THE FABRIC OF SOIL–CEMENT AND ITS FORMATION

by

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ABSTRACT

THE electron microscope has been used to study changes in particle size, shape, and arrangement that occur during the hydration of clay in soil–cement. Samples of kaolinite, of a mixture of silica flour and montmorillonite, and of a natural silty clay were mixed with sufficient portland cement to produce soil–cement. Specimens were cured at constant moisture content for periods up to 32 weeks after compaction at optimum moisture content for the mixture. At the end of the curing period surfaces of fresh fractures were replicated, and the replicas were studied using the electron microscope.

All three mixtures showed similar behavior. Initially the fabric is one of separate portland cement grains distributed throughout the clay soil. As hydration of the cement proceeds, cement hydrate gel forms along the edges of groups of clay particles. Reaction between the soil and the cement is observed early in the hydration period. As hydration continues the soil grains are more and more broken down and the cement gel diffuses more extensively throughout the mass. Eventually, the breakdown of the soil minerals and the formation of hydration products reaches a point where the soil and cement can no longer be distinguished as separate phases.

INTRODUCTION

BECAUSE of the very extensive use of soil–cement as a construction material for roads, airfields and other purposes, many laboratory and field studies have been carried out in connection with evaluation of its properties and performance. The Highway Research Board Bulletin 292 (1961) presents an excellent compilation of information resulting from such studies. More recently a number of studies aimed at better defining the reactions and interactions developing during the hydration of soil–cement and soil–lime mixtures have been carried out to develop improved methods of treatment (Goldberg and Klein 1952; Eades and Grim, 1960; Moh, 1962; Glenn and Handy, 1963; Herzog and Mitchell, 1963; Diamond, White and Dolch, 1964; and El Jack, 1965).

These studies established quite clearly that interactions develop between the hydrating cement and soil minerals. Most of these investigations have involved the use of chemical, thermal, and X-ray diffraction analyses, along with the determination of mechanical properties. The results of such studies have been used to infer details of morphological and compositional changes during hydration.

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Recently the electron microscope has been used to examine clays after treatment with lime by Diamond, White and Dolch (1964), and Sloane (1964). Little use has been made heretofore, however, of the electron microscope for the study of soil–portland cement mixtures.

This paper reports the results of an electron microscope study of soil–cement samples examined after different periods of moist curing. Emphasis is placed on the fabric of soil–cement and its changes during curing with consideration given to particle morphology, the distribution of clay, cement, and reaction products, and conditions at interfaces between clay and cement particles.

ACKNOWLEDGMENTS

METHODS

Sample Preparation

The clays used in this study were:
1. Kaolinite (K): a fine grained Georgia kaolinite distributed by the Georgia Kaolin Company as Hydrite UF.
2. A mixture of 80% silica flour and 20% sodium montmorillonite (Wyoming bentonite) (SM).
3. Vicksburg silty clay (VSC): a tan silty clay from Vicksburg, Mississippi.

Properties of these clays are given in Table 1 and grain size distribution curves are shown in Fig. 1. A Type I low alkali portland cement (P) was used. The amounts of cement required to produce soil–cement* were 13, 15 and 24% by weight using the Vicksburg silty clay, silica flour–montmorillonite, and kaolinite, respectively.

Cylindrical samples of soil–cement 1.4 in. in diameter and 3.5 in. in height were prepared at optimum moisture content using kneading compaction. Thorough, controlled mixing of the soil, cement, and water was carried out prior to compaction. After compaction samples were cured at constant water content for periods of 1, 4, 12 and 32 weeks. Control specimens of neat portland cement paste with a water : cement ratio of 0.4 were also prepared.

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* Soil–cement is defined as a hardened material formed by curing a mechanically compacted, intimate mixture of pulverized soil, portland cement and water. Durability as measured in standard wet–dry and freeze–thaw tests as specified by American Society for Testing and Materials designation D559–57 and D560–57, is used as the basis for determination of the amount of cement required.

TABLE 1.—Properties of Soils Investigated

Properties	Kaolinite K	80% silica flour plus 20% montmorillonite SM	Vicksburg silty clay VSC
Textural composition,* % by weight			
Sand: 0.06 mm to 2 mm	—	9	36
Silt: 0.002 mm to 0.06 mm	—	57	42
Clay: finer than 0.002 mm	100	24	22
Physical properties			
Liquid limit, %	63	98	35
Plastic limit, %	36	34	24
Plasticity index, %	27	64	11
Soluble salts, meq/100 g	0.85	3.4	0.70
pH	5.5	8.4	7.2
Cation exchange capacity, meq/100 g	10	16	14
Specific gravity	2.63	2.65	2.73
Max. dry density,† lb/cu. ft.	98.2	103.9	104.0
Opt. water content,†	34.4	21	17.2
Cement, % by wt.‡	24	15	13

* M.I.T. classification system.

† By standard proctor compaction test.

‡ According to Portland Cement Association and Americn Society for Testing and Materials criteria for soil cement.
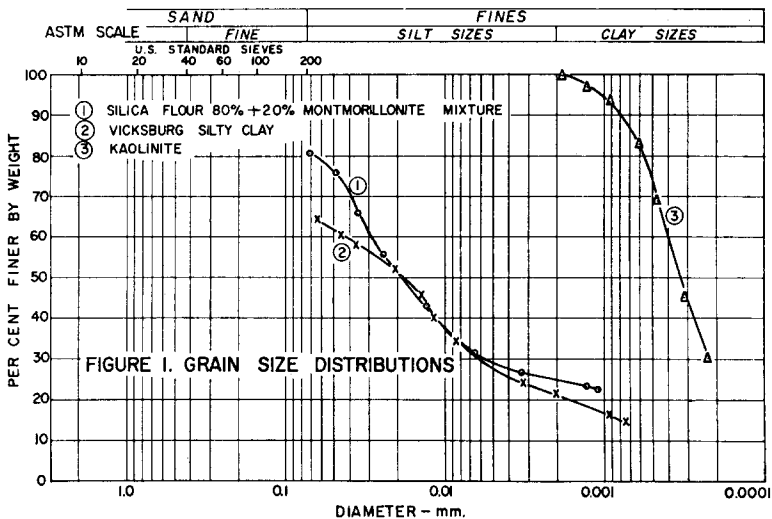

FIG. 1. Grain size distributions.

At the end of the curing periods determinations of strength, moisture content and volume change were made, and samples were prepared for study with the electron microscope. The variation of unconfined compressive strength with curing time is shown in Fig. 2. The progressive increase in strength with curing time indicates that cement hydration was proceeding normally.
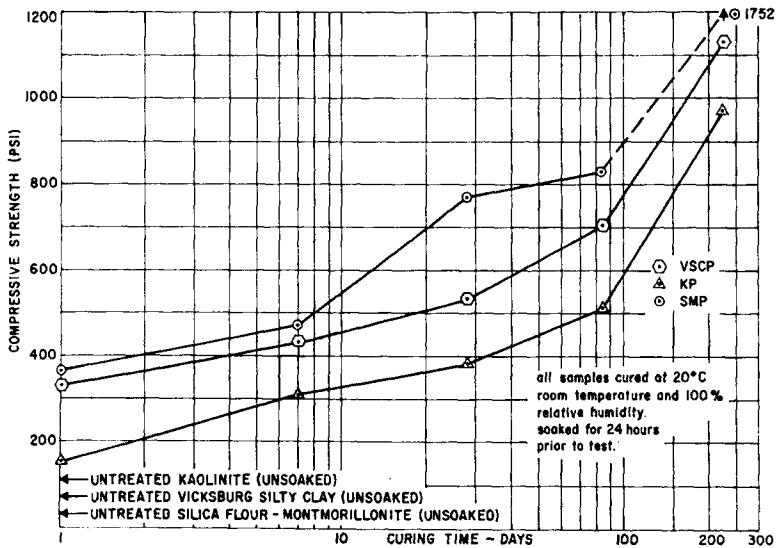

FIG. 2. Variation in strength with curing time.

Preparation of Replicas for Electron Microscope Study

At the end of the desired curing periods samples of untreated compacted soil, soil–cement, and neat cement paste were air-dried. Specimens were then fractured in directions parallel and perpendicular to the direction of compaction. The fresh fracture surfaces were blown free of loose particles and were then ready for replication.

A negative replica of the fracture surface was obtained by pressing it against a polystyrene disc which had been softened on a hot plate at 160°C. The polystyrene disc was then placed face down on 48% HF to remove any adhering clay particles. The disc was thoroughly rinsed with distilled water and air-dried.

The polystyrene impression was platinum shadowed at an angle of 30–45° in a Mikros VE-10 evaporator, followed by evaporation of a carbon film onto the specimen. The surface film thus deposited was scored into 2×2 mm squares and the polystyrene was dissolved away using ethylene dichloride.

The surface replicas were picked up on standard 3 mm diameter No. 200 copper electron microscope specimen grids.

At least two hundred surface replica grids were studied in detail using a Hitachi HS-6 electron microscope. Four grids from each surface were thoroughly scanned and about 300 micrographs were taken. The interpretation of the results was aided by additional analyses not described in this paper. These were single crystal electron microscopy and electron diffraction, X-ray diffraction, and chemical analyses for pH, soluble salts, free $Ca(OH)_2$, and sodium, potassium, and calcium content (El Jack, 1965).

RESULTS

The micrographs (Plates 1 through 13) were selected to illustrate the different types of structure that were observed to develop during the hydration of soil–cement. While they are representative of the various specimens observed, it cannot be stated categorically that they are typical of all zones within the material. All replicas were prepared from fracture surfaces, which may reflect zones of weakness through the specimens. Thus it is possible that the surfaces examined are more representative of weak spots than strong spots. While it is impossible to refer to the homogeneity of the fabric without reference to scale, the fabric in most cases was reasonably homogeneous when observed to a tens of microns scale, although some inhomogeneous areas were also observed.

Portland Cement Paste

Micrographs of fracture surfaces through wet cement paste after curing periods of 1, 4, and 32 weeks are shown in Plates 1, 2 and 3. At the end of 1 week the structure, which was composed initially of discrete silt-sized cement grains, is essentially a network of closely interconnected particles of very irregular morphology. Calcium silicate hydrates (CSH), of both high and low Ca : Si ratio, and calcium hydroxide could be detected at this stage.

At the end of 4 weeks (Plate 2) the structure contains both elongated, needle-like, lime-rich CSH (II) particles indicated by "A" and surrounded particles and fibers of CSH (I) of low Ca : Si ratio which tend to concentrate along the edges of the bulky particles. After 12 weeks needle-like particles are no longer present and the structure contains wrinkled and randomly distributed chains of fibrous gel. The fabric appears markedly different at the end of 32 weeks (Plate 3). Larger particles of irregular shape have formed that appear to be welded closely together, and different phases have become virtually indistinguishable.

Kaolinite–Cement

Untreated kaolinite particles were observed to be regular very well-crystallized hexagonal flakes. Plate 4 shows the fabric of a fracture surface through a compacted sample of untreated kaolinite. The particles are seen

to be densely packed and oriented predominantly in the plane of the photograph. This orientation may have been a consequence of shear deformations during compaction. It cannot be concluded that similar orientation existed throughout the sample, because the fracture surface may have followed a path of weakness as would result from orientation of the type shown. This is quite likely because the fabric appeared the same whether the fracture was made in the vertical or horizontal direction through the sample. The particles in Plate 4 appear somewhat more irregular than those observed prior to compaction. This may be a consequence of particle breakdown during compaction.

Plates 5, 6, and 7 show the progressive changes in structure that have developed at the end of 1, 12, and 32 weeks of curing of the kaolinite–cement. At the end of 1week (Plate 5) irregular elongate particles from the hydrating cement are in close alignment with the edges of the kaolinite particles. Virtually all of the clay particles have been degraded as a result of inter-action with the cement. Attack along the edges is pronounced. At the end of 4 weeks the same general pattern was observed and the average size of the kaolinite particles was further reduced. The reaction products appear to interlock the chemically attacked kaolinite.

After 12 weeks curing further chemical attack on the kaolinite particles is evident. Plate 6 shows the dark elongate particles to be less abundant than at earlier ages. They appear to have transformed in part to irregular plate-like particles. Distinction between the cement and kaolinite phases is virtually impossible at the end of 32-weeks curing (Plate 7). The calcium silicate hydrate cement hydration products, the degraded clay particles, and new compounds formed during hydration appear to have blended into a homogeneous mass.

Silica Flour–Montmorillonite–Cement

Typical micrographs illustrating the structure of the silica flour–mont-morillonite–cement mixtures are presented in Plates 8 through 11. Fracture surfaces in the untreated soil showed randomly distributed, thick, angular particles of silica flour which range in size from 2 to 75 microns, although no particles larger than about 5 microns appeared in the micrographs. The montmorillonite particles, which comprised 20% of the mixture, are fluffy with indefinite size and shape. Little anisotropy of structure as a result of compaction was observed.

After cement addition and curing for 1 week, cement hydration leads to the formation of elongate tobermorite-like gels, CSH (I), which form around the edges of the soil particles, producing a compact structure (Plate 8). Plate 9 shows that considerable chemical attack on the silt particles developed during the first week of curing. After 4 weeks (Plate 10) the CSH fibers binding the soil particles together have become more abundant and distinct mont-morillonite particles could no longer be observed.

At the end of 12-weeks curing, broken fragments of particles present at the end of 4 weeks appeared to be bonded into aggregates. Individual particles in the aggregates were smaller in size than the original silica particles in the untreated mixture. After 32 weeks both the soil and cement particles are broken down and intimately welded together (Plate 11). At this stage distinction between the cement and soil is difficult.

Vicksburg Silty Clay–Cement

The Vicksburg silty clay, representative of soils used in field soil stabilization projects, was included in the present study to determine whether natural soils behaved in a manner similar to the simpler clay–cement systems discussed above. The dominant minerals in this soil, in order of decreasing abundance are quartz, illite, halloysite, and montmorillonite.

The structure of a compacted specimen of untreated soil was composed of randomly oriented aggregates of particles. No significant anisotropy was introduced during kneading compaction at optimum moisture content. The effect of cement treatment on the structure is quite similar to that observed in the kaolinite–cement and the silica flour–montmorillonite–cement test series.

Plate 12 shows that at the end of 1 week irregular, elongate cement gel particles are distributed around soil particles bonding them at the edges. At the end of 4 weeks (Plate 13) chemical attack on particles may be seen and the cement and soil phases are much less distinct. Packets of parallel particles oriented normal to the plane of the photograph may be observed at the top and right edge of the photograph. At the end of 32 weeks separate cement and soil phases were virtually indistinguishable.

DISCUSSION

These results, taken in conjunction with the results of electron microscope observations and electron diffraction of single crystals, X-ray diffraction, and various chemical and mechanical tests (El Jack, 1965), suggest the following sequence of compositional and fabric changes accompanying the hardening of soil–cement.

At early stages of curing low $Ca : Si$ ratio calcium silicate hydrates begin to form. Lime liberated during hydration of the portland cement enters into almost immediate reaction with the clay particles resulting in rapid consumption of lime and breakdown of the clay phase. As time of curing increases, the abundance of cement hydrate increases. Some of the soil particles become poorly crystalline and may lose their identity completely.

The fabric changes developing during the curing of soil–cement lead from a mixture of discrete soil particles and cement grains to a more homogeneous fabric of indistinguishable components. Even a very well mixed soil–cement must initially appear non-homogeneous on a micron scale because many of the unhydrated cement grains are several microns in diameter; the clay
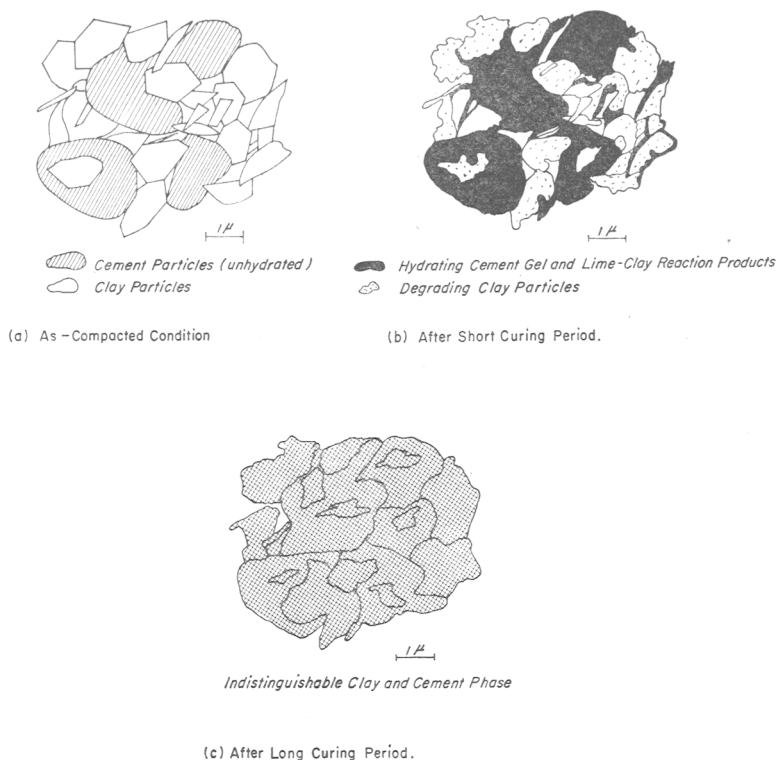
Cement Particles (unhydrated)
Clay Particles

Hydrating Cement Gel and Lime-Clay Reaction Products
Degrading Clay Particles

(a) As–Compacted Condition

(b) After Short Curing Period.

Indistinguishable Clay and Cement Phase

(c) After Long Curing Period.

FIG. 3. Schematic diagram of changes in clay–cement structure during curing.

particles are generally less than 2 microns in size. Figure 3 illustrates schematically the general sequence of changes that may take place.

As hydration of the cement begins, cement gel forms along the edges of groups of particles, probably diffusing away from the initially unhydrated cement grain. Simultaneously, lime liberated from the hydrating cement begins to break down the soil particles. Additional calcium silicate hydrates can form from breakdown of clay particle surfaces and calcium aluminum silicate hydrates may develop at particle edges.

As these processes progress the structure becomes more and more inter-laced with cement gel, particle surfaces become more irregular and the mixture appears more compact. Ultimately, the breakdown of the soil particles and diffusion of the cement gel progresses to the point where the separate phases are virtually indistinguishable.

These changes in composition and fabric are reflected by the mechanical properties of the soil–cement in terms of increases in strength and durability as curing proceeds. The various hydration processes may continue for very long periods of time, but at a continuously decreasing rate.
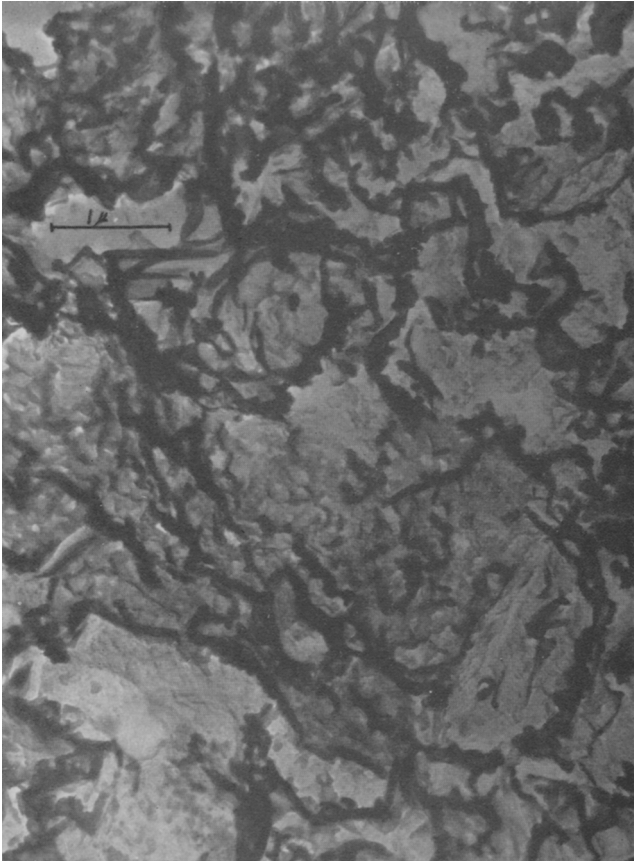
PLATE 1. Fabric of portland cement paste after curing for 1 week.
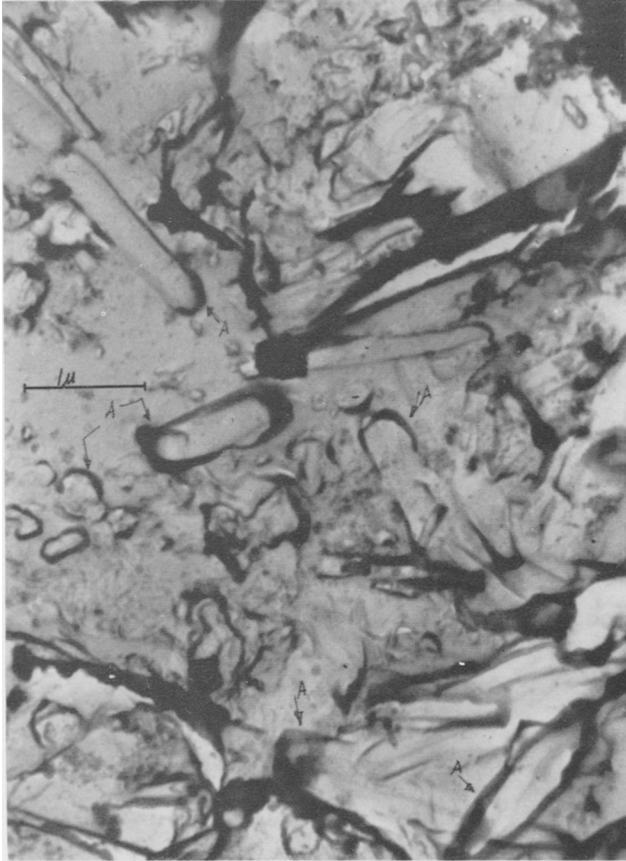
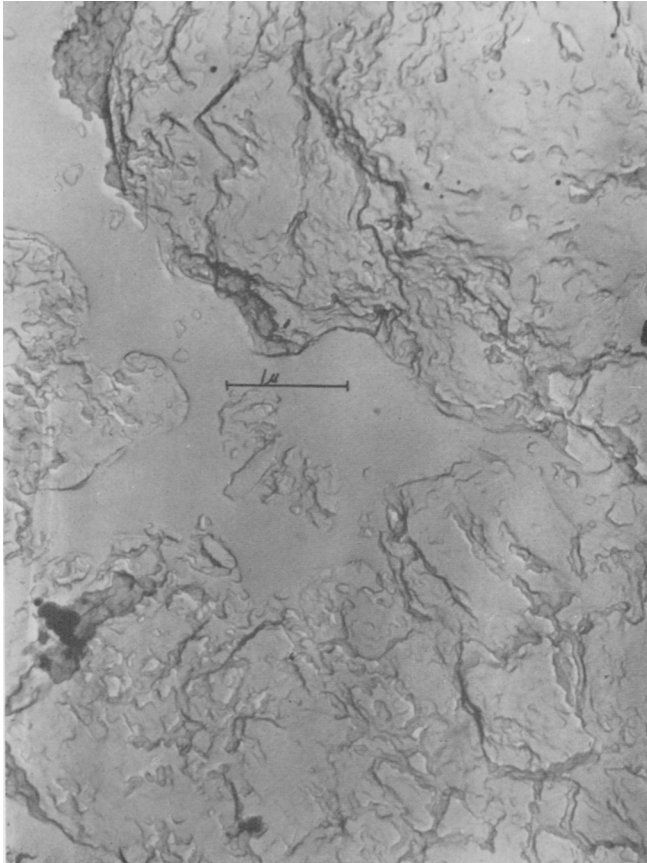PLATE 2. Fabric of portland cement paste after curing for 4 weeks.

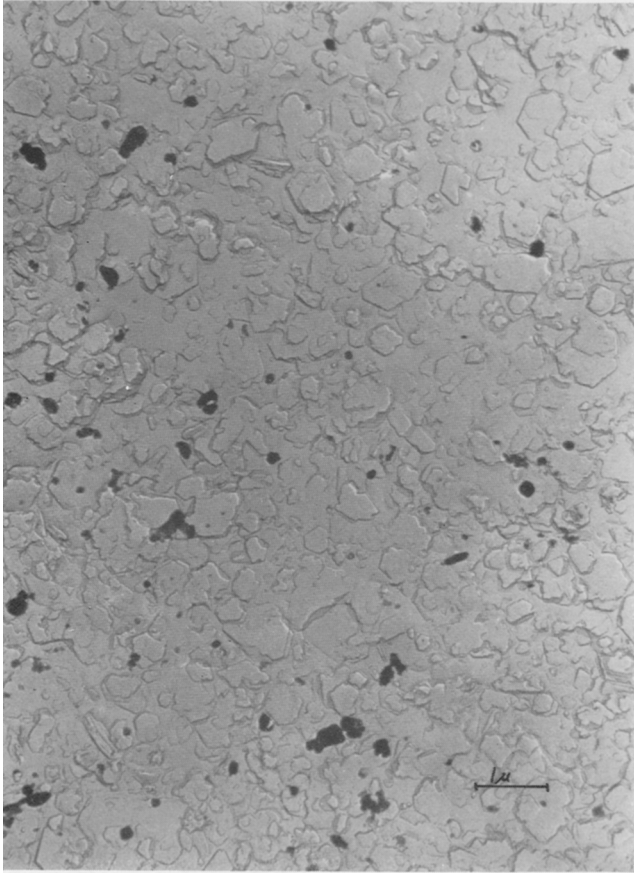PLATE 3. Fabric of portland cement paste after curing for 32 weeks.

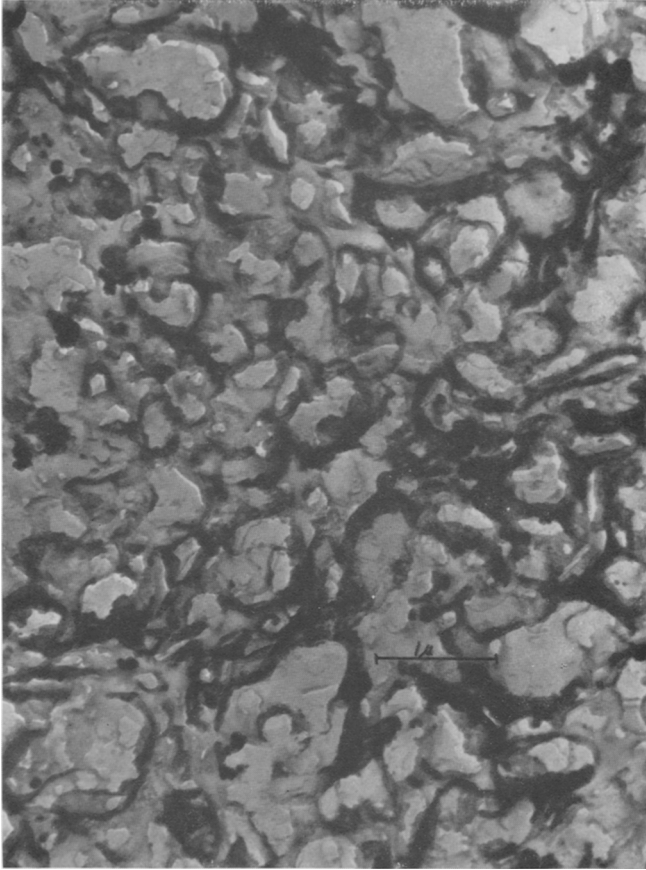PLATE 4. Fabric of untreated compacted kaolinite.

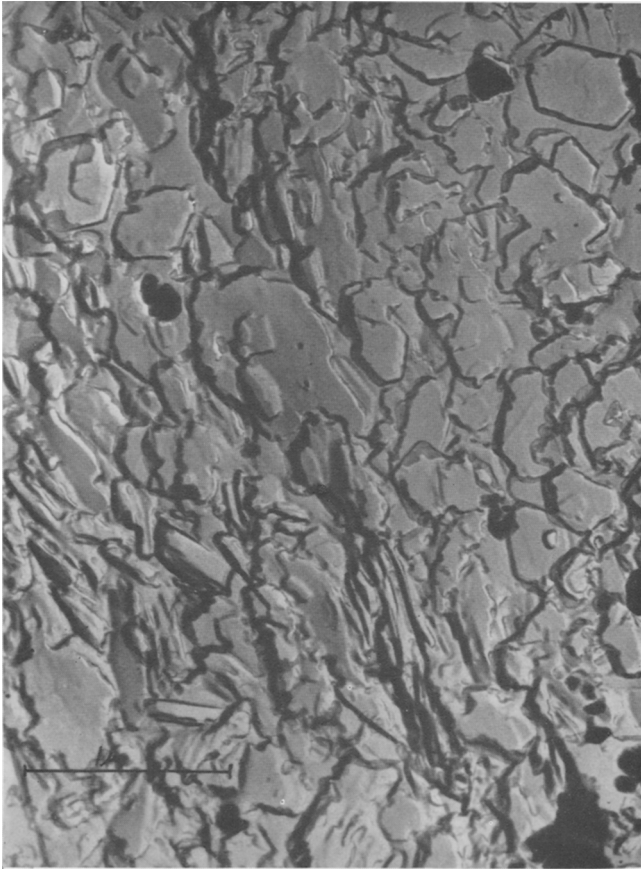PLATE 5. Fabric of kaolinite–cement after curing for 1 week.

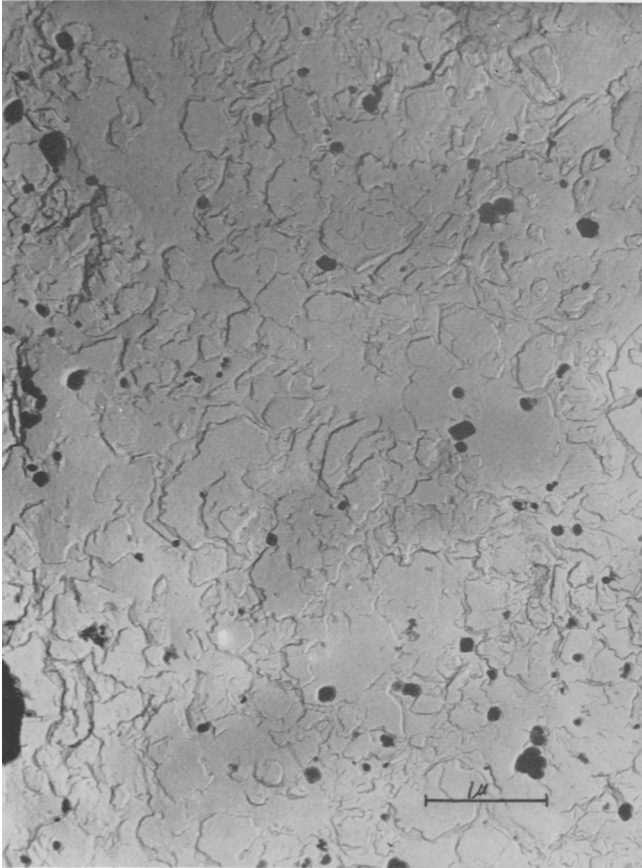PLATE 6. Fabric of kaolinite–cement after curing for 12 weeks.

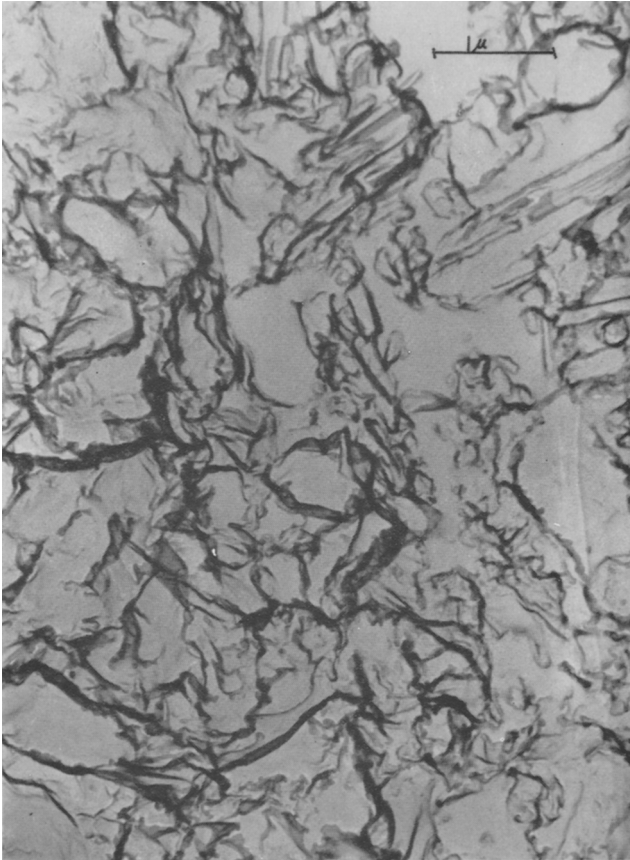PLATE 7. Fabric of kaolinite–cement after curing for 32 weeks.

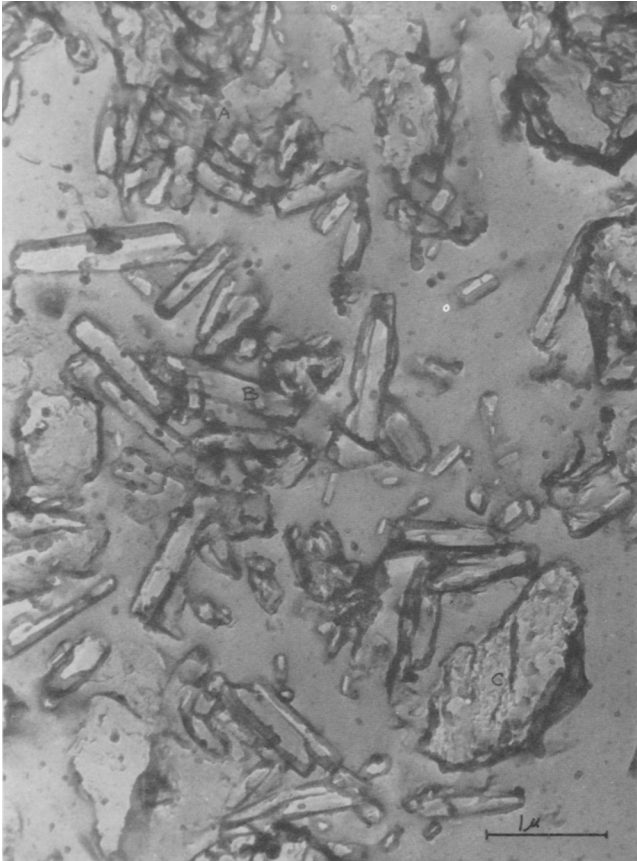PLATE 8. Fabric of silica flour–montmorillonite–cement after curing for week.

PLATE 9. Fabric of silica flour–montmorillonite–cement after curing for 1
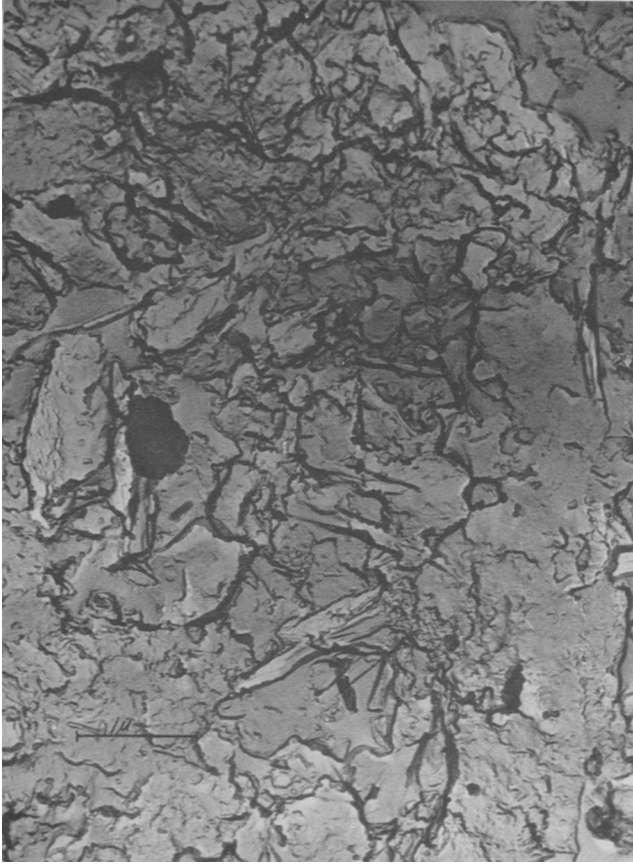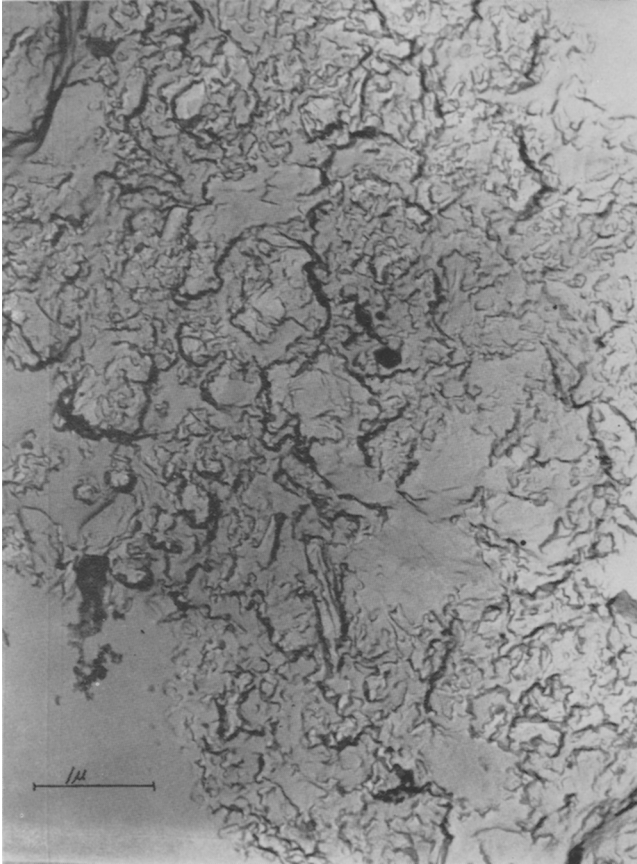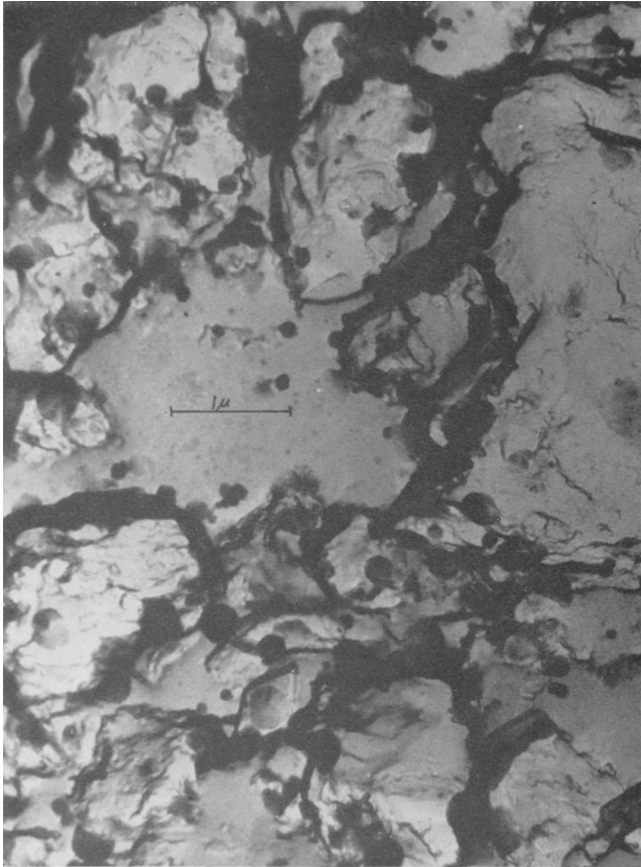week showing degraded particles.

PLATE 10. Fabric of silica flour–montmorillonite–cement after curing for 4 weeks.

PLATE 11. Fabric of silica flour–montmorillonite–cement after curing for 32 weeks.

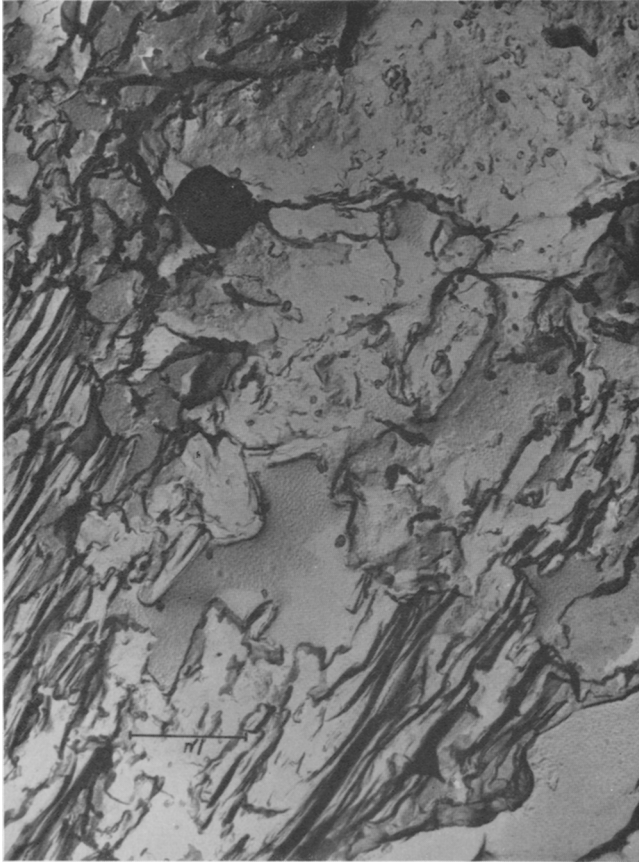PLATE 12. Fabric of Vicksburg silty clay–cement after curing for 1 week.

PLATE 13. Fabric of Vicksburg silty clay–cement after curing for 4 weeks.

SUMMARY

Examples have been given to illustrate the structural changes that develop during the hydration of clay–cement. Sufficient portland cement was mixed with kaolinite, with a silica flour–montmorillonite mixture, and with a natural silty clay to give soil–cement as defined by durability test criteria. Replicas of fracture surfaces through specimens cured for different times were studied in the electron microscope. The results show that the mixture of soil and cement changes from an initial fabric of discrete particles to a homogeneous, cemented fabric of indistinguishable components after prolonged periods of curing. Chemical attack on the soil particles by the cement components, mainly lime, results in the formation of additional cementitious material. No significant preferred orientation of platy clay particles was observed in either the untreated or the treated materials after sample preparation at optimum moisture content using kneading compaction.

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