Effect of Preliminary Heat Treatment on the Shear Strength of Kaolinite Clay

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•BECAUSE OF their mechanical properties some rocks are classified in the region between soils and rocks. Natural rocks of this kind, such as clay shale, conglomerate, or lignite, and artificially treated rocks such as stabilized soils, are of great interest in experimental and applied rock mechanics in mining engineering. This paper deals with some results of fundamental investigations concerning the mechanism of stabilization of cohesive soils. It is a part of a research program at the Department of Mining Engineering, Technical University Clausthal (Germany).

Thermal stabilization is here defined as an irreversible and effective increase of the shear strength of soil or rocks. Earlier papers report on the fundamentals of stabilization by heat treatment and the possibilities of application (2, 3). Heat treatment is the only procedure known till now to stabilize cohesive soils successfully in situ in the way the stabilization has been defined. Cohesive soils are aggregations of minerals or rocks with a higher percentage of fine-grained particles ($< 2\mu$) consisting largely of clay minerals. Cohesive soils are plastic within certain limits of water content. They are determined by a characteristic tensile strength. Their hydraulic permeability is relatively low ($k < 10^{-4}$ cm/sec).

When the experimental program was determined the aim was to work under reproducible testing conditions. This principle was of particular influence when the cohesive soil, the method of preliminary treatment, and the testing procedure were selected. Besides the stabilization, another aim of the investigation was to produce and characterize the change from a cohesive soil to a quasi-rock.

DETERMINATION OF SHEAR STRENGTH OF ROCKS

The shear strength of soils and rocks depends on physical and physicochemical properties of the rock (i.e., mineral content, structure, porosity, filling of voids, and related boundary effects), the initial conditions, and the dynamic and geometric limiting conditions. The influence of different factors, especially with regard to the shear strength of soils, has been described by others (5, 9).

The shear strength of samples in laboratory tests in most cases is determined by compression tests with multiaxial external loading and uniaxial tension tests. Best-known are triaxial tests on cylindrical specimens with axially symmetrical external loading, which were applied in this investigation program, too.

In soil mechanics it is common in the region of relatively low loading to plot Mohr's envelope of soils as a straight line. This simplification is no longer possible when the triaxial tests reach a wider part of the failure curve. Normally Mohr's envelope of soils or rocks is slightly curved. Therefore a different method was used to analyze the triaxial tests and to describe the envelope; it is discussed in the following.

When σ_{1i} and σ_{3i} (respectively σ_{1i}' and σ_{3i}' as effective stresses) are defined as maximum or minimum principal stresses under failure conditions it is possible to construct the corresponding stress-circles in $\sigma - \tau$ coordinates with $1/2\ (\sigma_{1i} + \sigma_{3i}) = \sigma_{oi}$ as coordinates of the centers and $1/2\ (\sigma_{1i} - \sigma_{3i}) = r_i$ as radii. By approximation the function (Fig. 1)

$$r = r\ (\sigma_o) \qquad (1)$$

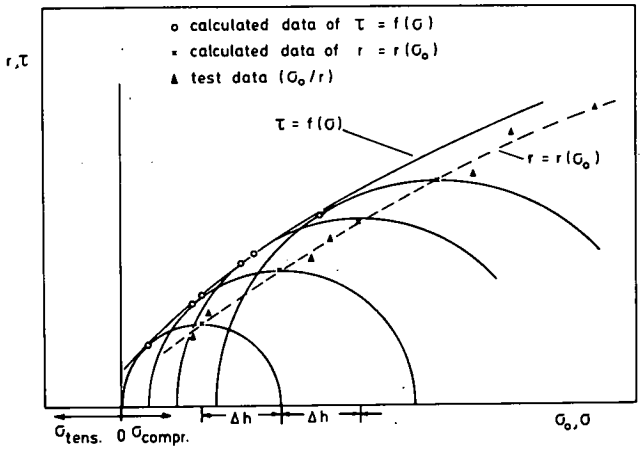


Figure 1. Construction of Mohr's envelope.

is formed. The equation of Mohr's envelope,

$$\tau = f(\sigma) \tag{2}$$

delivers

$$G(\sigma, \tau, \sigma_0) = (\sigma - \sigma_0)^2 + \tau^2 - r^2(\sigma_0) = 0 \tag{3}$$

and

$$\frac{\partial G}{\partial \sigma_0} = (\sigma - \sigma_0) + r \frac{\partial r}{\partial \sigma_0} = 0 \tag{4}$$

when σ_0 has been eliminated.

From Eq. 4 follows

$$\sigma_0 = g(\sigma) \tag{5}$$

The general equation of Mohr's envelope is now

$$\tau = \pm \left\{ r^2 [g(\sigma)] - [\sigma - g(\sigma)]^2 \right\}^{0.5} \tag{6}$$

Because of the relationship $r = r(\sigma_0)$, Eq. 6 is to be found in equations of different degrees of difficulty. Several solutions have been analyzed (4).

If Eq. 1 is written in the mathematical form of a straight line,

$$r = a + b\sigma_0 \tag{7}$$

the equation of the envelope (Coulomb's equation) changes to

$$\tau = \pm (a + b\sigma) \left(\frac{1}{1 - b^2} \right)^{0.5} \tag{8}$$

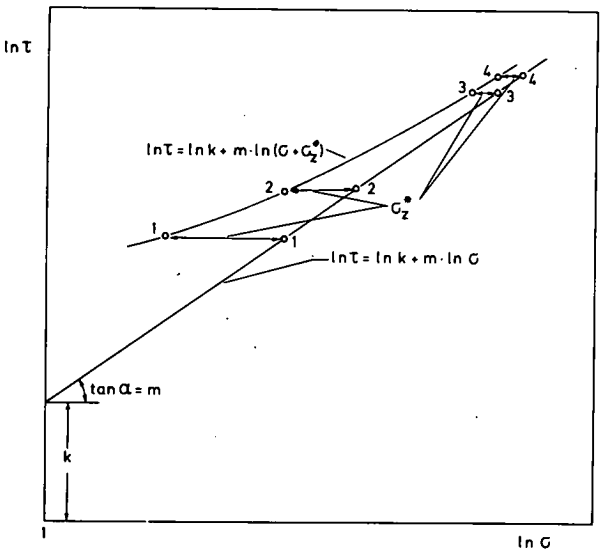


Figure 2. Determination of the parameters k, m, and σ_Z^*.

While analyzing the triaxial tests, which will be discussed later, the data σ_{0i} and r_i were equalized by an approximate polynomial. Within each measuring range (Fig. 1) the envelope consists of finite segments, which are plotted by steps of Δh with $r = r(\sigma_0)$. The envelopes have been calculated by a computer.

To describe the curved envelope an exponential equation of the form

$$\tau = \pm k \left(\sigma + \sigma_Z^*\right)^m \qquad (9)$$

is useful. If m = 1 the equation is identified by a straight line. In log-log coordinates the parameters k and m can be determined easily; σ_Z^* marks theoretically calculated data of the triaxial tensile strength of rock. From Figure 2 it follows that σ_Z^* can be calculated approximately only from the run of the failure line in the region of compressive loading (4, 10). Plotting Mohr's envelope in log-log coordinates, the line runs slightly curved as σ_Z^* was randomly equalized to zero. The real envelope referring to Eq. 9, however, has to be a straight line having the distance σ_Z^* from the corresponding curved line. Therefore it is possible to define the parameters k, m, and σ_Z^* with a good approximation. These results were also calculated by using a computer. As in soil mechanics, the parameter cohesion c, angle of internal

TABLE 1

PROPERTIES OF UNTREATED KAOLIN Ia FROM ZETTLITZ

Procedure	Constituent	Percent
Chemical analysis	SiO_2	47.37
	Al_2O_3	37.50
	Fe_2O_3	0.85
	TiO_2	0.20
	CaO	0.65
	MgO	0.15
	K_2O, Na_2O	0.65
	Loss on ignition	12.63
Analysis by Berdel method	Clay	95.5
	Quartz	1.0
	Feldspar	1.5
Analysis by Kallauner method	Clay	93.0
	Quartz	4.0
	Feldspar	3.0
Unit weight of solid constituents	2.629 g/cm^3	
Index of pH	8-8.5	
Grain-size distribution	< 1μ	57
	< 2μ	16
	< 3μ	12
	< 5μ	11
	<10μ	2
	<15μ	0.5
	>15μ	1
Liquid limit	0.529	
Plastic limit	0.346	
Plasticity index	0.183	

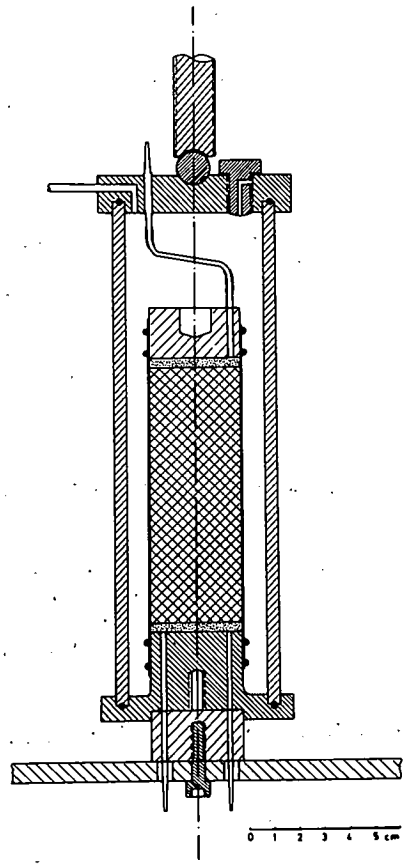


Figure 3. Consolidation cell.

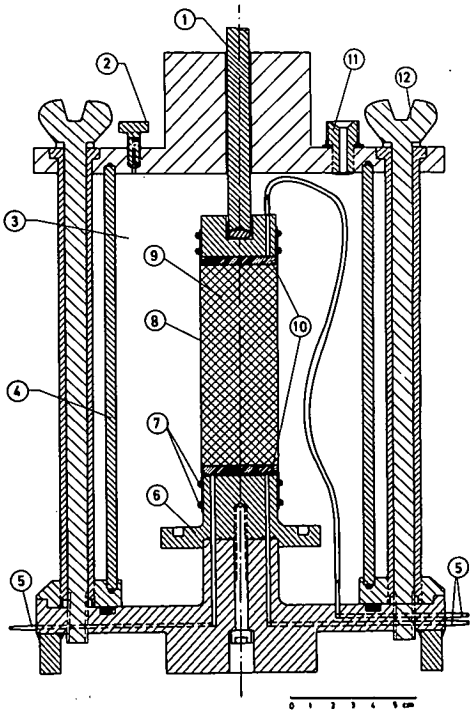


Figure 4. Triaxial cell: 1—piston; 2—air release valve; 3—water; 4—Perspex cylinder; 5—drainage or pore pressure connection; 6—lower part of consolidation cell; 7—rubber O-ring; 8—rubber membrane; 9—sample; 10—porous disc; 11—connection to pressure supply; 12—tightening screw.

friction ϕ, m, and k, and to some extent σ_z^*, too, can be expressed only as mathematical parameters of an equation, by which the run of the envelope and hence the shear strength of a rock can be described with sufficient accuracy.

TESTING PROCEDURE

For the investigation a standard material (Kaolin Ia from Zettlitz, CSSR) was selected that is characterized by optimal properties of drying and heating. The thermal reactions are well known (7). Table 1 shows some of the most important mineralogical data and index properties of the kaolin. For 14 days the kaolinite clay was kept with an initial water content of 48 to 50 percent. After that, samples that were to a great extent homogeneous and de-aired were prepared by a vacuum-extrusion press (diameter 40 mm, length 120 mm). The samples were dried for about 28 days, and then were heated for 48 hours in an electric furnace with temperatures up to 600 C. The increase of temperature by which the samples were heated was 60 C/hour. The treated samples were kept for 14 days in a desiccator and after that the dry and water-saturated specimens were tested with regard to their shear strength.

Wetting the specimens and the preconsolidation were performed in special consolidation cells (Fig. 3). Lowe and Johnson (6) described a similar method that was used after wetting the samples in order to get as high a saturation as possible. When water had passed the specimen at the upper porous stone, reduced pressure was brought to both ends of the sample. At the same time that the triaxial pressure outside of the partly de-aired specimen was taken, a pore water pressure was obtained. This action was repeated several times every 3 hours. Using the back-pressure method it was possible to get a saturation of more than 96 percent. The specimens in the cells were

consolidated at a pressure of 1, 3, 7, 12 or 25 kp/sq cm, i.e., at the same confining pressure applied on the triaxial tests. Because of the special construction of the consolidation cells it was possible to install the lower part of the consolidation cell together with the preconsolidated specimen in the triaxial cell (Fig. 4) so that the triaxial tests could be carried out after 1 or 2 days.

To achieve constant cell pressures up to 25 atm, special units were used that were developed by the second author (Fig. 5). The pressure pistons are moved by a back-geared motor to decrease the friction between piston and cylinder. This causes a fast automatic adaptation of the pressure system to volume changes in the cell.

With regard to water-saturated samples, the consolidated undrained (CU) triaxial tests were performed with measurement of pore water pressure. To measure the pore water pressure two specially designed and constructed devices were used that work automatically with null indicator controlled by a photoelectric tube (Fig. 6). The pump is driven by an electric motor combined with a solenoid brake. The system, because of its photoelectric control, is characterized by high operating reliability and small deviation.

The triaxial tests were performed using Geonor cells up to 7 atm and Farnell cells up to 25 atm, three each, as well as two testing machines with a screw jack operated by an electric motor and gearbox for tests with a controlled rate of strain. All data were registered photographically.

The same testing equipment was used when triaxial tests were performed on pre-treated dry specimens, but the porous stones were exchanged for metal plates. In addition to the triaxial tests, uniaxial compression and tension tests were made (Fig. 7).

Preliminary investigations concerning the influence of all different factors on water-saturated samples showed that a feed motion of 0.005 mm/min of the testing machine

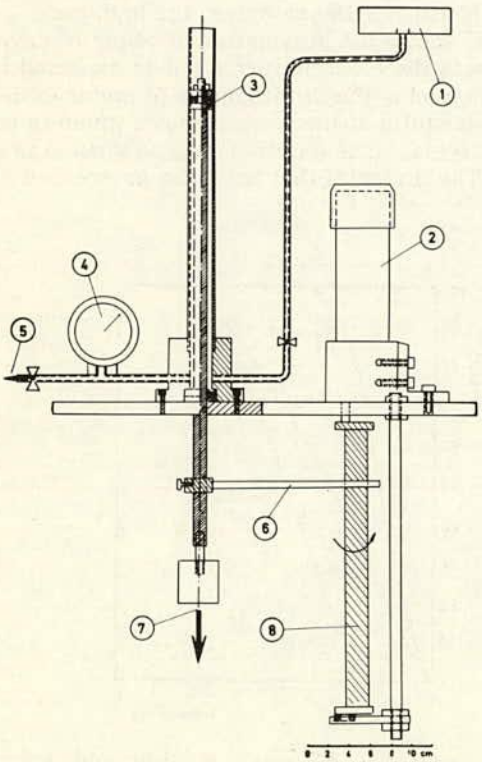


Figure 5. Apparatus for controlling cell pressure.

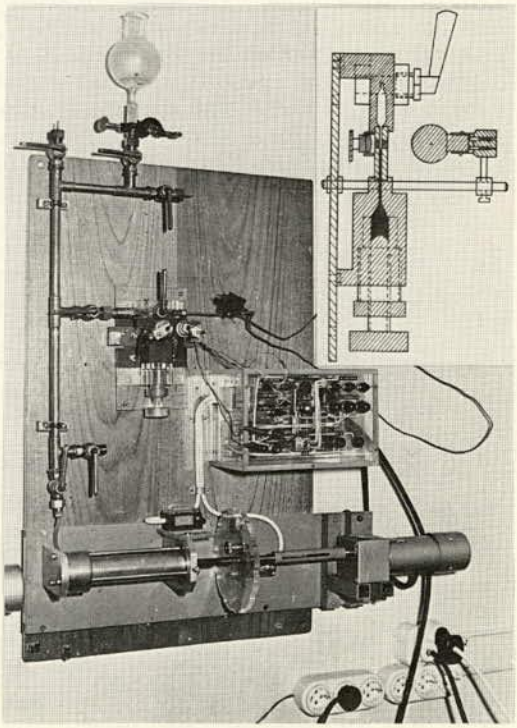


Figure 6. Apparatus for measuring pore pressure.

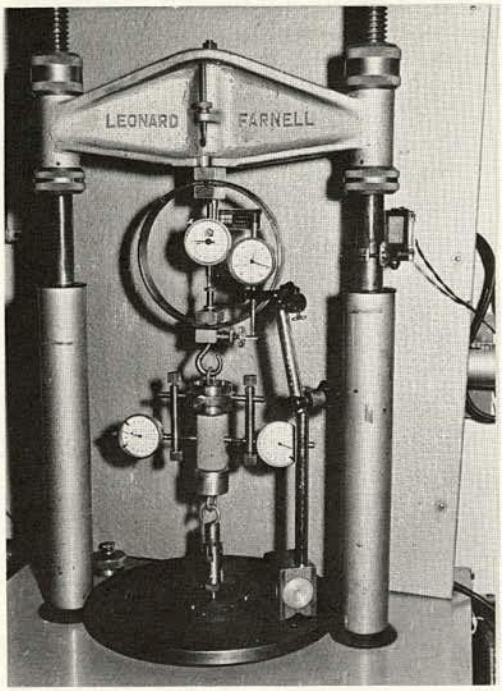


Figure 7. Apparatus for tensile tests.

was necessary, while for compression and tension tests of dry samples a maximum rate of strain of 0.01 mm/min was best.

The testing program was as follows: At least ten single tests at different confining pressures were carried out to define Mohr's envelope at every step of pretreatment (i.e., temperature of heating, dry, and water-saturated). Besides those tests necessary in determination of shear strength, several additional investigations were run that are discussed in the following section.

TEST RESULTS

The thermal reactions of the kaolinite, and especially those of the kaolin from Zettlitz, are known from numerous publications in ceramics research (7). Figure 8 gives an idea of the drying process of the samples that had an initial water content of 48 to 50 percent. The differential heating curve of kaolin (Fig. 9) shows a weak endothermal reaction in the region between 100 and 300 C that results from the dissipation of the residual adsorption water. Because of the relatively high velocity of heating of the samples, the high endothermal effect starts around 500 to 600 C, and shows the dissipation of water of crystallization. While heating the samples slowly in the electric furnace it is expected that destruction of the crystalline structure and beginning the development of metakaolin lie around 440 to 450 C. The modification to metakaolin at the temperatures given is confirmed by the results of X-ray diffraction analysis. The kaolin shows no change in the crystalline structure up to 400 C (Fig. 10). The material that has been pretreated at

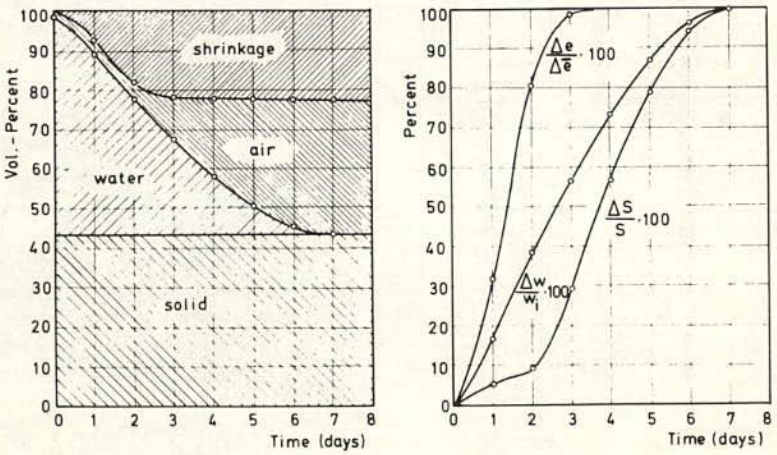


Figure 8. Drying process of the kaolin samples: e—void ratio; $\Delta\bar{e}$—total change of void ratio; S—degree of saturation; w_i—initial water content; Δw—change of water content.

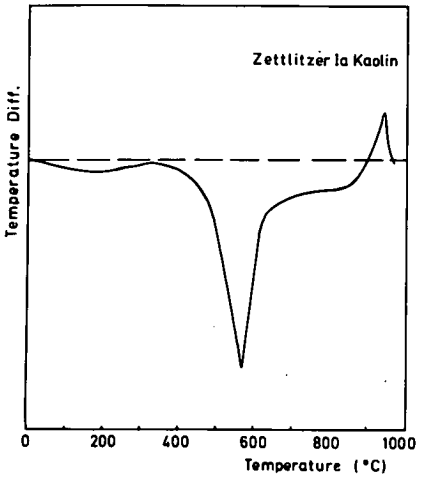


Figure 9. Differential thermal analysis of Kaolin Ia from Zettlitz.

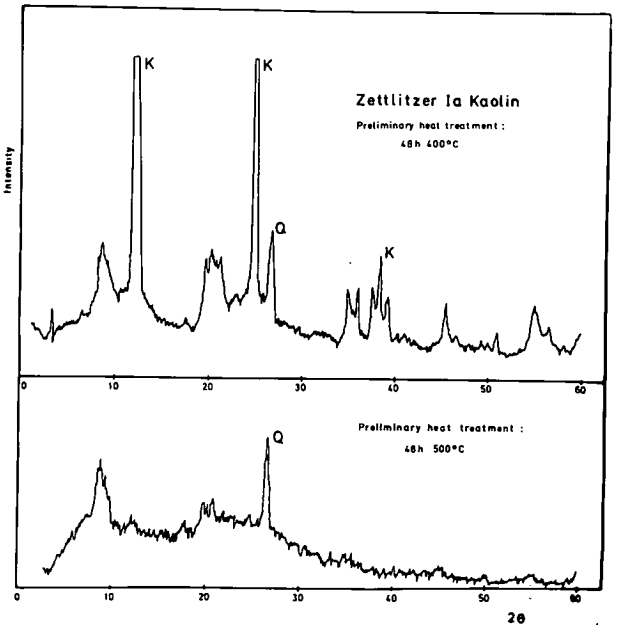


Figure 10. Structural analysis by X-ray diffraction of Kaolin Ia from Zettlitz.

500 C, however, has a clearly visible destroyed crystalline structure. In the diagram only the quartz is obvious.

Further investigations had to answer the question of how far any other characteristic data of the kaolin besides shear strength change in dependency on thermal pretreatment. Table 2 gives the results of those investigations. Except for the void ratio of the dry specimen, which shows the shrinkage of kaolin, these results, too, demonstrate that the specific gravity, flow limit, plastic limit, and plasticity definitely change after a treatment of more than 500 C. It is of great importance to the ensuing strength tests that the kaolin treated at more than 400 C no longer has a plasticity and does not swell when water is delivered.

TABLE 2

RESULTS OF HEAT TREATMENT

Preliminary Heat Treatment	Unit Weight of Solid Const. (g/cm^3)	Void Ratio e of Dry Samples	Liquid Limit	Plastic Limit	Plasticity Index
Untreated	2.629	–	0.529	0.346	0.183
48 hr 105 C	2.629	0.800	0.530	0.340	0.190
48 hr 200 C	2.622	0.814	0.525	0.336	0.189
48 hr 300 C	2.622	0.819	0.520	0.351	0.169
48 hr 400 C	2.620	0.815	0.522	0.378	0.144
48 hr 500 C	2.456	0.860	–	–	–
48 hr 600 C	2.485	0.848	–	–	–

TABLE 3

UNIAXIAL TENSILE STRENGTH OF DRY SAMPLES

Preliminary Heat Treatment	Uniaxial Tensile Strength (kp/cm^2)
48 hr 105 C	4.71
48 hr 200 C	5.06
48 hr 300 C	5.24
48 hr 400 C	5.43
48 hr 500 C	3.05
48 hr 600 C	4.68

Table 3 gives the results of uniaxial tensile tests run with dry pretreated samples. Figure 11 shows the failure line as a result of triaxial compression tests. In order to plot the graph in a distinct manner only the equalized curves are given. Because of the curved run of the envelope it is described by an exponential equation (Eq. 9). The theoretically calculated part of the failure line for tensile strength and small compres-

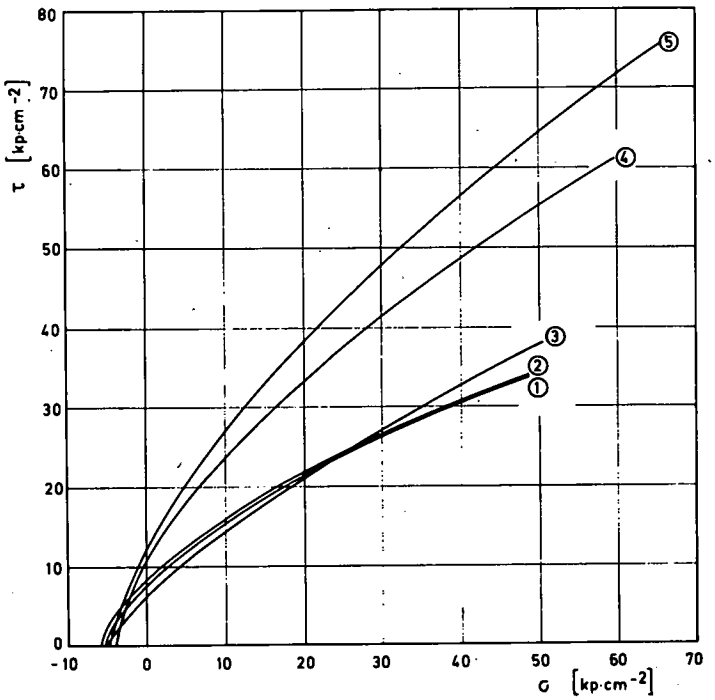


Figure 11. Mohr's envelopes of preliminary heat-treated dry samples (testing temperature 20 C). Equations of Mohr's envelopes:

(1) $\tau = \pm 2.50\ (\sigma + 5.3)^{0.66}$; pretreatment, 48 hr 200 C
(2) $\tau = \pm 2.95\ (\sigma + 5.8)^{0.61}$; pretreatment, 48 hr 300 C
(3) $\tau = \pm 1.73\ (\sigma + 5.0)^{0.78}$; pretreatment, 48 hr 400 C
(4) $\tau = \pm 4.38\ (\sigma + 4.0)^{0.64}$; pretreatment, 48 hr 500 C
(5) $\tau = \pm 4.29\ (\sigma + 4.65)^{0.68}$; pretreatment, 48 hr 600 C

sive strength correspond fairly well with the results of the experimentally found uniaxial tensile strength.

While the failure lines of the samples pretreated at temperatures of 200, 300, and 400 C show a similar run, it is to be recognized that the specimens treated at 500 and 600 C show a higher increase of shear strength in the region of compressive strength. Compared with the results of the tensile tests, it may be concluded that the change from kaolin to metakaolin at more than 400 C refers at the beginning to a higher decrease of uniaxial tensile strength. However, the specimens pretreated at 600 C develop a higher tensile strength corresponding to their increasing shear strength. The influence of thermal treatment at temperatures above 400 C on dry samples of kaolinite clay may be described in the way that the specimens achieve more and more the character of brittle rocks.

Because the kaolin keeps its plasticity up to 400 C, it is expected that the influence of water on the shear strength of pretreated samples up to that temperature is notable. Because the shear strength of rocks depends to a great extent on the void ratio and deformation conditions, these parameters were examined carefully in connection with the triaxial tests of the water-saturated specimen. The results of the CU tests in the form of the equalized and theoretically extrapolated failure lines are shown in Figure 12. The shear strength curves for samples at 105, 200, and 300 C are largely identical, so that Figure 12 only shows the failure lines for the untreated material and that which has been treated at temperatures ranging from 300 to 600 C.

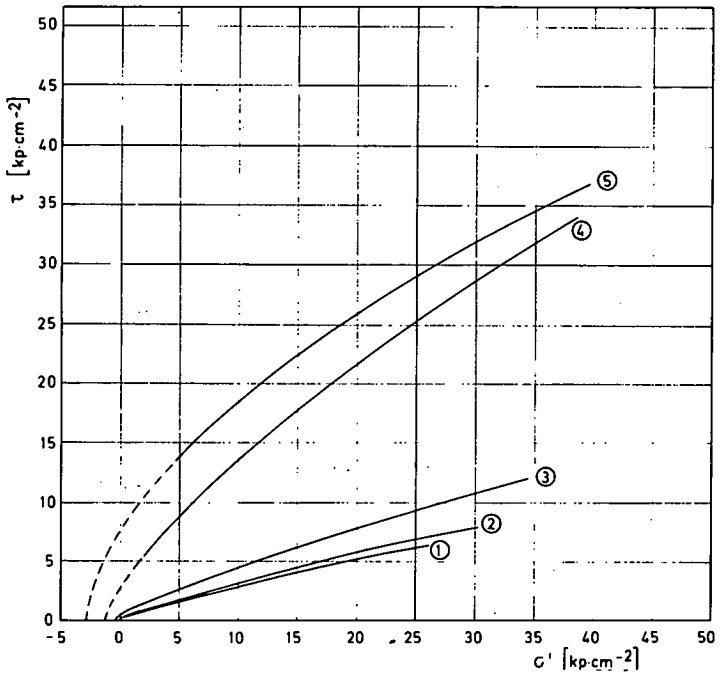


Figure 12. Mohr's envelopes of preliminary heat-treated water-saturated samples (testing temperature 20 C). Equations of Mohr's envelopes:

(1) $\tau = \pm 0.41\ (\sigma' + 0.10)^{0.84}$; untreated
(2) $\tau = \pm 0.49\ (\sigma' + 0.27)^{0.82}$; pretreatment, 48 hr 300 C
(3) $\tau = \pm 0.61\ (\sigma' + 0.61)^{0.83}$; pretreatment, 48 hr 400 C
(4) $\tau = \pm 2.38\ (\sigma' + 1.22)^{0.72}$; pretreatment, 48 hr 500 C
(5) $\tau = \pm 4.26\ (\sigma' + 2.80)^{0.58}$; pretreatment, 48 hr 600 C

It is recognized that the higher, durable stabilization increasing influence of the thermal treatment does not occur except above 400 C. The increase of shear strength up to 300 C is only due to the capillary tensile stress and when the sample is water-saturated due to the lasting initial stress, while in a specimen pretreated at 400 C first changes in the crystalline structure may occur. By referring to pressure-void ratio diagrams (Fig. 13) and stress-deformation properties of the samples (Figs. 14a-e), the influence of pretreatment may be shown more distinctly than relating to the failure lines. The pressure-void ratio lines show that the mobility of the solid skeleton decreases when the temperature of the pretreatment increases. The void ratio at the end of consolidation, which is of essential influence on the shear strength when CU tests are performed, is a function of the rigidity of the grain skeleton and also defines the stress-deformation behavior of the samples in triaxial tests.

In Figures 14a-e the specific axial strain ϵ_1 of the samples is plotted against the deviator stress. Looking at the untreated samples, the maximum of the deviator stress runs with increasing confining pressure σ_3 to a smaller axial strain ϵ_1. This result refers to the widely varying void ratio at different consolidation stresses σ_{3k} after consolidation has ended. On the other hand the samples pretreated at 400 C (Fig. 14c) show very clearly the typical stress-strain behavior of a quasi-rock. When σ_3 increases the maximum of the deviator stress moves to a higher axial deformation.

The increased rigidity of the solid skeleton is also noted quantitatively in a corresponding decrease of the specific axial strain up to the state of failure. That is especially distinct when Figures 14c-e are compared. In spite of the basic similarity,

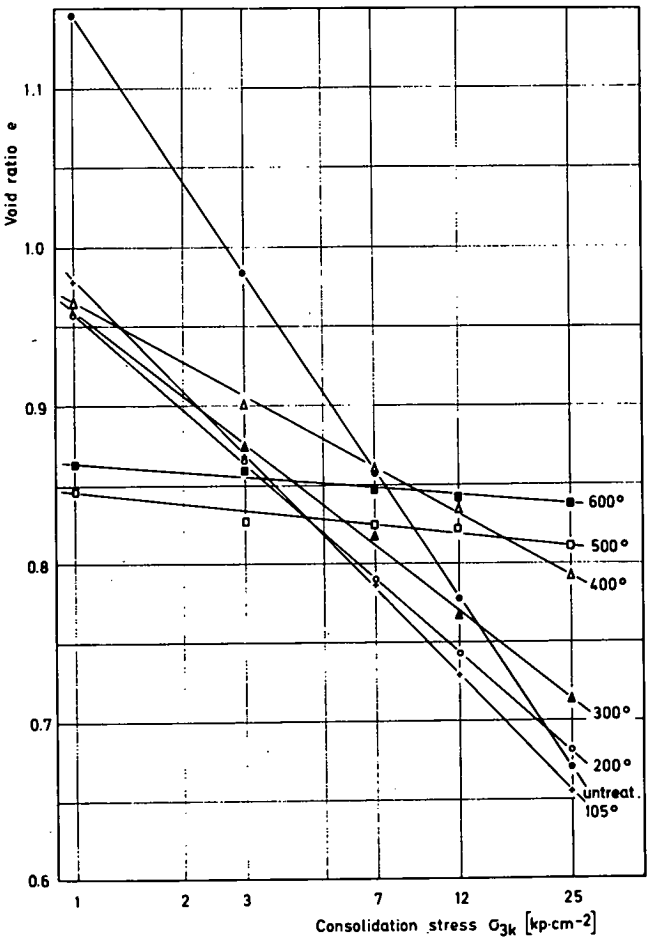
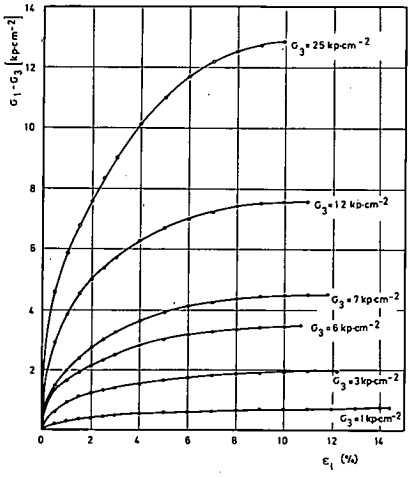


Figure 13. Pressure-void ratio lines of water-saturated samples.

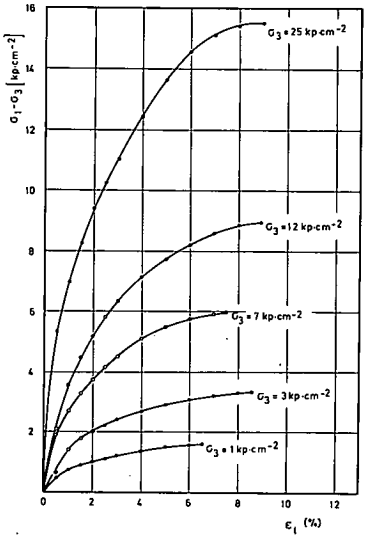
the samples treated at 500 and 600 C show in comparison to those at 400 C an increased shear strength that is connected with a very much decreased axial deformation.

The mechanical properties of the stabilized specimen are to be recognized from the results of triaxial tests in addition from the dependency on the deformation in direction of the major principle stress. Investigations of that kind, although with a different testing technique, have been carried out by Schmertmann and Osterberg (8) and Broms (1). In both publications the failure line of rocks is described by a straight line, and it follows that the mobilization of the shear strength is defined with the aid of the mobilized shear strength parameters c and ϕ. It is indicated that the cohesion c of a rock is already mobilized at a much lower axial strain than the angle of internal friction.

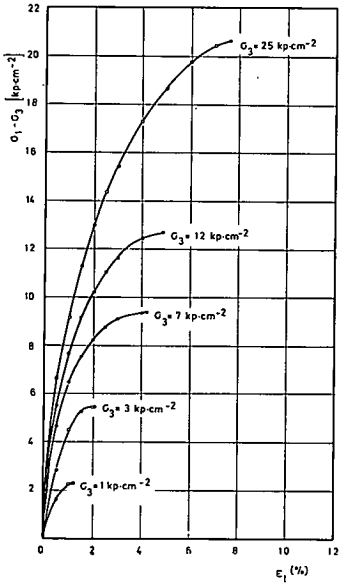
Cohesion and internal friction, however, are first of all mathematical parameters of an equation describing a straight line, and secondly an index of soil mechanics data. Regarding curved failure lines as a result of triaxial tests at porous soils or rocks within a wider stress distribution, it is not very advantageous to use the parameters c and ϕ to characterize the failure line. It would be of much more sense to indicate the mobilization of shear strength at a certain normal stress σ or σ' in dependency on ϵ_1 in percent of the corresponding maximum shear strength of a rock to each normal stress. In Figure 15 Mohr's envelopes of the mobilized shear strength are plotted in σ-τ coordinates. It is easy to construct from the diagrams the percentage mobilized



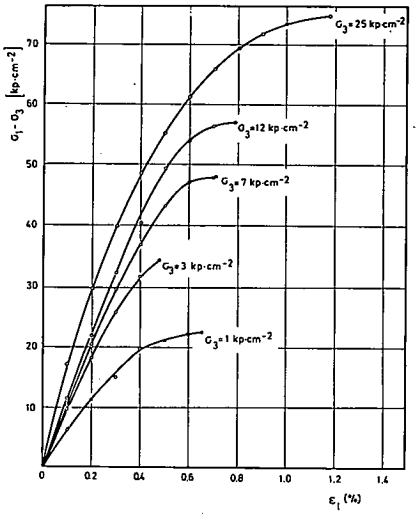
(a) Untreated samples.



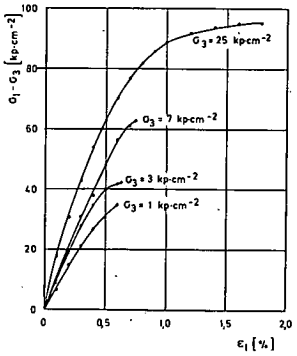
(b) Preliminary heat treatment 48 hr 300 C.



(c) Preliminary heat treatment 48 hr 400 C.

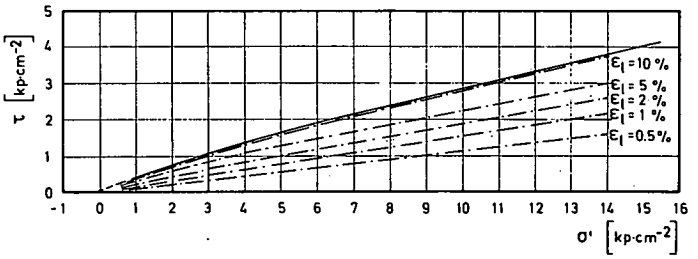


(d) Preliminary heat treatment 48 hr 500 C.

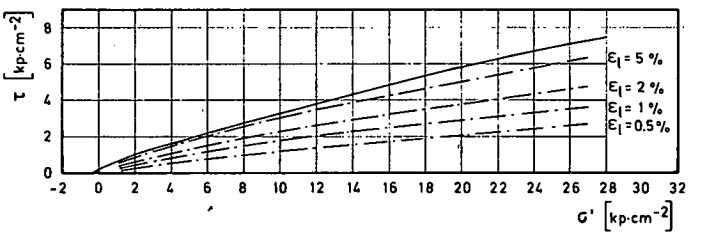


(e) Preliminary heat treatment 48 hr 600 C.

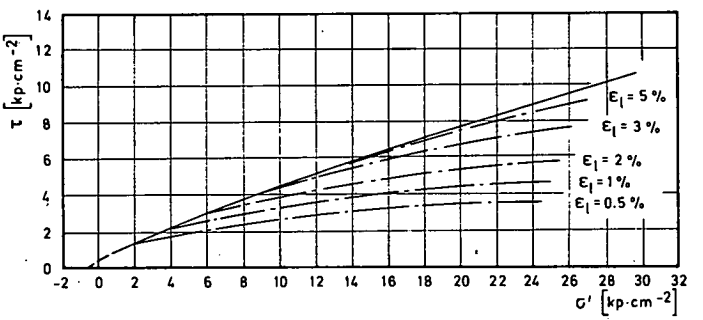
Figure 14. Stress-strain curves of preliminary heat-treated and water-saturated samples (testing temperature 20 C).



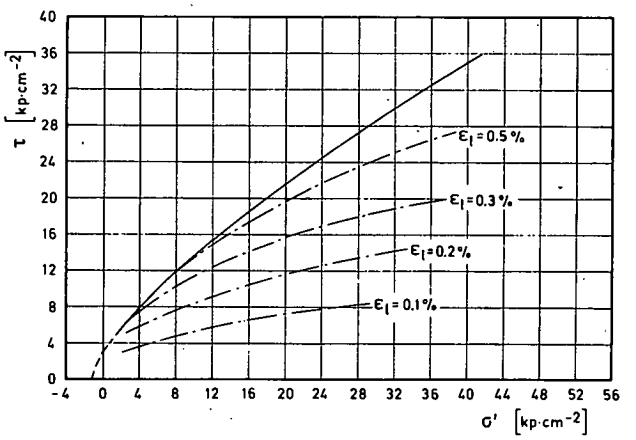
(a) Untreated samples.



(b) Preliminary heat treatment 48 hr 300 C.



(c) Preliminary heat treatment 48 hr 400 C.



(d) Preliminary heat treatment 48 hr 500 C.

Figure 15. Mobilization of shear strength with axial strain ϵ_l on preliminary heat-treated water-saturated samples.

shear strength. The run of the curves shows from the stress-strain behavior the difference in the strength behavior of a rock that has been changed by stabilization artificially.

CONCLUSIONS

The results of experiments concerning the shear strength of preliminary heat-treated samples of kaolin from Zettlitz described in this paper permit the following conclusions:

1. The preliminary heat treatment of kaolinite clay, especially of kaolin from Zettlitz, provides effective stabilization, i.e., a permanent increase of the shear strength.
2. The successful stabilization can be characterized by Mohr's envelope of the material. This presentation can be improved by the mobilization of the shear strength in relation to axial strain in the direction of the major principal stress.
3. The slightly curved envelope can be described with sufficient accuracy by an exponential equation. The triaxial tensile strength that is to be calculated theoretically represents a characteristic factor. If the envelope is described in that way it is not necessary to use the normal parameters, cohesion and angle of internal friction, and to give them a different physical meaning. Because of the great number of factors influencing the shear strength and thus the stabilization, it cannot be expected that the influence of these factors can be noticed in the amount or in the mobilization of the parameters of shear strength. The reason is that these factors are only mathematical parameters of a certain approximated equation for the envelope.
4. Concerning the shear strength of preliminary heat-treated samples of a cohesive soil, the water as a void filling is of special importance. In the case of a material in which the increasing strength is only due to its capillary tension and not to changing structure, the effect of stabilization when reducing this capillarity by adding water is irreversible with a small amount. The increasing strength and with it the alteration of the mechanical properties mentioned is of less influence on the run of the envelope than on the stress-strain properties. For a better understanding of the mechanism of stabilization, other methods to determine stress-strain relation than those described here should be used. The influence of other polar and also apolar liquids on the same solid should especially be investigated.
5. Earlier investigations in this field have shown that several simplifying presumptions and suitable methods with regard to the preparation, pretreatment, and testing of the samples are necessary to keep the number of variable factors within a certain limit. In the case of an in situ treatment of the soil or rock, many additional influences are to be expected that will change considerably the picture concerning the effect of stabilization by heat treatment of kaolinite clays. To answer certain questions further investigations are necessary.
6. The results discussed here should have shown that it is possible to investigate and to characterize artificially modified rocks in the region between soils and rocks in a similar way. Therefore it seems justified to perform similar investigations on natural soils and rocks that are of great interest in civil and mining engineering.

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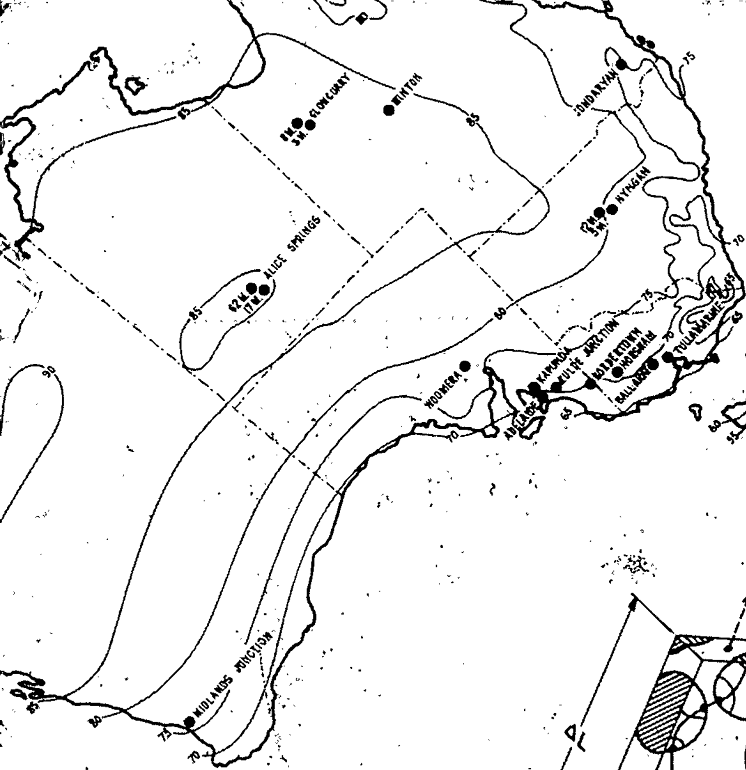
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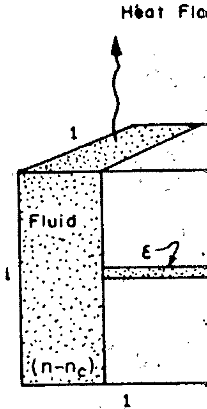
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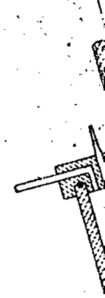

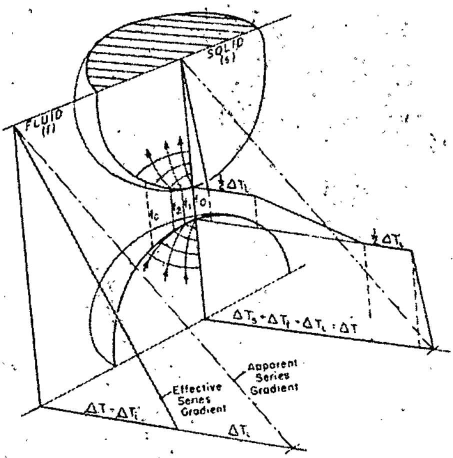
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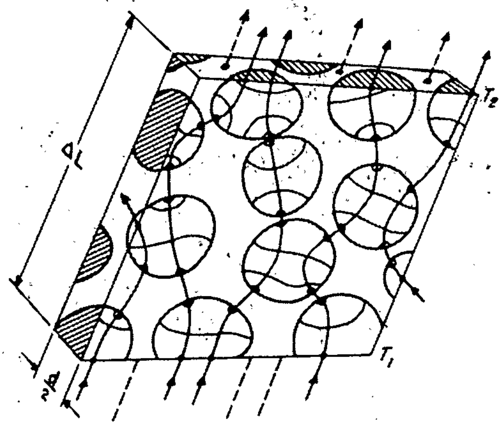
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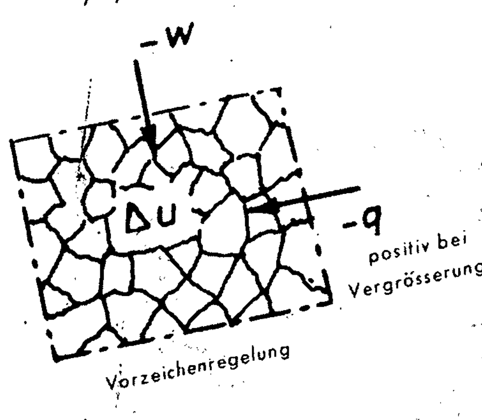
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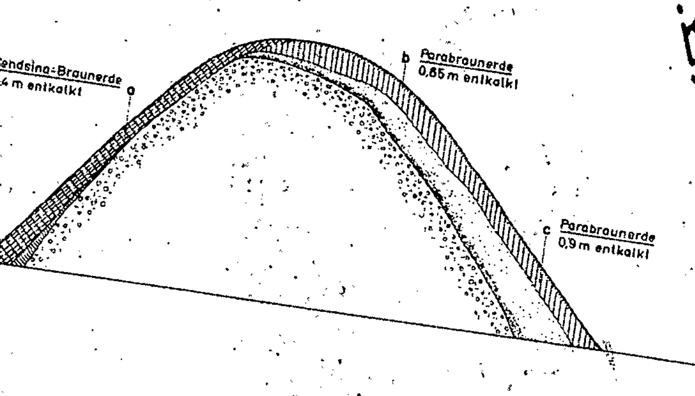
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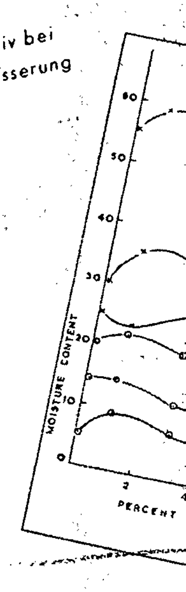
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