simple shear tests are suitably used but they are impractical for quick checking of whether the soil material is cemented or not. The shear box is better suited for such tests although the stress situation in the sample is too complex to allow for accurate evaluation of other strength parameters.

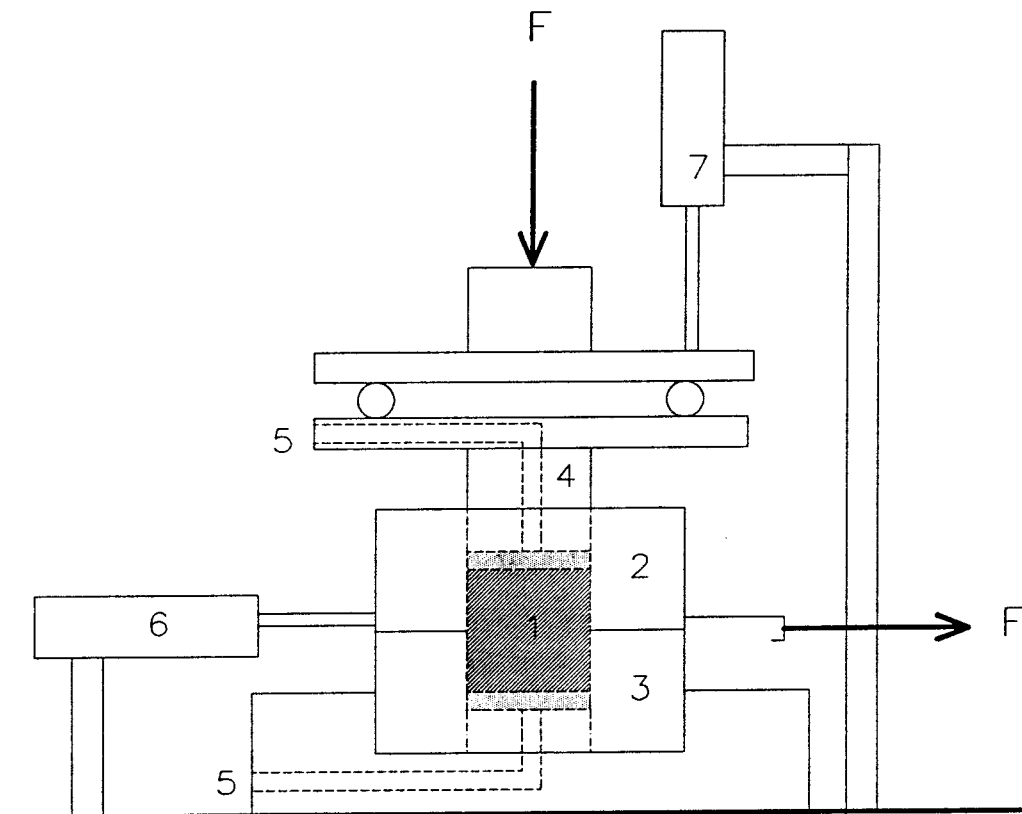


Fig.4 Shear box for general characterization of clay samples by direct shear testing

- 1) Sample confined between filters, 2 & 3)
- Shear-box halves, 4) Piston, 5) Water in- and outlets, 6 & 7) Strain gauges, F) load

The shear box has a diameter of down to 2 cm and can be used both for artificially prepared samples as described in the preceding text, and for carefully trimmed undisturbed clay samples. The theoretical background of the stress/strain relationships used for evaluating the standard parameters A, B and t_0 of the creep equations (1) and (2) that can usually be applied, is given in (6).

$$\gamma = B \ln(t + t_0) + A \quad (1)$$

$$\gamma = B (t + t_0)^{-1} \quad (2)$$

where γ = angular shear strain
 t_0 = constant (cf. Fig.5)

The parameter B is a measure of the deformability. High values indicate a low shear modulus, and low ones a high stiffness. A low B-value associated with a low or negative t_0 -value indicates cementation, which is also usually manifested by jerky creep.

Typical data of different types of smectite material prepared from air-dry powder are the following:

Material	Dry density g/cm ³	A	B	t_0 x10 ⁻⁴ s
Na-smectite	1.75	-20	5	2000
Ca-smectite	1.65	-20	4	1600
Ca-smectite (cemented)	1.80	116	6	-150

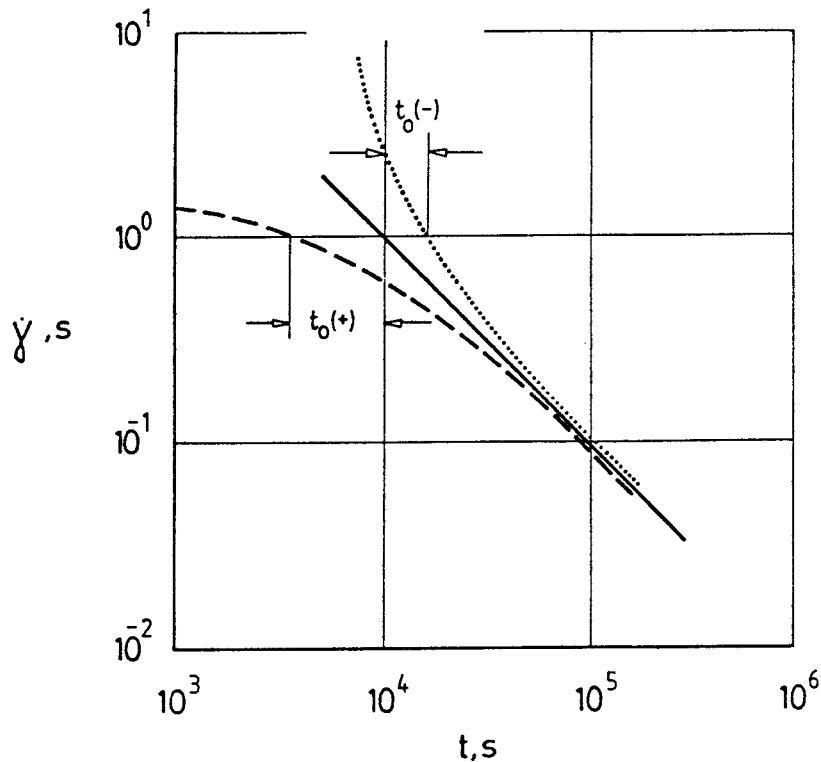


Fig.5 Creep curves showing the meaning of t_0

Thermal Properties

The heat conductivity of water saturated samples is relatively easily measured by use of a rod-shaped heat sond that is inserted in the sample, the technique being applied as a standard at several laboratories*.

The principle is that the sond is heated, yielding a transient heat flow, and that its surface temperature, which is directly related to the heat conductivity of the surrounding soil sample, is measured for about 30 minutes. The conductivity is evaluated from plottings of temperature versus log time, with the energy input and geometry of the sond as known parameters.

*Pers. comm. Sven Knutsson, Division of Soil Mechanics, University of Luleå

3.1 General

Clay samples from a natural bentonite deposit in Niederschönbuch, southern Germany, have been tested applying the proposed laboratory scheme. The samples were taken immediately above and below the redox front that is claimed to have been developed in the more than 2 m thick bed which is located at about 15 m depth (Fig.6). The bentonite is being exploited by the German company Sud-chemie for producing finely ground bentonite powder in calcium form, the processing involving drying and grinding.

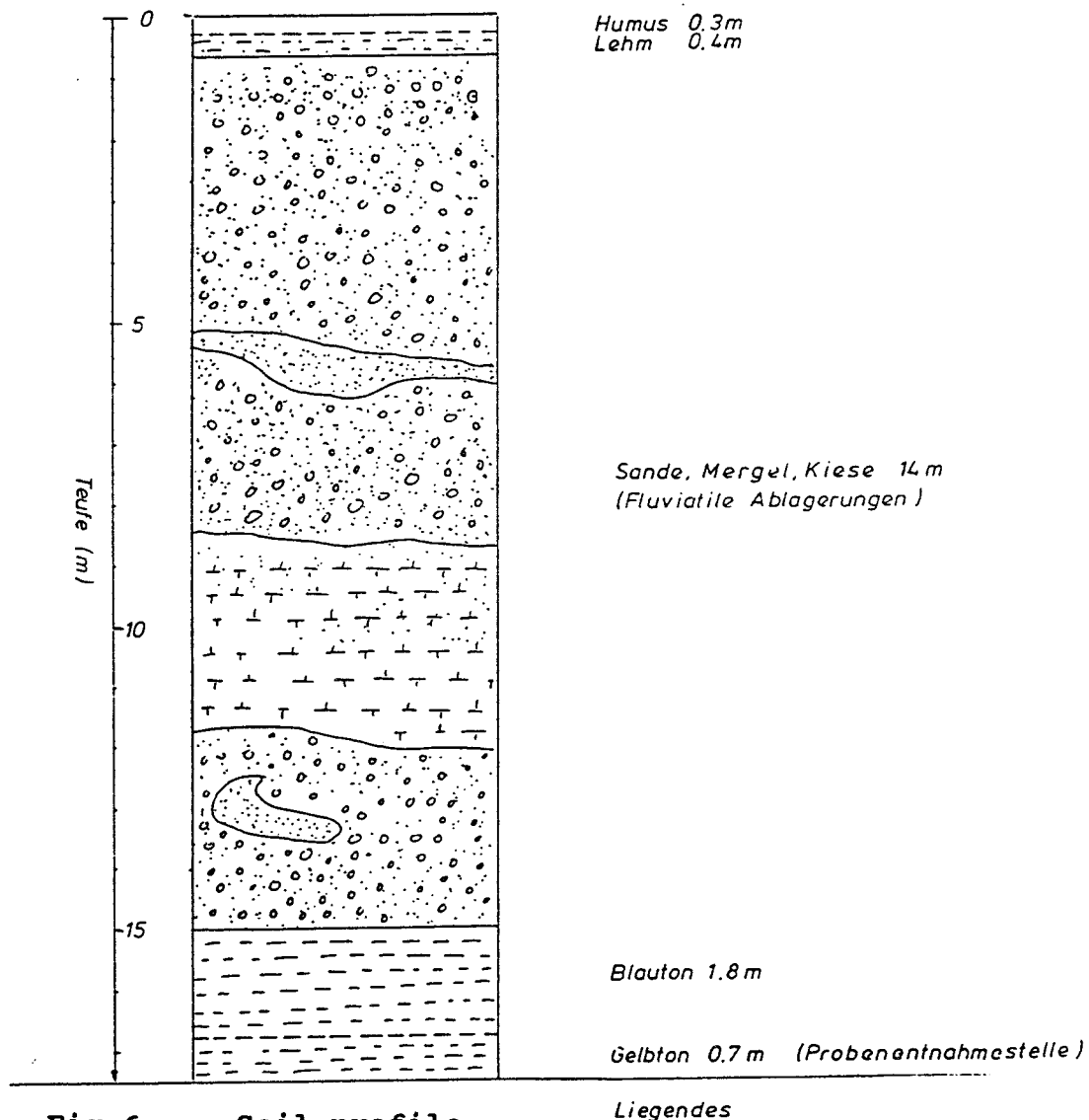


Fig.6 Soil profile

The present study comprised all the main tests specified in the preceding chapter except the measurement of thermal properties. In addition to the ordinary chemical analysis the content of iron was determined with respect to Fe^{2+} and Fe^{3+} .

3.2 Laboratory testing

3.2.1 *Granulometry, density and water content*

The Moosburg bentonite material has a clay content of about 70 %, the fresh samples from the beds that were provided by Sud-Chemie, having a natural water content of 43 % of the blue/green unoxidized sample and 46 % of the yellowish oxidized clay. This difference probably represents the normal water content variation in the bed. We will use the terms "Moosburg Blue" (MBB) for the unoxidized material and "Moosburg Yellow" (MBY) for the oxidized material in the present report.

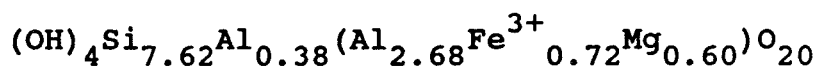
Density determinations using paraffine oil showed that the natural clay was completely water saturated. The overburden pressure can be estimated at about 150 -200 kPa, meaning that the bentonite should have a density that gives a swelling pressure of approximately this magnitude, provided that the clay is not cemented to an extent that has prevented expansion.

3.2.2 *Chemical composition*

The chemical composition, expressed in percent units and corrected for the LOI, was the following:

Mat.	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	K ₂ O	Na ₂ O	S
MBB (bulk)	65.1	19.9	6.19	4.50	1.57	1.69	0.14	0.11
MBB (clay fract.)	63.8	20.1	5.61	4.98	1.74	0.79	0.13	<0.1
MBY (bulk)	65.1	20.5	6.61	4.18	1.29	1.50	0.15	<0.1
MBY (clay fract.)	63.3	20.6	6.71	4.71	1.48	0.65	0.12	<0.1

One concludes from these analyses that the clay fraction has a composition that is rather typical of montmorillonite, and that the similarity between the composition of the clay fraction and the bulk material shows that the montmorillonite content is very high, i.e. at least 60-65 % of the bulk material. The iron content is fairly high both in the clay fraction and in the coarser material. This is in agreement with reported analyses of Bavarian clays which indicate that a typical formula, excepting adsorbed cations, is of the type:



The composition shows that iron and magnesium are present to about the same extent in octahedral positions, together with aluminum.

The matter of iron with special respect to redox conditions, will be further discussed later in the text.

3.2.3 Mineralogical composition

General

XRD, CEC, and w_L determinations, as well as scanning microscopy have been made.

XRD

Rectified XRD diagrams are shown in Fig.7, from which one finds that the spectra of the clay fraction and the bulk material are very similar, except for the marked presence of 10 Å minerals also in the clay fraction.

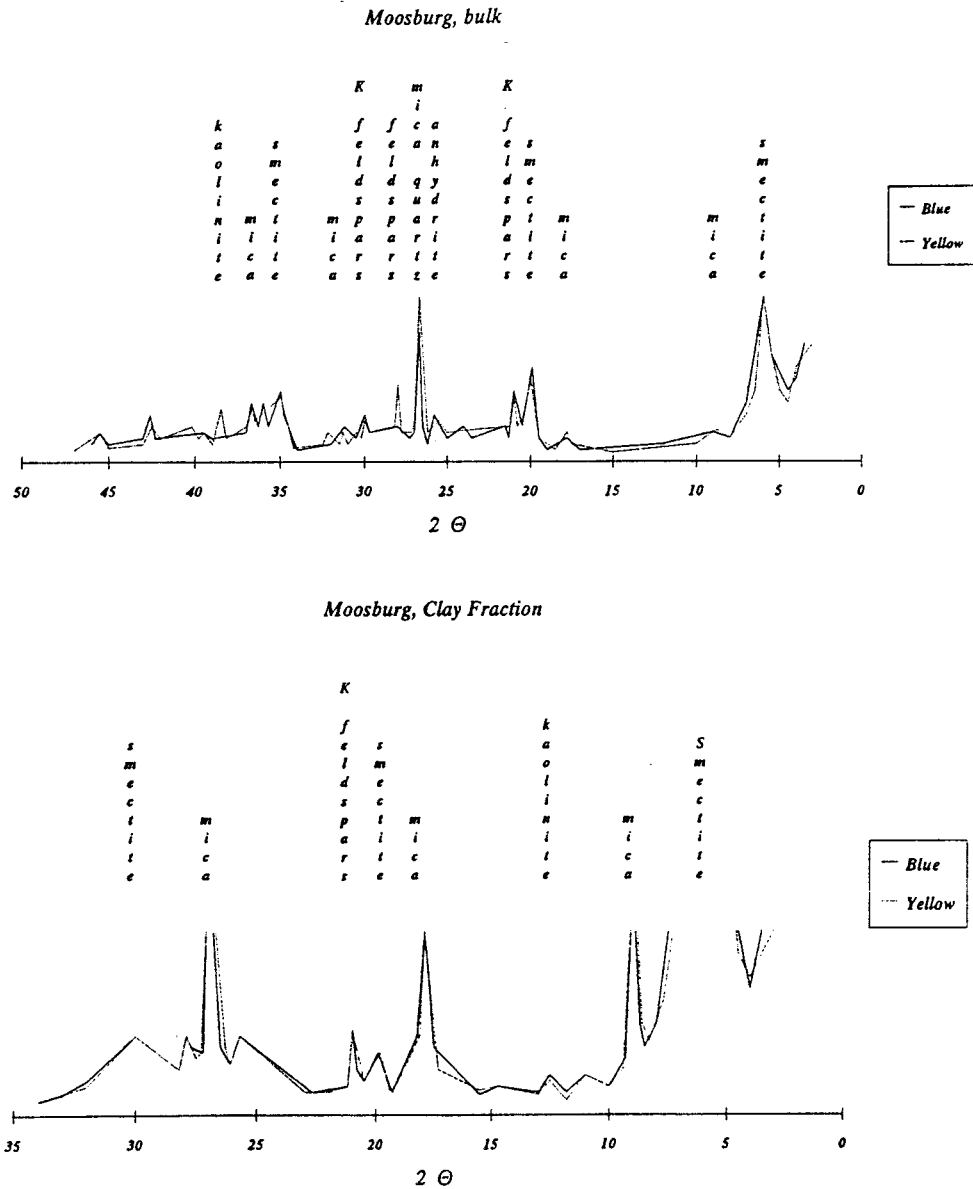


Fig.7 XRD spectra of the Moosburg clay

MBB and MBY show very similar patterns, both indicating that smectite is the dominant clay mineral. Glycol treatment gave a basal spacing of 16.8-17.3 Å, indicating a high smectite content and no vermiculite.

The major identified peaks used for identification of the minerals in the clay fraction are:

Mineral	d-value (Å)	Relative intens. of respective mineral (100 max)
Smectite	14.7	100
Mica	10.05	100
Mica	4.98	45
Kaolinite	4.48	80
Smectite (020)	4.47	50
K-feldspar	3.71	70
Anhydrite	3.49	100
Quartz	3.32	100
Feldspars	3.20	20-100
Smectite	3.00-3.20	20-70
K-feldspar	2.99	50
Mica	2.87	40
Smectite (202)	2.56	20
Hematite	2.51	70
Mica	2.46	80
Smectite (203)	2.34	-
Kaolinite	2.34	90

This listing shows that the following minerals are present:

- Smectite (Mg and Ca saturated)
- Mica
- Kaolinite
- K-, Na-, and Ca-feldspars
- Quartz
- Anhydrite
- Hematite (MBY)

Cation Exchange Capacity

The result of the cation exchange capacity testing can be summarized as follows:

Clay	Analysis	CEC, meq/100 g
MBB	NH_4^+	67
	Adsorbed cations:	
	* Total	84
	* Ca	44
	* Mg	38
	* K	0.5
	* Na	<0.5
MBY	NH_4^+	67
	Adsorbed cations:	
	* Total	74
	* Ca	41
	* Mg	35
	* K	0.5
	* Na	<0.5

Since zeolites and vermiculite are not present one concludes that the rather high CEC value of both clay types indicates a smectite content of not less than 70 % of the clay fraction, and thus at least about 60 % of the bulk material. It is clear that there is practically no sodium in the exchange positions, while calcium and magnesium are present at approximately the same extents. The physical properties are not significantly dependent on whether Ca or Mg dominates as adsorbed cation.

It should be noticed that the oxidation has led to a slight drop in CEC (84 to 74), an effect that was observed also for clays in the Spur Clay area in Wyoming (7).

Atterberg Consistency Limits

The liquid limit was found to be 110 % for the MBB clay and 121 % for the MBY clay. Since the CEC analyses show that Ca and Mg are in the exchange positions it is concluded that we deal with clays with a montmorillonite content of at least 70 % of the bulk material, on which the w_L determinations were made.

Electron Microscopy

TEM electron microscopy was applied to reveal the microstructural pattern, and SEM for the same purpose and for areal element analyses (EDX).

The micrograph shown in Fig.8 demonstrates the characteristic wavy pattern of montmorillonite clay, the clear tendency of a general alignment of stacks being typical of uniaxially consolidated sediments. The presence of a small number of accessory minerals is obvious and one can estimate the amount to be around 5-10 weight percent, which is very much in line with the previous conclusions. The upper picture shows a piece of clay that was stored in ordinary atmosphere for a few months. Its original blue/green color was changed in that period of time to a yellowish color, giving the obvious very distinct boundary between the outer oxidized and the inner unoxidized part.

Fig.9 illustrates SEM pictures showing the general character of the exposed surface of the fractured MBB clay, and EDX mappings. The general microstructure is less well illustrated than by TEM pictures but the EDX analyses are valuable since they can be used for estimating the size, shape and distribution of impurities, and for determination of the spatial distribution of elements. Pyrite crystals could easily be detected in the MBB clay.

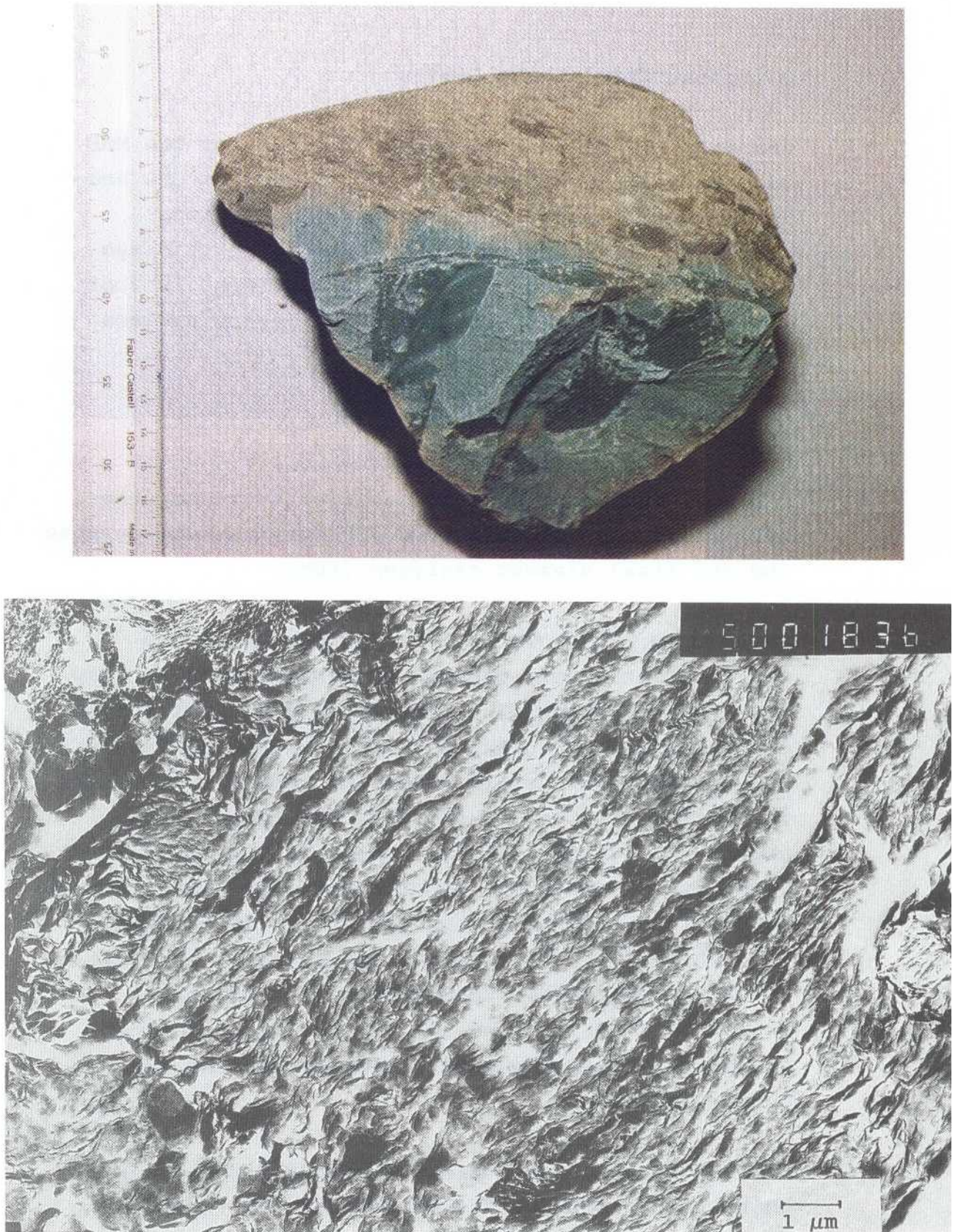


Fig.8 Appearance of Moosburg clay. Upper: Oxidation of a piece of clay yielding an external, yellow zone. Lower: Typical microstructure of a natural sediment rich in smectite

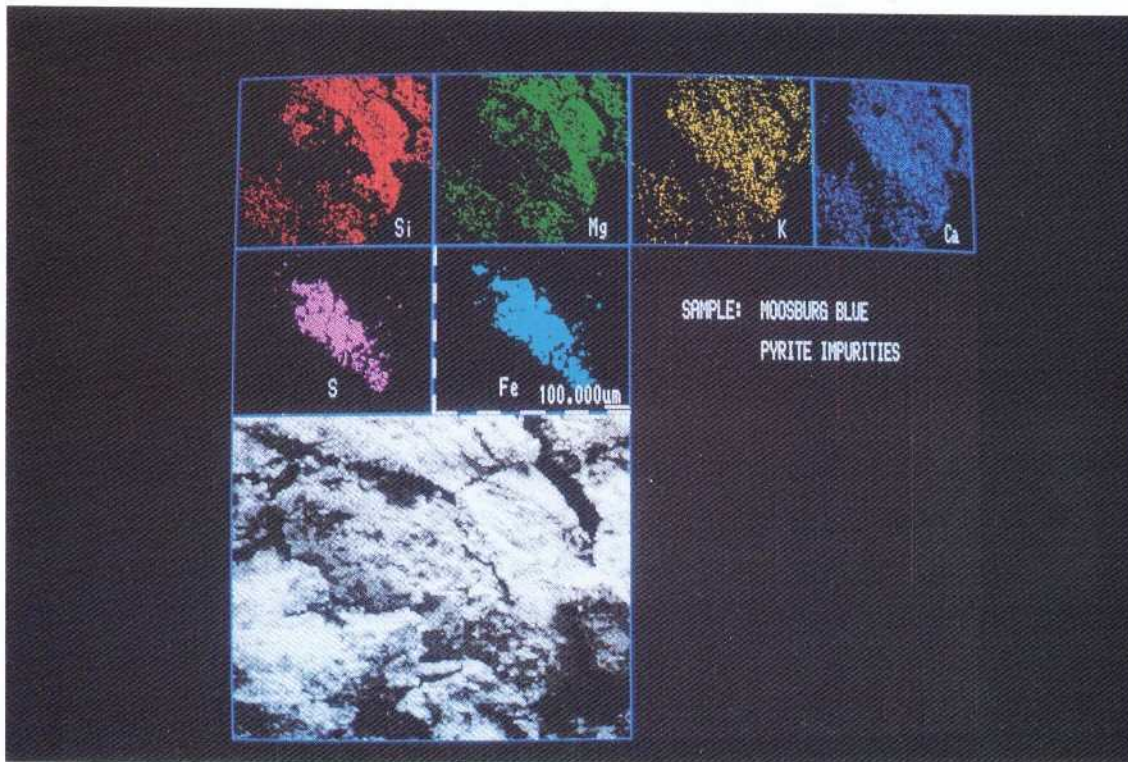


Fig.9 SEM pictures with EDX analyses of unoxidized MBB sample, showing the distribution of Si, Mg, K, Ca, S and Fe (colored) in the area indicated in the black- and white SEM picture. Note the pyrite crystal in the center!

3.2.4 Redox conditions

General

The sharp change in color from bluish to yellowish in the natural Moosburg clay profile indicates a redox front. This was checked by determining the $\text{Fe}^{2+}/\text{Fe}^{3+}$ ratio and by making an attempt to identify the iron forms on each side of the front.

Determination of Fe^{2+} and Fe^{3+} was made by the Swedish Geological Survey, Uppsala, applying Wilson's titration method.

As indicated by the typical composition of Bavarian smectites they are montmorillonites with an appreciable content of iron in octahedral positions, yet, it is not a matter of nontronite. The iron can be in Fe^{2+} as well as in Fe^{3+} form depending on the stage of oxidation. It is not known whether transfer from unoxidized to oxidized conditions, involving transfer from octahedral Fe^{2+} to Fe^{3+} , produces the typical color change from bluish to yellowish.

Color changes associated with oxidation can usually be explained by alteration of iron compounds in the soil and this may be the case also for the presently investigated bentonite bed. Also at low concentration, iron oxides, including oxyhydroxides and hydroxides, have a strong pigmenting power and give the soil a characteristic color. Typically, a redox front in a natural soil under anaerobic conditions separates yellowish material where the front has passed, and bluish material that has not yet been reached by the front. The most common yellowish iron forms in soil - often X-ray amorphous crystallites - are the following ones:

Hematite	Fe_2O_3	(Bright red)
Maghemite	Fe_2O_3	(Reddish brown)
Goethite	FeOOH	(Yellowish brown)
Lepidocrocite	FeOOH	(Orange)
Ferrihydrite	$\text{Fe}_5\text{HO}_8, 4 \text{H}_2\text{O}$	(Reddish brown)

The iron forms that give the soil bluish color are magnetite (Fe_3O_4) and hydromagnetite, $\text{Fe}_3(\text{OH})_8$, of which the latter represents a spectrum of $\text{Fe}^{2+}\text{Fe}^{3+}$ hydroxy compounds and probably governs the redox potential of soils under anaerobic conditions (8). Black compounds of this sort are precursors of the orange-colored lepidocrocite, which is expected to become converted to goethite in many soils. However,

transformation from ferrihydrite and hydromagnetite to goethite is not favored in the presence of soil constituents with abundant hydroxyl groups. Such inhibitory effects instead favor the transformation to hematite rather than goethite, which is therefore the most probable product in smectite-rich clay like the Moosburg bentonites. This actually fits the XRD data.

Chemical analyses respecting the actual $\text{Fe}^{2+}/\text{Fe}^{3+}$

The analyses showed that the ratio of $\text{Fe}^{3+}/\text{Fe}^{2+}$ of the unoxidized MBB clay was 0.3 while it was 32 for the oxidized MBY, indicating almost complete oxidation of ferrous to ferric iron. Expressed in terms of FeO , the content was found to be 4.76 % in MBB and 0.20 % in MBY.

Comments

In general, Fe^{2+} ions are more likely to occur in soils than Fe^{3+} if E_h is sufficiently low. Under unoxidized conditions, Fe^{2+} is most probably precipitated as mixed $\text{Fe}^{2+}\text{Fe}^{3+}$ compounds, of which $\text{Fe}^{2+}\text{Fe}^{3+}$ hydroxy compounds like hydromagnetite dominate. However, siderite, sulphides and - less probably $\text{Fe}(\text{OH})_2$ - may also appear depending on pH, sulphur concentration and partial pressure of CO_2 .

One finds that in the Moosburg case, the unoxidized MBB contains small amounts of iron sulphide. It could not be detected by XRD but 200 μm large pyrite crystals, associated with potassium and calcium, were readily found by use of SEM/EDX (cf. Fig.9). Siderite was not identified.

The outcome of the present study of the effect of

iron can be summarized as follows, disregarding from the influence of organic matter and from possible intermediate iron compound forms.

- * Almost complete transformation of black $\text{Fe}^{2+}\text{Fe}^{3+}$ hydroxy compounds to yellowish/reddish hematite
- * Transformation of octahedral Fe^{2+} to Fe^{3+} possibly associated with color change from bluish to yellowish

The results of the study are in good agreement with those of the Spur Clay investigation but they are contradictory to those of Eriksen, who conducted tests in which samples prepared from MX-80 bentonite powder were exposed to α and γ radiation (9). He concluded that only a fraction of the total Fe content of the investigated clay was accessible for reactions with radiolytically formed oxidative and reducing species, and thus that Fe^{2+} is not abundant.

The discrepancy between the results of the present studie and the Spur Clay investigation on the one hand and Eriksen's investigation on the other, is explained by the fact that the two first-mentioned ones concerned undisturbed, natural clay, while Eriksen's investigation was made on MX-80 clay, which, as all commercial bentonite powders, has been largely oxidized due to atmospheric oxygen in the processing.

3.2.5 *Physical properties*

The hydraulic conductivity, swelling pressure, and creep behavior were investigated using the procedures specified in Chapter 2.

Hydraulic Conductivity

The hydraulic conductivity of the unoxidized MBB clay and of the oxidized MBY clay were found to be as summarized below:

Clay	Dry density g/cm ³	Hydraulic conductivity m/s
MBB	1.11	2.3×10^{-11}
	1.59	8.7×10^{-14}
MBY	1.11	4.0×10^{-12}
	1.59	6.6×10^{-14}

The hydraulic conductivity is on the same order of magnitude as one usually finds for Ca montmorillonite, indicating again that both clays have a high content of this smectite mineral. We see that the oxidized MBY clay has in fact a slightly lower conductivity than the unoxidized MBX.

Swelling Pressure

The following swelling pressures were recorded:

Clay	Dry density g/cm ³	Swelling pressure kPa
MBB	1.11	245
	1.59	10100
MBY	1.11	287
	1.59	11500

Like the hydraulic conductivities, the swelling pressures are typical for clays rich in Ca montmorillonite when saturation is made with distilled water. Also the swelling pressure measurements indicate a slightly higher smectite content of the MBY

than of the MBB clay. It should be noted that the swelling pressure roughly corresponds to the overburden pressure, indicating that the clay has expanded to reach equilibrium with the external load.

Creep Testing

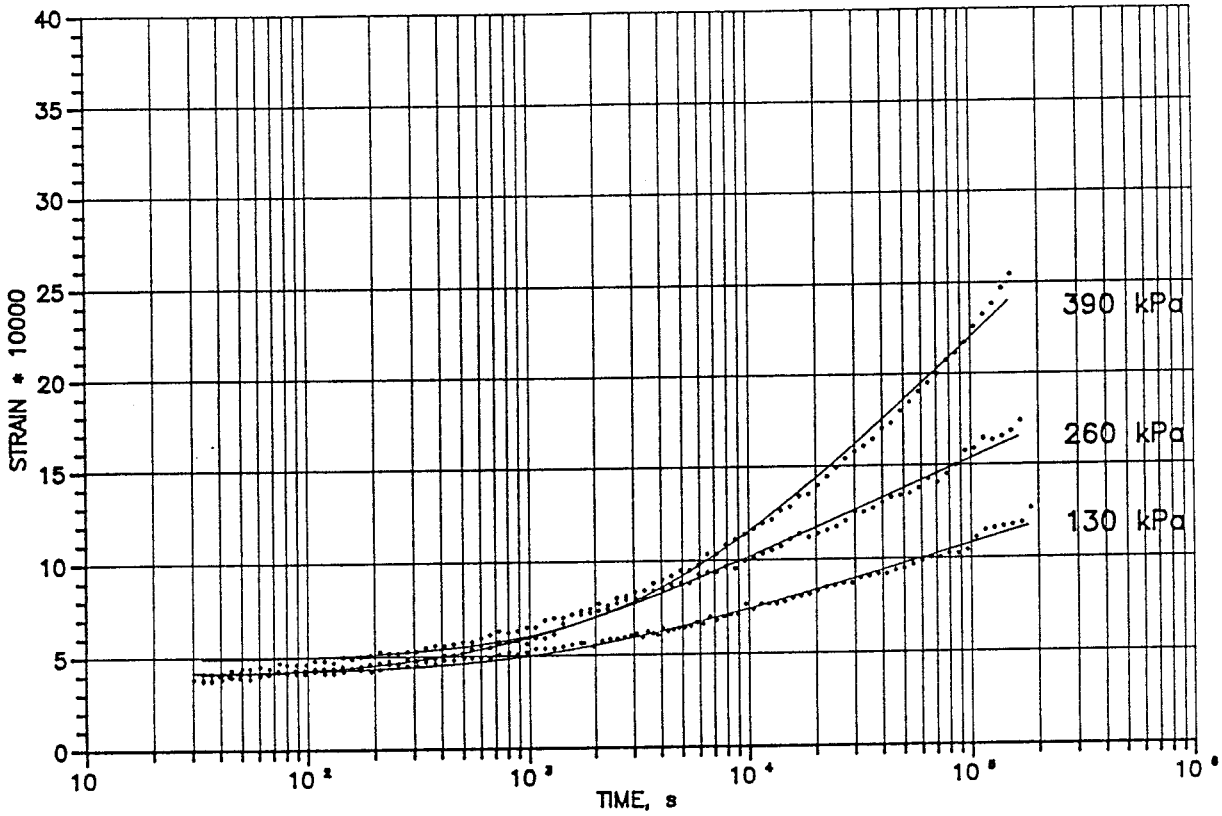
Creep testing gave smooth curves and similar appearance of the strain/time curves at shear stresses exceeding 130 kPa (cf. Fig.10). At the lowest shear stress level, 130 kPa, the oxidized MBY sample gave somewhat less strain than the unoxidized clay and it appears that the time-dependent deformation was also smaller. This indicates some slight cementation, which is also indicated by the jerks at about 10^5 s. It is highly probable that this effect is due to precipitation of cementing iron compounds in the oxidation process, while it is hard to believe that transfer of octahedral Fe^{2+} to Fe^{3+} would yield effects of this sort. The fact that jerks did not take place at higher shear stresses is explained by complete breakdown of the very weak cementation bonds at the first, low shear stress.

The fact that the shear displacement of the unoxidized MBB sample was very small and on the same order of magnitude at the onset of the three load steps, indicates strong cohesion of the unoxidized MBB due to "cementation" although without yielding brittle behavior. Amorphous silica/aluminum complexes and iron hydroxy compounds are commonly assumed to be responsible for such effects.

The creep parameters were found to be as follows:

Clay	Density g/cm ³	A	B x10 ⁻⁴	t _o s
MBB	1.59	-38	5	3800
MBY	1.59	-25	4	2300

Moosburg Blue
Normal Stress 3 MPa



Moosburg Yellow
Normal Stress 3 MPa

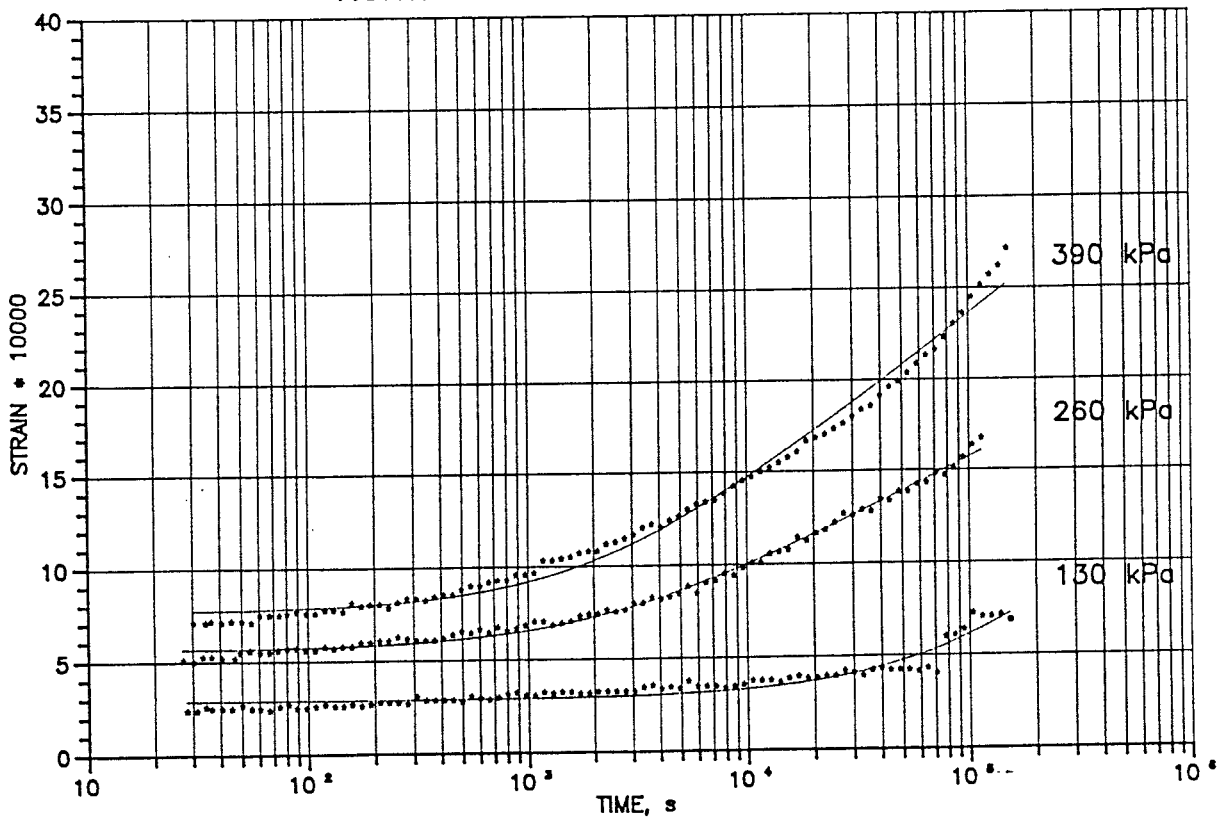


Fig.10 Creep curves of MBB (upper) and MBY (lower)

The creep data are not very different from what is considered to be typical of Ca montmorillonite clay and the rather high t_0 's indicate that cementation is not very significant.

After completion of the creep testing, the samples were allowed to expand under a normal load that was 15 % lower than the load at the creep tests, and one finds from Fig.11 that the MBB sample expanded less than the oxidized MBY sample. This behavior may well be due to partial coating of the smectite stacks by silica gel or hydroxy iron compounds.

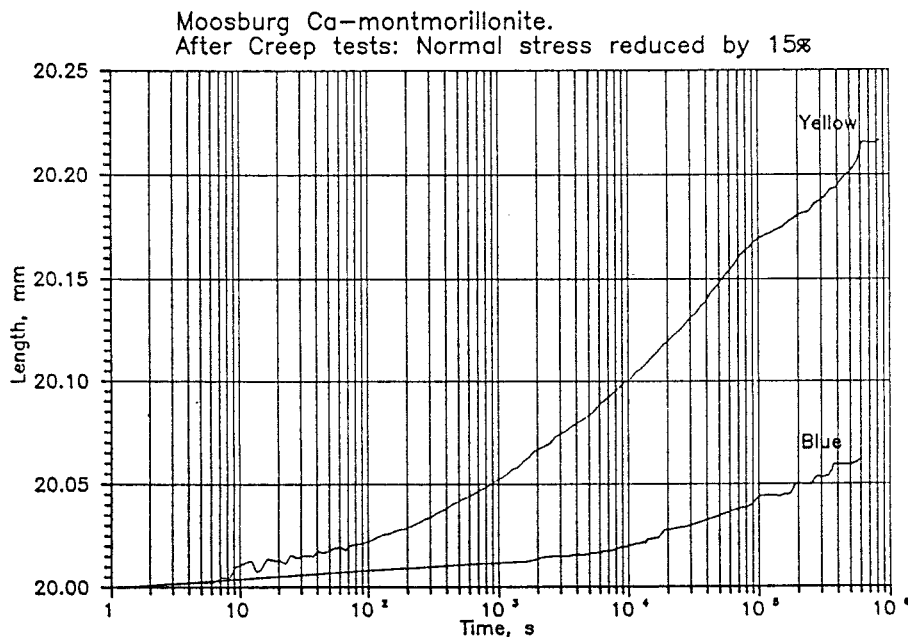


Fig.11 Expansion of MBB and MBY under reduced normal load

3.3 Conclusive remarks

The application of the test program for characterization of the Moosburg clay gave very good information on the value of the respective test and of the integrated scheme. A major conclusion is that while the individual tests did not give definite information on the mineral composition or the physico/chemical state of the clay material, the test results combined to give a very detailed picture of its nature.

There is unanimous evidence that it is a matter of very montmorillonite-rich material with Ca and Mg in the exchange positions. The importance of conducting several types of testing is illustrated by the fact that without XRD one would not know for certain whether the major clay mineral type is smectite or vermiculite, and that without the CEC measurements the major cation would not have been safely identified.

Further, the characteristic physical behavior of the clay certified that it belongs to the group of clays with a large amount of expanding minerals that is of interest in the present context. Still, the swelling and creep tests gave a clear indication of slight cementation which is not given by any other analysis.

It remains, however, to find out how well the standard testing scheme works in the case of clays that are less smectite-rich or contain undesired minerals, and it is therefore strongly recommended to apply it to some suitable, well defined materials like a number of commercially available Spanish bentonites, which represent significantly less cost than the presently investigated clay. Also, it is obvious that there is a need for developing a simple sensitive technique for quick routine checking of variations in smectite content.

As to the redox study, it is concluded that while the unoxidized Moosburg clay was characterized by dominating Fe^{2+} in both iron compounds in the bentonite clay and in octahedral positions in the montmorillonite crystal lattice, the oxidized clay horizon holds almost entirely Fe^{3+} in iron compounds as well as in the smectite lattice. A practically important conclusion is that commercial bentonite powder is largely oxidized with Fe^{3+} as dominant iron state.

4 RECOMMENDATIONS

The following three moments are suggested for future work:

1. Application of the test scheme to commercially available clays that are known to have good and less good properties:
 - * Montmorillonite-rich Greek bentonite
 - * Montmorillonite-rich Spanish bentonite from Serrata de Nijar
 - * Slightly cemented montmorillonite clay from Escullos, Spain
 - * Beidellitic smectite from Trancos, Spain
 - * Saponitic smectite from Cerro del Aquila, Spain

2. Development of a quick method for checking the smectite content based on the liquid limit of *clay material saturated with Na through cation exchange* ("normalization")

3. Repetead study of the iron state in iron-rich bentonites of potential use in Swedish repositories, i.e. one or two of the types mentioned in paragraph 1

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