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Dedicated to Prof. Dr. H. J. Seifert on the occasion of his 60th birthday

THERMAL AND MICROSTRUCTURAL STUDIES ON MUD WITH ADDITIVES

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Mixtures of mud with various additives were studied to explain the reasons for the change in geotechnical properties. The additives were: lime, cement, fly ash, water-glass containing either Na_2CO_3 or $CaCl_2$ and phosphogypsum. An increase in strength was usually associated with increase of weight loss, both on static or dynamic heating. An exothermic peak occurred between 420°C and 490°C, being especially high in the presence of water -glass, together with $CaCl_2$. XRD indicated an increase in calcite content and the possible formation of calcium aluminate silicate hydrate.

SEM showed a non-homogeneous microstructure and big pores in case of mixtures of low strength (water-glass addition). A homogeneous aggregated structure was obtained in the case of higher strength (fly ash, phosphogypsum).

Mud with a high water content was planned to be used for reconstruction of river embankments in a depressive Zulawy region in the vicinity of Druzno lake. Its properties need to be improved, especially its swelling and shrinkage had to be limited. Thus some additives were studied as mentioned in the literature on Soil Mechanics and Foundation Engineering (SMFE, IC indicates International Conference). The following additives, improving geotechnical soil properties, were described in the literature:

- unslaked lime (6–10%; mixed in situ with soil by the deep mixing method) [1-4],

- high-pressure injection of lime slurry [5–7]; (lime addition causes a reduction of compressibility and an increase in shear strength and permeability of soft clays) [8, 9],
- lime-fly ash-water grout for injection in landfills at solid to water ratio 1:2 by weight and lime-to-fly ash ratios commonly of 1:3 to 1:4 (generally 1:2 to 1:10), [10],
- lignite ash with hydrated lime and cement [11],
- coal ash with 6% cement (resulting in puzzolanic reactions and formation of calcium silicate hydrates) [12],
- cement-bentonite grouting in gypseous ground [13],
- gypsum [14–16],
- gypsum and lime addition, (leading to needle-like ettringite formation) [17–18]. Increase in strength may be 10-fold in comparison to the separate addition of these compounds and also the rate of the chemical reaction during the first 3–6 months is increased [16, 19],
- polymer aluminium hydroxide $Al(OH)_{2.5}Cl_{0.5}$ which is based upon chains or hexagonal rings which can exist both in solid or liquid state. In this case the main cation is a combination of 7 hexagonal ring structures with the formula $[Al_{24}(OH)_{60}(HOH)_{24}]^{18+}$ [20],
- sodium silicate with sodium aluminate (used to form horizontal membranes) [21],
- water glass silicate with some reactive to regulate setting-hardening time [22].

The addition of resin is not considered here. A review of solid stabilization methods is given by Broms and Anttikoski [23].

In this study simple and inexpensive additives were used, i. e. lime, cement, fly ash, water-glass (also with sodium carbonate or calcium chloride addition) and phosphogypsum. A study of the microstructure and thermal behaviour was undertaken in order to explain the reasons for changes in soil properties. In the present paper we report the results of thermal studies as well as other test results which help in their interpretation.

Further information will be published in geotechnical and/or clay mineralogy literature.

Experimental

Materials

Mud from Gronovo Dolne, westwards of Druzno Lake was taken from a depth of 0.5–1.4 m. It forms a young holocene deltaic sediment of the Wisla River. Its stratum, of a thickness of about 20 m, contains some sand lenses. Its properties are similar to those of the marine clay from the bottom of the Baltic sea close to the sea shore and in both cases they are sensitive to sample pretreatment (freezing, storing in dry state etc., [24]).

The mud was taken from the ground by the ordering institution (IMUZ-Falenty), kept in a big plastic bag and the study started after several days. In analogy to the marine clay mentioned above [24] this mud showed instability in properties such as water sorption and geotechnical parameters, which changed with time of storage in wet (undried) state (Table 1).

Table 1 Geotechnical characteristics of the parent material

Property	As supplied %	After 1/2 year at W_n %
Plastic limit, W_p	30	55.6
Liquid limit, W_l	81.2	84.7
Water content, W_n	68	54
Water sorption at $p/p_o = 0.5$	3.27	3.88
Water sorption at $p/p_o = 0.95$	7.7	8.4
Values estimated from water sorption:		
External specific surface		
I. $p/p_o = 0.5$	19.1 m^2/g	22.7 m^2/g
II. $p/p_o = 0.95$	30.0 m^2/g	32.8 m^2/g
Particle thickness I.	41.4 unit layers	34.8 unit layers
Particle thickness II.	26.3 unit layers	24.1 unit layers
Montmorillonite content index	14.5%	17.2%
Organic matter from	ΔG (220°C–400°C)	1.9%

The following commercial additives were studied (percentage of additive relative to weight of oven dry mud):

Group I: active CaO

Sample 1: 5% hydrated lime, sample 2: 5% portland cement, sample 5: 5% fly ash from Konin with a high content of active calcium (15.9%) and a high organic matter content (16.9%); it was obtained by combustion of brown coal

Group II: active silica (water glass)

Sample 8: 2% Na_2CO_3 + 10% water-glass with a density of 1.35 g/cm^3, containing about 29% dry solid, sample 11: 10% water-glass, sample 12: 5% water-glass + 5% $CaCl_2$

Group III:

Sample 15: 5% phosphogypsum - a waste material of sulphur production, composed mainly of gypsum with a small phosphate content, not exceeding 4%.

Geotechnical studies can be summarized as follows:

Group I: Active CaO resulted in improved geotechnical properties. The strength, as measured by cone penetration test, increased in the order sample 1 < sample 2 < sample 5. The addition of fly ash from Konin gave the greatest increase in strength properties.

Group II: Water-glass (active silica) has an unfavourable influence on the geotechnical properties. Both the increase in water content from water-glass addition and some microstructural changes described below are the possible reasons.

Group III: Phosphogypsum had the best influence on the strength properties.

Methods

Untreated mud with the natural water content, Wn = 68%, was mixed thoroughly. The above mentioned additives were added in amounts as indicated above and the specimens were mixed again. All samples were prepared at the same time, stored and studied under the same conditions. Some geotechnical properties were measured after the sample preparation and after a certain storing time. Cubes of about 3 x 3 x 3 cm were formed (also of the parent material), they were stored for 28 days in a humid chamber and air dried at 30^o in a constant temperature cupboard, and were studied by various experimental techniques.

TG, DTA and DTG curves were recorded simultaneously with dynamic heating using a derivatograph Type 1500 QD, MOM, Budapest. The sample weight was 250 mg, the heating rate was 10 ^{o}/min, from 20 ^{o} to 1000 ^{o}, the sensitivity was 100 mg in TG (500 μV), 250 μV in DTA and 1 μV in DTG measurements the chart speed was 1 mm/min. Calcined Al_2O_3 was used as reference material.

XRD diagrams were obtained by a Siemens Kristalloflex D 500 instrument with Ni-filtered CuKα radiation. Some identification work was done with Kristalloflex D 501, equipped with a graphite monochromator CuKα and a computer SICOMP PC 16–20 with files of basal spacings of various standard minerals.

Scanning electron microscopic studies were performed using the ISI, model SS–40, equipped with KEVEX and applying the nominal thickness of 200–300 Å of gold.

Results

Weight loss on dynamic heating and DTA

Results are shown in Fig. 1 and in Tables 2 and 3. The parent mud (Nr 0) exhibited a total weight loss of 11.6% and a high dehydration between 20° and 230° (4%). Part of the weight loss between 230° and 700°, which amounts to 5.8%, may be due to organic matter and the rest to dehydroxylation of clay minerals. Between 700° and 1000° the decomposition of $CaCO_3$ present in the sample took place. The weight loss was 1.8%, corresponding to 4% $CaCO_3$ and decomposition reaction.

All the samples showed an endothermic dehydration peak between 100 ° and 120 °. Sample 15 showed another peak at 145° due to water escape from $CaSO_4 \cdot 2H_2O$ and its transformation to $CaSO_4 \cdot 1/2H_2O$ [25]. All the samples analyzed showed an exothermic peak between 305° and 325° due to the oxidation of organic matter.

All the mixtures showed a more or less pronounced exothermic peak at about 420° to 490°. This peak is especially high for samples containing some water-glass and either $CaCl_2$ or Na_2CO_3. It is attributed here to formation of new phases from the amorphous material in the mud, including the amorphous silica, contained in water-glass. In the presence of Ca ions it results most probably in a calcium silicate aluminate hydrate (amorphous or crystalline, see [26] and [27]), which influences the strength of the material.

The endothermic peak at about 550–560° corresponds to the dehydroxylation of clay minerals. The endothermic peak about 747–777 ° is due to the decomposition of calcite present in the parent material and in the additive [28].

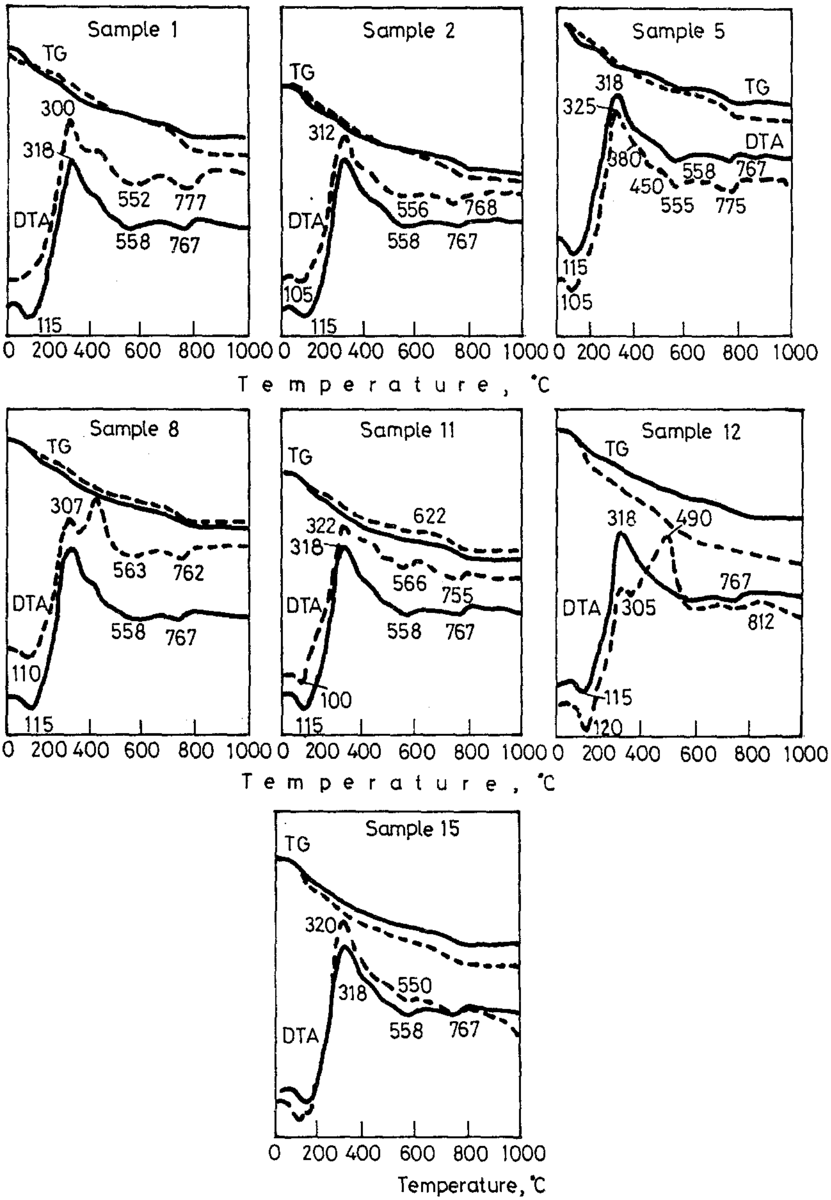

Fig. 1. DTA and TG curves of the parent material (full line) and of the mixtures (broken lines)

The total weight loss of the air dry samples is either close to that of the parent material (11.6%) or a little higher (12%–14%). Exceptions are sample 11 with a lower weight loss and sample 12 with a much higher weight loss (17.6%), which is only partly due to the higher water content of this sample (7% as compared to 2.8–4.4% of the remaining samples).

Table 2 Peak temperatures (in °C) on the DTA curves of the parent material in absence and presence of additives

Sample No	Additives	Endo	Exo	Exo	Endo	Endo	Endo
0	–	115	318		558	767	
1	5% $Ca(OH)_2$	100	305	+	552	777	
2	5% cement	105	312	+	556	768	
5	5% fly ash K	105	325	+	555	775	
8	2% Na_2CO_3 + 10% water glass	110	307	428	563	762	
11	10% water glass	100	322	418	556	755	
12	5% water glass + 5% $CaCl_2$	120	305	490	≈560	≈770	1000
15	5% phosphogypsum	105 145	320	+	≈560	747	1000

All the differences in thermal behaviour, including the highest weight loss, occurred within the temperature range 230–700°. At lower temperatures (20°–200°) the weight loss was approximately equal to the water content, thus it was due to dehydration. At more elevated temperatures (200°–700°) it was due (1) to the dehydroxylation of clay minerals at about 560° and (2) to the formation of some new phases in exothermic reaction at 420° to 490°. At about 770° there is an endothermic peak corresponding to (1) decomposition of calcite, (2) eventual destruction of the lattice of calcium aluminate silicate hydrate, either formed due to the addition of new material or present in the parent mud. This heating effect was connected with only a small change in the weight of the sample (700°–1000°), i. e. 1.8% in the parent material.

In most cases the mixtures exhibited a greater total weight loss than the parent material (see Table 3):

– by 1–2% in the case of fly ash K and portland cement (sample 2 and 5)

– by 2.6% in the case of phosphogypsum (sample 15)

– by 6.0% in the case of water-glass and $CaCl_2$ (sample 12), a part of which was due to a higher initial water content in the air dry state. Only the water-glass addition and eventual Na_2CO_3 resulted in a smaller total weight loss.

In the study of various other mixtures it was observed that the increase in weight loss on heating was accompanied by improved strength properties [29]. Also in this case favourable properties were observed with samples 1, 2, 5 and 15, as mentioned above.

Table 3 Weight loss ΔG (%) in dynamic heating of the parent material in the absence and presence of additives

Sample No	Additives	W_O from Table 3	Total ΔG 20–1000°C	Partial ΔG within temperature range °C		
0	–	3.9 %	11.60%	20–230 4.06%	230–700 5.76%	700–1000 1.78%
1	5% $Ca(OH)_2$	3.4 %	13.34%	20–130 3.52%	130–700 7.22%	700–1000 2.61%
2	5% cement	3.15%	12.06%	20–200 3.16%	200–700 7.11%	700–1000 1.78%
5	5% fly ash K	3.1 %	13.38%	20–230 3.25%	230–700 7.38%	700–1000 2.75%
8	10% water glass + 2% Na_2CO_3	2.8 %	11.29%	20–200 2.67%	200–610 6.14%	610–1000 2.47%
11	10% water glass	3.0 %	10.33%	20–200 3.00%	200–560 4.63%	560–1000 2.70%
12	5% water glass + 5% $CaCl_2$	7.0 %	17.63%	20–200 5.74%	200–630 9.71%	630–1000 2.18%
15	5% phosphogypsum	4.4 %	14.20%	20–200 4.20%	200–650 7.60%	650–1000 2.40%

When the TG and DTG curves are compared, it is surprising that in sample 8 the peak at 428° is more intense, whereas the weight loss is lower than that of the parent material, thus it may be due to phase transition. This effect is smaller in sample 11, whereas in sample 12 both the peak heights and the weight loss are the highest. Thus for this reaction the presence of both calcium ions and silica is most favourable which indicates that it may be connected with the formation of hydrated calcium silicate of various hydration states or dehydrating at various temperatures. The degree of hydration is most probably the highest in the case of sample 12 ($CaCl_2$ and water-glass as additives) as it has the highest water content in air dry state and exhibits the highest weight loss at 500–600°.

Weight loss on static heating

Three samples of 5 g of every mixture were dried for 1 day at 110°, for 8 h at 220° , heated for 4 h at 400° and for 1 h at 800°.

The weight loss within the given temperature range was calculated relative to the sample weight at 110°, i. e. weight loss between 110° and 220°, 110° and 400° and between 110° and 800° (Table 4). If the water content W_o of the air dry specimen is added to the last value, the total weight loss should be approximately equal to the total weight loss in dynamic heating, which indeed is the case. The small difference is due to the fact that ΔG (dynamic) was calculated with respect to the initial weight of the air dry sample, in which the water content is small.

Table 4 Weight loss on static heating of the parent material in the absence and presence of additives

Sample No	Additives	W_o %	110–220°C %	110–400°C %	110–800°C %
0	–	3.9 ±0.2	2.6 ±1.0	4.48±0.98	8.04±1.63
1	5% $Ca(OH)_2$	3.39±0.07	1.36±0.16	4.54±0.24	10.21±0.22
2	5% cement	3.15±0.07	3.00±0.27	4.02±0.07	9.27±0.04
5	5% fly ash K	3.08±0.11	3.78±0.14	5.39±0.30	10.01±0.11
8	2% Na_2CO_3 + 10% water glass	2.75±0.30	2.60±0.38	4.32±0.43	8.10±0.70
11	10% water glass	3.02±0.16	2.45±0.15	4.15±0.22	7.77±0.38
12	5% water glass + 5% $CaCl_2$	7.03±0.26	2.24±0.46	5.97±0.56	12.87±0.24
15	5% phosphogypsum	4.40±0.13	2.02±0.53	4.86±0.20	9.41±0.47

The weight loss (ΔG) between 110° and 220° is 2–3%, except for samples 1 and 5. Within the temperature range 110° to 400° only samples 5 and 12 exceed the average value of 4–4.9%.

The standard deviation is small (0.1 to 0.5%), except for the parent material. Mixtures 8 and 11 had a total weight loss similar to that of the parent material (ca. 8%), 2 and 15 had a somewhat higher ΔG. The highest ΔG was observed for mixture 12 with $CaCl_2$ and water-glass (ca. 13%).

As it was mentioned above a high weight loss is favourable for the strength properties, as it may indicate the formation of some microstructure, that might result in phase transformation at more elevated temperatures.

X-ray diffraction

X-ray diffraction patterns of the parent material in air dry state and after the usual treatments for clay identification [30], indicate that it is composed of smectite (partly interlayered), chlorite, illite, kaolinite, quartz, feldspar and calcite.

The mixtures present very complicated XRD patterns due to the appearance of small intensity peaks, in addition to those of the parent material and the added material. As stated in previous papers [26, 27], a calcium silicate aluminate hydrate can be formed after adding fly ash to the clay soil. The weak and broad diffractions at 12.5 Å, 2.85 Å, 1.6 Å and the increase in intensity of the diffraction at 6.28 Å may be attributed to the presence of this compound. It is probably poorly crystallized, as suggested by the low intensity and width of the peaks. The increased intensity of the diffraction peaks of the calcite in the XRD patterns of all mixtures is also remarkable.

It should be mentioned that ettringite has not been found in our specimens, contrary to the description by Kujala [31] in soils stabilized with lime and gypsum.

SEM

Air dry samples were broken to obtain a vertical surface for microscopic observation. From the many micrographs available those were chosen which indicate the formation of a new amorphous material. A magnification of 3000× was applied, thus the picture size is 33 μm× 46 μm (Fig. 2).

The untreated parent material indicates the clay matrix characteristic of illite with clay particles of variable thickness between grains of accessoric minerals (038).

Sample 1 is composed of thicker and less flexible particles which are cemented and stuck together in bigger aggregates, mainly in face-to-face contacts, but there are also aggregates in edge-to-face orientation. A grain of the newly formed material of poor crystallinity is visible in the centre of the picture and inside it the particles have a tendency of parallel arrangement (043).

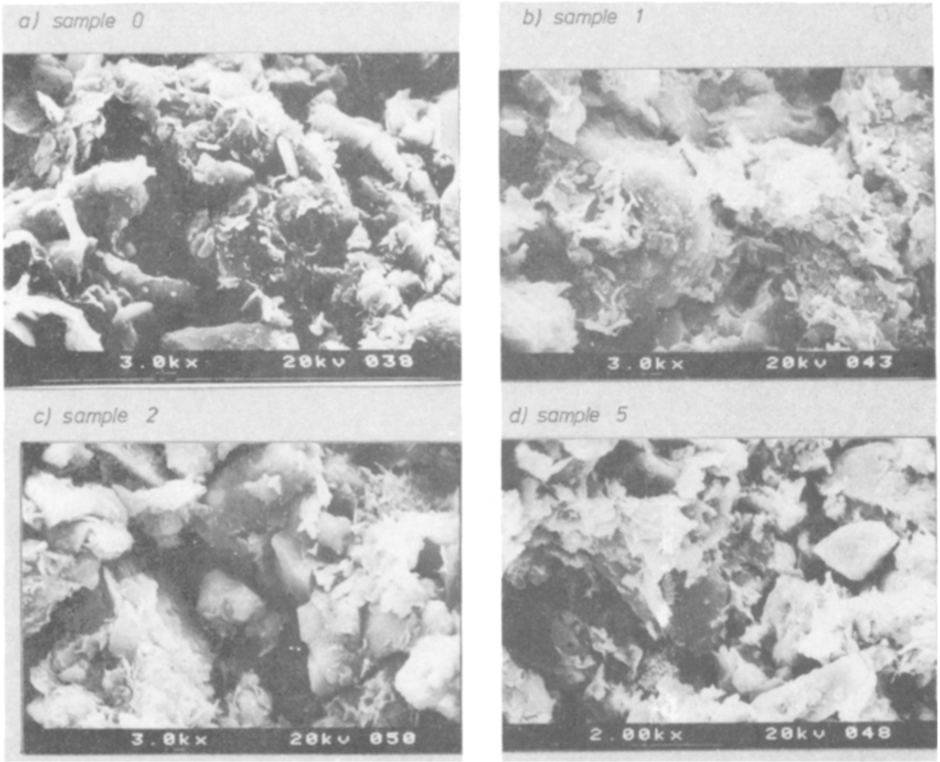

Fig. 2a–d Scanning Electron Micrographs of the parent material (*a*) and of the mixtures: (*b*) sample 1, (*c*) sample 2, (*d*) sample 5
Note: the size of SEM photographs is 33x46 μm, except (d) sample 5 of the picture size 50x70 μm

Sample 2 indicates also a pronounced cementation, the presence of big grains and of pores between them in the form of channels. Clay particles are aggregated around or between some relatively big grains, which have formed after cement addition, as they are more numerous than the amount of material added and than their proportion in the parent material (050). The aggregates between grains have edge-to-face orientation of particles.

Sample 5 shows a similar picture. Here the clay matrix seems more homogeneous, the grains are less numerous and they are smaller, though it should be noted that the magnification here is 2000×, thus the picture size is 50 μm × 70 μm (0.48).

Sample 8 has very big grains separated from the clay matrix by big pores. Clay particles are sticking together in a dense arrangement, mainly face-to-face. The whole grain at the lower right edge of the picture, if shown completely, would exceed the whole surface of the picture (33 × 46 μm), (059).

Particles in mixture 11 are completely "glued" together by the added water-glass. Big grains of a possible new phase are also visible here, but they are not as well formed as in sample 8. Practically no separate particles can be seen in the clay matrix, all of them are stuck together in face-to-face orientation (066).

To a lesser extent the same is seen in mixture 12, as the water-glass addition was smaller here, and it was bound by calcium chloride, probably into calcium silicate hydrate. Similar grains of gel-like calcium silicate hydrates were observed in aged cement-laggoned ash mixtures [12], see also [32] and [33].

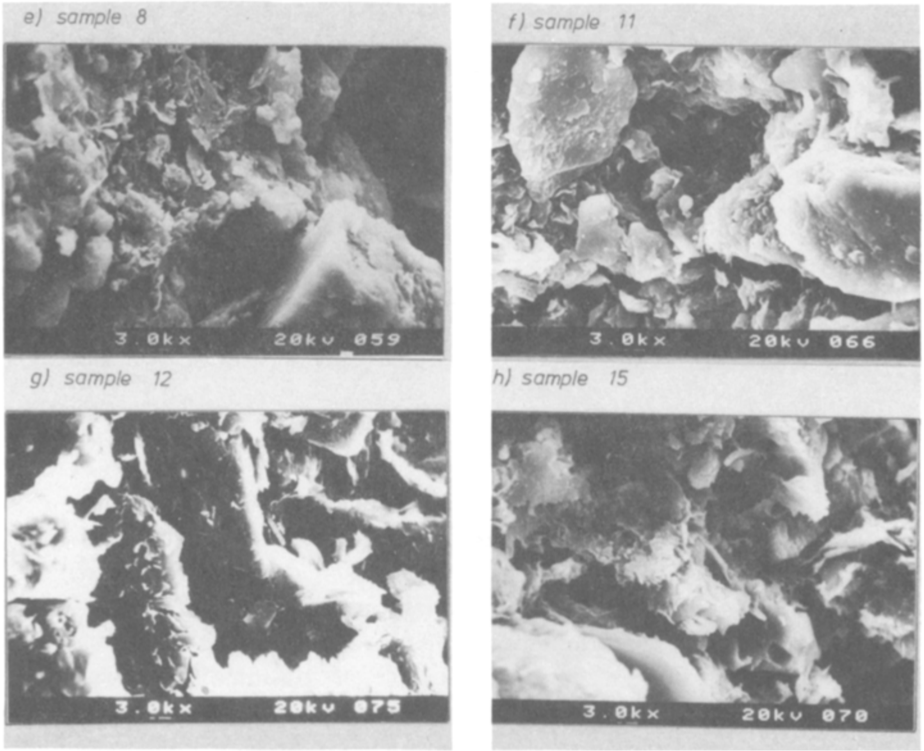

Fig. 2e–h Scanning Electron Micrographs of the mixtures: (*e*) sample 8, (*f*) sample 11, (*g*) sample 12, (*h*) sample 15
Note: the size of SEM photographs is 33 ×46 μm, except (*d*) sample 5 of the picture size 50 ×70 μm

The grains are relatively small and covered by clay particles, thus they may be just their agglomerations. Also the matrix seems to have a more crystalline form as composed of thicker and bigger particles (075).

The matrix in sample 15 is the least influenced by the additive, though here also the particles become bigger, thicker and stiffer. These domains or aggregates are glued together by edge-to-face contacts. Pores are big, elongated, channel-like, but they are smaller than in the other samples. A nicely formed crystal of gypsum morphology was found in the clay matrix (not shown here) (070). No needles of ettringite ($3CaO \cdot Al_2O_3 \cdot 3CaSO_4..32H_2O$) weight detectable in sample 15 either by XRD or by SEM. Such needles were found by Kujala [17], [18] and [31] in gypsum-lime stabilized soil and the corresponding XRD peaks were detected. Probably in our case the content of calcium ions was not high enough for this compound to be formed.

Discussion

Indications were obtained that the increase in strength of the parent material, when mixed with an additive, is due to :

– formation of calcite, which may precipitate on the particle edges, linking them into aggregates and thus increasing their strength. The increase in calcite content was found by XRD in all the mixtures under study,

– formation of cementing material, most probably a calcium silicate aluminate hydrate, of the formula $Ca_2Al_2SiO_7 \cdot 8H_2O$, as indicated by XRD, but it was not unambiguously proved,

– the addition of active Ca in proper (optimum) amount causes an improvement of strength properties and increase in weight loss on heating. Such a proper amount of active Ca was added in form of the fly ash, which induced the best improvement of the material under study,

– the addition of amorphous silica in the form of water-glass results in a decrease of the weight loss on heating but some exothermic reaction occurs at 420°–490°. A compound containing little calcium was formed which was not introduced into the mixture, but was present in the parent material,

– the addition of amorphous silica and calcium chloride results in the formation of a compound having the highest exothermic peak at 490° and the greatest weight loss. Most probably this is the production or crystallization of the calcium silicate hydrate or calcium silicate aluminate hydrate, which calcium was mentioned above. The new phase, or the amorphous material, from which this new phase may form, are visible is SEM,

– the addition of water-glass and other components to the parent material results in a small decrease in strength. This may be explained from the SEM pictures: at such a high water content as in the parent material under study, the additive that induces a high strength, causes the collapse of the microstructure into big grains of a high strength with big macropores between them. Thus the external load does not need to deform the grains, it may cause only their mutual displacement to deform the whole system. Thus it is understandable that mixtures 8, 11 and 12 had a low strength,

– a higher strength was shown by mixtures 5 and 15 of a more homogeneous distribution of structural elements and of smaller pores between them than in case of other mixtures, which were less homogeneous, though the strength of their aggregates could have been high.

In summary it may be stated that the increased strength of the mixtures under study is due to cementation of the matrix by calcite and probably also by calcium aluminate silicate hydrate which are formed in the mixture. The cementing agent should be added to the investigated soil in a proper amount, which is especially important if the water content is high: if the amount is too high, a collapse may occur to big aggregates, separated by broad pores, which induces an easy deformability of the system, and hence a small strength. The amount of the additive may be monitored by checking the weight loss on heating and the exothermic effects at 420°–490°. XRD and SEM studies support this statement.

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Zusammenfassung — Gemische aus Schlamm mit verschiedenen Additiven wurden untersucht, um Änderungen in geotechnischen Eigenschaften zu erklären. Diese Additive waren: Kalk, Zement, Flugasche, Wasserglas mit entweder Na_2CO_3 oder $CaCl_2$ und Phosphogips. Die Zunahme der Festigkeit war stets mit der Zunahme des Gewichtsverlustes verbunden, sowohl bei statischem als auch bei dynamischem Erhitzen. Zwischen 420°C und 490°C konnte ein exothermer Peak beobachtet werden, der in Gegenwart von Wasserglas mit $CaCl_2$ besonders hoch war. Röntgendiffraktion weist auf ein Ansteigen des Calcitgehaltes und eine mögliche Bildung von Calciumaluminatsilikat-Hydrat hin.

SEM zeigte ein inhomogenes Mikrogefüge und große Poren im Falle von Gemischen geringerer Festigkeit (Zusatz von Wasserglas). Im Falle höherer Festigkeiten (Flugasche, Phosphogips) wurde eine homogenes aggregiertes Mikrogefüge erhalten.