Effect of Petrographic Variations of Southwestern Iowa Loess on Stabilization with Portland Cement

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● LOESS, the dominant surface deposit in Iowa and in areas of adjoining states, is the first soil material to be studied for stabilization purposes by the Iowa Engineering Experiment Station. In general, loess areas are areas where shortages of aggregate exist or are expected, and a method of loess stabilization is highly desirable. From a scientific point of view loess is an excellent material for study because preliminary studies show that its properties vary gradually over wide areas, and the effects of these variations on stabilization problems may be evaluated.

The largest deposits of loess in North America are Wisconsin in age and are located in the southwestern Iowa area shown in Figure 1. The detailed property and stabilization studies are being made on loess from this area. By means of similar, somewhat - less - detailed studies, the loesses in other areas of Iowa will be compared to the southwestern Iowa loess so that their behavior may be predicted to some degree.

Properties of the southwestern Iowa loess vary in general, from west to east: The loess becomes finer textured and contains more clay, the carbonate content decreases, and the thickness of the deposit diminishes. Figure 2 is a tentative map showing the areal variations in clay content. On the basis of data from the loess-property-variation studies (1, 2, 3) four loess samples were chosen as representative of variations in the Wisconsin loess of southwestern Iowa. The sample locations are shown in Figure 1, and a brief description of each sample is given in Table 1.

PETROGRAPHIC METHODS

Particle Sizes

Mechanical analyses were performed on the four loess samples by hydrometer and pipette methods which have been described (4, 5). Sodium metaphosphate was used as the dispersing agent.

Mineralogical Composition

Silt Fraction. Each loess sample was dispersed in water and separated into size fractions with a rising-current elutriator described in Appendix A. Samples from each size fraction above 5 microns were then separated into light and heavy minerals by allowing the grains to float or sink on bromoform (sp. gr. 2.87). Because the particle sizes are so small, centrifuging with a special centrifuge tube, described in Appendix B, was used to speed the separations. Light minerals were mounted in Canada balsam, heavy minerals in piperine, and identifications made under a petrographic microscope. Mineral percentages were determined by a statistical measurement of grains made with a Leitz integrating stage.

Clay Fraction. Minus -2 -micron material was separated from each loess sample by removing a layer from a suspension and drying (6). The clays were then subjected to a number of determinative tests, including differential thermal analysis, X-ray diffraction, clay mineral staining tests (7), cation exchange determinations, particle - size measurements, and measurements of optical properties. The test methods and data on the loess samples have been recently summarized (8).

Grain Shape and Roundness

Sphericity is an expression for grain shape and was estimated for individual grains by visual comparison with charts showing grains of known sphericity (9). The charts are based on Krumbein's (10) formula that sphericity is equal to grain width divided by grain length. Averages for a number of grains in each loess fraction were calculated.
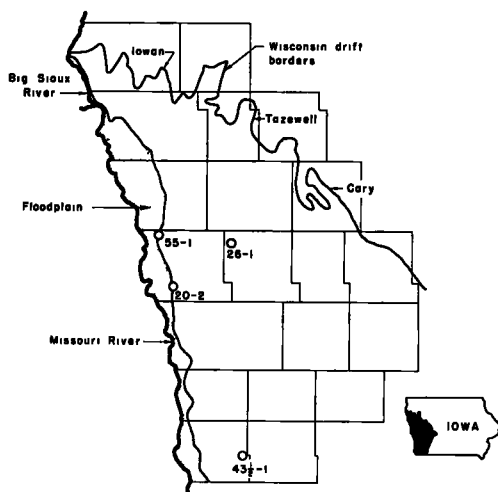
Figure 1. The Wisconsin loess area in southwestern Iowa, showing locations of the four samples selected for detailed studies.

Roundness refers only to the relative sharpness of grain corners and was noted qualitatively for a number of grains.

Chemical Tests

Chemical tests performed on the four loess samples include the following: (1) cation exchange capacity, by an ammonium acetate method (11, 12); (2) exchangeable cations, determined with a flame photo-meter; (3) pH, by an electrometric method (15g soil in 30 ml water); (4) carbonate content expressed as percent $CaCO_3$, by a versenate method; (5) sulfate content, by a water-soluble sulfate determination (13); (6) chloride content, by Mohr's method; (7) free iron oxide content, by Jeffries' method (14); (8) total iron content (15); and (9) organic matter content, by a dichromate oxidation method (13).

PETROGRAPHIC DATA

Particle Sizes

Particle-size accumulation curves for the four loess samples are presented in Figure 3. Curves for other Wisconsin loess samples from southwestern Iowa are similar in shape, and most would fall between the limits shown.

Mineralogical Composition

Non-clay minerals. Mineralogical

TABLE 1

BRIEF DESCRIPTION OF THE FOUR LOESS SAMPLES SELECTED FOR DETAILED STUDY

Sample No.	Description	Geological Age	Engineering Classification
55-1	A very friable loess (low clay and high carbonate contents). Sampling depth 2½-3½ ft	Upper Wisconsin (?)	A-4 (8)
20-2	A typical friable loess, higher in clay than in the above sample. Total loess thickness over 100 ft. Sampling depth 39-40 ft.	Wisconsin (Undifferentiated)	A-4 (8)
26-1	A typical medium-textured loess. It is leached from the surface, but the underlying calcareous loess is not so calcareous as the friable samples listed above. Total loess thickness 30-40 ft. Sampling depth 4-5 ft.	Wisconsin (Undifferentiated)	A-6 (9)
43½ -1	A typical moderately plastic loess. The entire loess section is leached. Total loess thickness 15-20 ft. Sampling depth 5½-6½ ft	Wisconsin (Undifferentiated)	A-7-6 (18)

Increasing Clay (vertical arrow spanning left side of table)

compositions of the four loess samples are shown graphically by means of histograms in Figure 4. In these histograms the areas are proportional to relative percents by volume of the various minerals in each size fraction. The mineralogical compositions are summarized in Table 2.
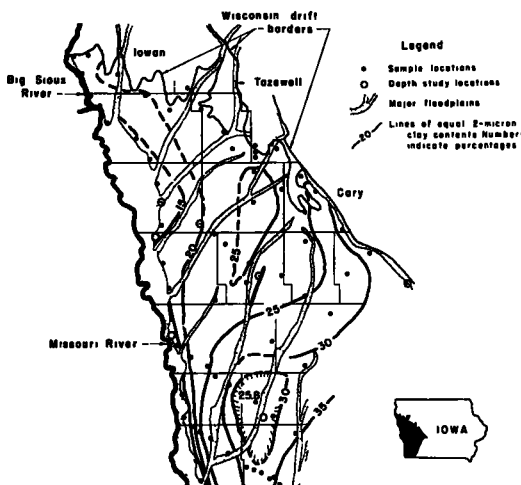

Figure 2. Tentative map showing areal distribution of clay contents in the Wisconsin loess of southwestern Iowa.

In general, as shown in Figure 5, the percentages of heavy minerals increase with decreasing grain size, probably due to sorting action during deposition of the loess. (Heavy mineral grains are more difficult to transport than light mineral grains of the same size.) The exception is in the sand fraction (74-115 microns),

which forms a minor part of each sample and contains much secondary iron.

Clay Minerals. Clay minerals occur in the minus-5-micron material and are much-more difficult to determine quantitatively. The clay minerals are thought to occur mainly as minus-2-micron material, and determinations were made on this fraction. The results and interpretations of the various tests are presented by Davidson and Handy (8), and are summarized in Table 3. Differential thermal curves are shown in Figure 6; the large initial peaks indicate montmorillonite, but the 700-deg. montmorillonite peak is very much subdued compared to the 550-deg. illite peak. This may indicate a randomly interstratified mixture of the two types of minerals.

Clay-Silt Relationships, Grain Coatings

Microscopic examination of the loess revealed that clay occurs mainly as coatings on larger grains, which tends to increase the effectiveness of the clay. The coatings are of two types: minute greenish specks adhering either individually or in clusters to the host grain (Figure 7) and as continuous, greenish, birefringent coatings partially covering the surfaces of larger grains (Figure 8). The lower photomicrograph of Figure 8 shows the birefringence of the clay coating. Since clay mineral grains are highly birefringent only when viewed on edge, they are apparently oriented flat against the host grain.
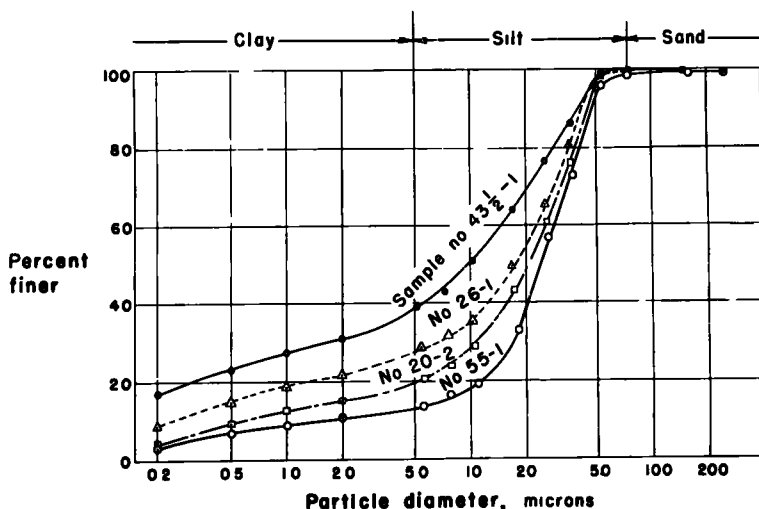
Grain coatings on the four samples are

Figure 3. Particle-size accumulation curves for the four selected loess samples.

Nontronite, a montmorillonite mineral high in iron, gives a 550-deg. endothermic reaction similar to that of illite. Although the iron content determined chemically in the clay is high, as discussed later, it is probably not high enough to suggest other than an iron-rich beidellite as the montmorillonite mineral.

Only the X-ray-diffraction data show any systematic variations in clay mineral composition. Nor could any correlation be found between these variations and the particle-size data: a further indication of an interstratified relationship. The X-ray data indicate an increase in the montmorillonite: illite ratio with increasing clay content.

summarized in Table 4. The estimated extent of coatings on the host grains is given but is, of course, only an indication of the differences between samples, since the estimates apply only to coatings visible under the microscope.

Sphericity and Roundness of Silt Grains

Sphericity is an index of grain shape; roundness is a measurement of the rounding of corners. The sphericity is probably most important for engineering applications, and as measured here it indicates the intermediate particle diameter divided by the maximum particle diameter (10). As seen in Figure 9, there is a range in

TABLE 2

MINERALOGICAL COMPOSITION OF MATERIAL LARGER
THAN 5 MICRONS IN THE FOUR LOESS SAMPLES
(Percent by Volume of the Whole Sample)

Sample No	55-1	20-2	26-1	43½-1
Total quartz	41	45	48	45
Undifferentiated qtz	37	38	40	40
Iron-oxide coated[a]	1	2	4	3
Clay coated[a]	1	1	2	1
Chert	2	4	1	2
Total feldspar	25	17	16	15
Undifferentiated feld.	19	9	10	10
Plagioclase	Trace	2	Trace	Trace
Microcline	1	Trace	Trace	0
Altered feldspar	6	5	6	5
Calcite	8	11	1	Trace
Volcanic glass	2	1	1	Trace
Muscovite	1	Trace	1	1
Biotite	Trace	Trace	Trace	Trace
Clay minerals (aggregates)	1	Trace	Trace	1
Total heavy minerals	5 6	4 9	4 0	3 1
Amphiboles	2 0	1 8	1 4	1 2
Pyroxenes	0 8	0 6	0 4	0 5
Dolomite	1 3	1 1	0 0	0 0
Iron oxides	1 1	1 0	1 1	0 9
Others	0 4	0 4	1 1	0 5
Minus 5 micron material	13 0	20.0	27 8	39 0

[a] After dispersion, elutriation, and bromoform separation

is not a variable, and its effect on engineering properties cannot be evaluated in this study.

Roundness of the grain corners was estimated, and no variations among the four samples were found. Angular or subangular grains predominate.

Chemical Data

Chemical data which may affect the performance of loess as a construction material are summarized in Table 5. The cation exchange capacity is directly related to the clay content, and carbonates contribute slightly to the exchange capacity. Calcium is the principle adsorbed cation. Calcium and magnesium were not separated in the analysis, but a spot test failed to reveal any magnesium.

The pH of the loess samples is related to the carbonate content, the calcareous samples being slightly alkaline and the leached samples being near neutral. The carbonate contents from chemical tests show close agreement with microscopic data and serve as a check. It should be

TABLE 3

RESULTS AND INTERPRETATIONS OF CLAY MINERAL TESTS ON MINUS 2 MICRON MATERIAL FROM THE LOESS

Sample No.	Amount of Minus 2 Micron Clay, %	Staining Tests	X-Ray Analyses[a]				Cat. Exch. Cap., m e./100 g		Differential Thermal Analyses	Optical Tests
			Montmor-illonite	Illite	Kaolin-ite	Quartz				
55-1	10.5	Montmor-illonite	Abundant	Almost equal to montmor-illonite	Small amount	About 10%	59 3	All indicate that mont-morillonite predom-inates	All indicate mont-morillonite and il-lite The 700°C montmorillonite reaction is largely masked, and no variations between samples are shown	All indicate mixture of minerals
20-2	15.4	"	Abundant	About ⅓ the abun-dance of montmor-illonite	Trace?	About 10%	58 6			
26-1	21 5	"	Abundant	Slightly less than in Sample 20-2	Small amount	About 10%	62 8			
43½-1	31 4	"	Dominant	Contains very little	Absent	About 10%	59.9			

[a] Analyses and interpretations by R E Grim

sphericities in any sample, and these may be averaged. Average sphericities for the four loess samples are the following.

Sample No.	Sphericity
55-1	0.76
20-2	0.76
26-1	0.76
43½-1	0.77

The sphericity was also found to be constant in the various particle size ranges above 5 microns. From this it may be concluded that grain sphericity of the loess

noted that the samples identified in the field as leached (26-1 and 43½-1) contain slight amounts of carbonates. Although calcium and magnesium carbonate were not separated chemically, the microscopic data indicate that dolomite (calcium-magnesium carbonate) constitutes some 10 percent of the carbonates present.

Soluble sulfates and chlorides are important, because of their possible deleterious effects on some stabilizing agents. They are absent or occur only as traces in the four loess samples.
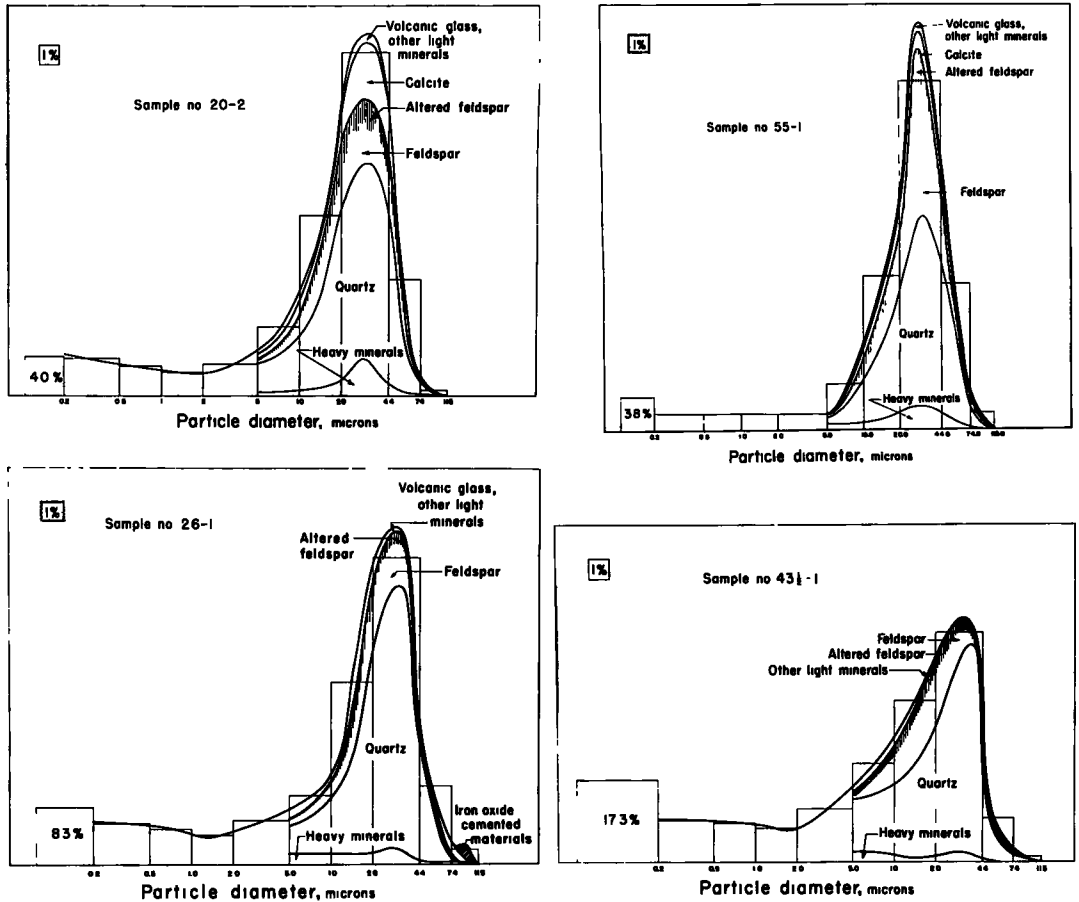
Figure 4. Mineral composition diagram for samples. Areas in the diagram represent mineral percentages by volume.

Free iron-oxide contents determined chemically correlate well with the microscopic data for iron-oxide-coated quartz, as is shown in Figure 10. The correlation between free iron and total iron is not close, and they are apparently unrelated. The free-iron content is greatest in the loess where there is a source of iron from

TABLE 4

GRAIN COATINGS IN THE FOUR LOESS SAMPLES

| Sample No.	Occurrence of clay particles	Est. % host grains are coated	Kinds of host grains	Other coatings
55-1	As individual particles and clusters of particles adhering to large grains. There are also a few continuous clay coatings.	0 to 80, averaging 17.2.	Quartz, feldspars (altered and fresh), volcanic glass. Clay coatings are not common on calcite or heavy mineral grains.	--
20-2	Same as in sample 55-1.	0 to 80, averaging 18.5.	Same as in sample 55-1 Some slightly coated calcite grains were noted	--
26-1	Same as in sample 55-1, but with a greater number of continuous clay coatings completely surrounding the host grains.	0 to 100, averaging 23.2.	Same as in sample 55-1.	Red-brown Fe oxide coatings. Host grains 0 to 100% coated, av. about 5%.
43½-1	Same as above, also dispersed as individual particles and in large aggregates which are dominantly clay.	0 to 100, averaging 38.0.	Same as in sample 55-1.	Red-brown Fe oxide coatings. Host grains av. about 5% coated.
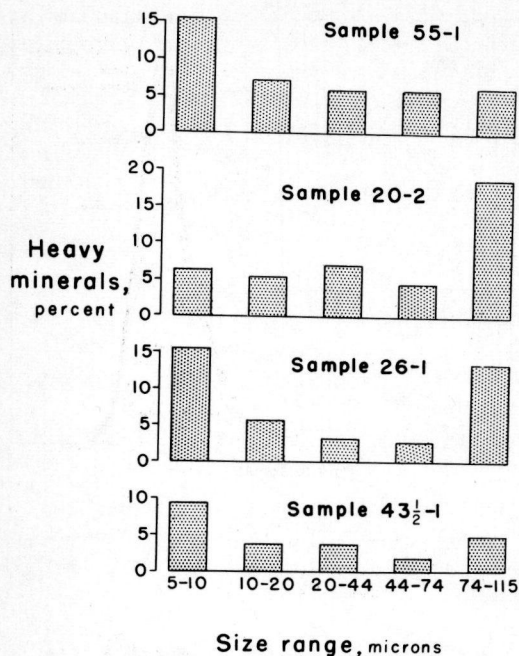
Figure 5. Percent by volume of heavy minerals in the various size fractions of each sample.

weathering in overlying soil horizons. Furthermore, secondary iron tends to become concentrated near the water table, accounting for the high free-iron content in Sample 26-1.
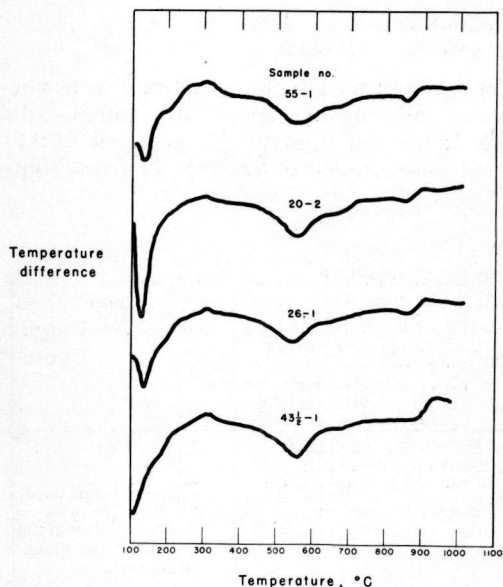

Figure 6. Differential thermal curves for minus 2 micron clay from the four loess samples.
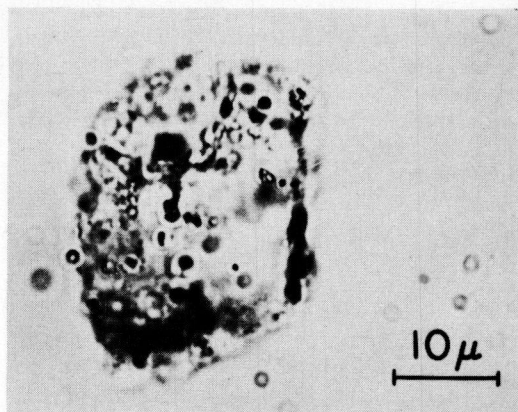

Figure 7. Photomicrograph of loess grains with particles of clay adhering.

The relationship between total iron content and heavy-mineral percentages from petrographic analysis is shown in
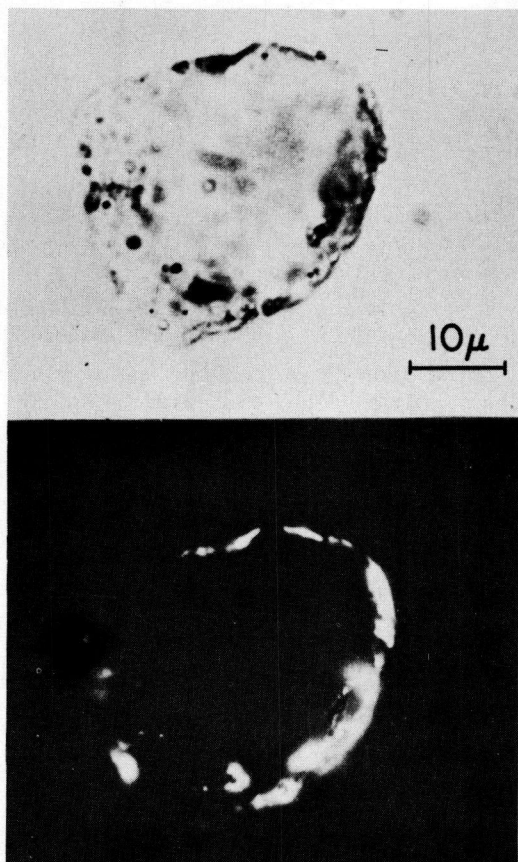

Figure 8. Continuous clay coatings on a loess grain. Above, under plane polarized light; below; under cross Nicols.

Figure 11. The correlation is almost negative, and it appears much better when the total iron is compared with the clay contents. Iron is common in the montmorillonite minerals and in the hydrous micas (illite and mixed-layer minerals).

Organic matter is present in small amounts in all samples. The content is somewhat higher in Sample 55-1, due to the presence of plant roots.
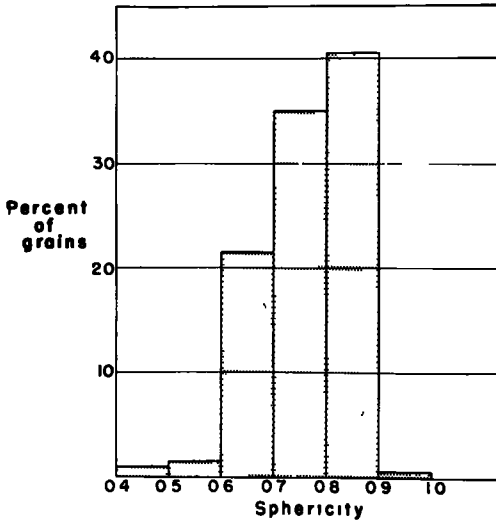

Figure 9. Histogram showing the range in sphericities of grains in a loess sample.

resulting increased cohesion and ability to hold water. Also, according to the X-ray data, as the total clay content in the loess increases, the percentage of montmorillonite in the clay also increases, amplifying the effect of the increase in clay content. For example, according to X-ray analysis Sample 55-1, with 10.5 percent of minus-2-micron clay, is about 5 percent montmorillonite; while Sample 43½-1, with 31.4 percent clay, contains about 30 percent montmorillonite.

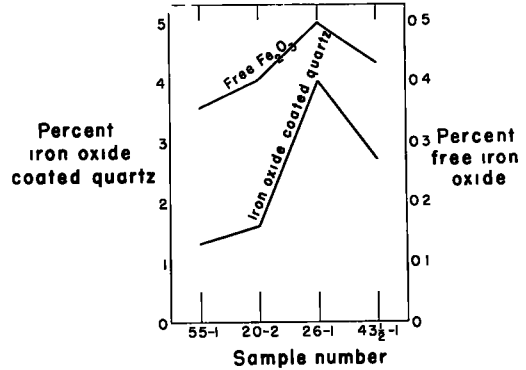The test for capillary rise gave some results which do not correlate directly

Figure 10. Comparison of data for free iron oxide determined chemically and iron oxide coated quartz grains measured microscopically.

TABLE 5

CHEMICAL DATA ON THE FOUR LOESS SAMPLES

Sample No.	Cat Exch. Cap., m.e./100g	Exch. Cations, m.e./100g			pH	CaCO₃, %	SO₃, %	Cl₃, %	Free Fe₂O₃, %	Total Fe₂O₃, %	Organic Matter, %
		Na	K	Ca							
55-1	11.2	1.4	2.0	7.8	8 4	10.3	Trace	0 01	0.356	2.70	0.24
20-2	13 4	1.5	1 6	10.3	8.7	9 4	0	0	0.403	2 42	0.17
26-1	18.2	0.9	1.2	16 1	7 0	1.9	Trace	0	0 495	3.02	0.18
43½-1	24.4	1.3	1 3	21.8	6.7	1.6	0	0	0.429	4.82	0.37

BEHAVIOR CHARACTERISTICS

A number of soil-mechanics tests were performed which give information on some of the behavior characteristics of the loess. These characteristics theoretically depend on such of the more-basic petrographic factors as the composition and particle sizes and shapes mentioned above. The tests now completed are listed in Table 6, together with the test methods and the data obtained. The sample numbers are arranged in the table from left to right to show the effects of increasing clay contents. All of the data show trends related to clay contents which are readily explained by the

with clay content. There is an increase in indicated capillary rise from Sample 55-1 to Sample 20-2, as expected with an increase in clay content and decrease in pore size. However, with successively higher clay contents the indicated rise is lower.

The capillary-rise test is essentially a measurement of the maximum head of water a column of initially loose, wet soil will support before air breaks through. Loess samples low in clay (55-1 and 20-2) apparently became puddled, or structureless, when put into the tube and soaked. Therefore, the finer the loess the smaller the pores and the higher the head which could be supported. Samples higher in

TABLE 6

BEHAVIOR CHARACTERISTICS OF THE FOUR LOESS SAMPLES

| Behavior Characteristics		Sample Number				Test Method
		55-1	20-2	26-1	43½-1	
Liquid limit, percent		29.6	30.8	39 4	51.9	AASHO Method T89-49
Plastic limit, percent		27.3	24.6	26.9	18 5	AASHO Method T90-49
Plasticity index		2.3	6.2	12.5	33 4	AASHO Method T91-49
Shrinkage limit, percent		27.2	22.3	23 3	19 1	AASHO Method T92-42
Standard Proctor density test	Max dry density, lb./cu. ft.	110.0	108.4	107.0	104.3	AASHO Method T99-49
	Optimum moisture content, %	17.8	18.0	17.7	19 1	
Modified Proctor density test	Max. dry density, lb./cu. ft.	--	120.2	116 2	113.2	Same as above except specimens are compacted in five layers by a 10-lb. hammer dropping from a height of 18 in
	Optimum moisture content, %	--	13.9	15.7	17.6	
California Bearing Ratio of soil at standard Proctor density, %	At optimum moisture	20.4	17.9	15 0	8 8	Essentially the same as the "Suggested Method of Test for California Bearing Ratio of Soils" submitted by Corps of Engineers, U S Army to ASTM (16) except that specimens prepared are at maximum density at optimum moisture content determined by standard Proctor density tests
	After 4-day soaking	15 1	8 6	4 5	1 5	
Capillary rise, in.		54	67	66	40	Essentially the same as the "Suggested Method of Test for Capillary Rise of Soil" submitted by Herman to ASTM. (16)
Engineering classification		A-4 (8)	A-4 (8)	A-6 (9)	A-7-6 (18)	AASHO Method M145-49
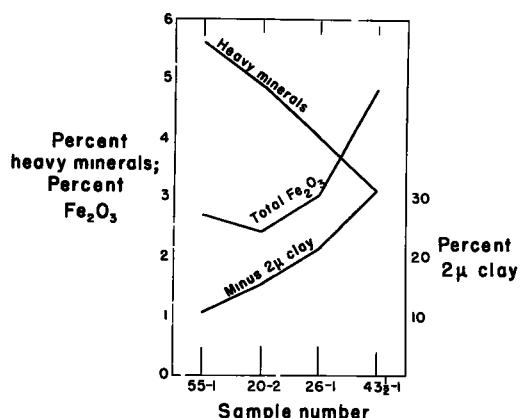
Figure 11. Relationship of total iron content to clay content in the four loess samples. The contribution of the heavy minerals to the total iron content is noticeable only in samples low in clay (55-1 and 20-2).

clay (26-1 and 43½-1) upon preliminary breaking up and sieving contained numerous granules or clusters of grains. These probably remained as granules throughout the test. The pores and air spaces between granules would be large, and less water head could be supported by such a sample. Higher clay contents would result in greater stability of the granules, due in part to lower permeability, and there would be a lower indicated capillary rise. The method of test used, therefore, succeeds at least qualitatively, probably as a result of some nonscientific good luck. It might be more reliable to measure the more basic responses, capillary potential and capillary conductivity, but these are more difficult to evaluate.

SOIL CEMENT STABILIZATION

Cement Requirements

Cement requirements for the four loess samples to meet Portland Cement Association criteria for soil cement were determined by Ally (17), using standard methods (ASTM D558-44, D559-44, and D560-44). The results are shown in Table 7, and the relationship between cement requirements and clay contents is shown in Figure 12.

TABLE 7

CEMENT REQUIREMENTS FOR THE FOUR LOESS SAMPLES TO MEET P C A. CRITERIA FOR SOIL CEMENT

| Sample No	BPR Soil group	P C A criteria			Cement requirements to meet criteria, % by volume					
		Maximum soil cement loess, %	Maximum volume change, %	Maximum moisture absorption, %	Soil-cement loss		Volume change		Moisture absorption	
					Wet-dry	Freeze-thaw	Wet-dry	Freeze-thaw	Wet-dry	Freeze-thaw
55-1	A-4	10	2	Not more than that necessary to fill voids at molding	<8 0	9.8[a]	8. 5	8.4	<8.0	<8.0
20-2	A-4	10	2	"	8.0	13.5	10.5	12.1	10 0	11.0
26-1	A-6	7	2	"	<14.0	14.6	14 2	15.6	<14.0	<14.0
43½-1	A-7-6	7	2	"	14 3	16.0	16.1	17.0	15 0	15.2

[a] Minimum cement contents which satisfy all criteria are underlined
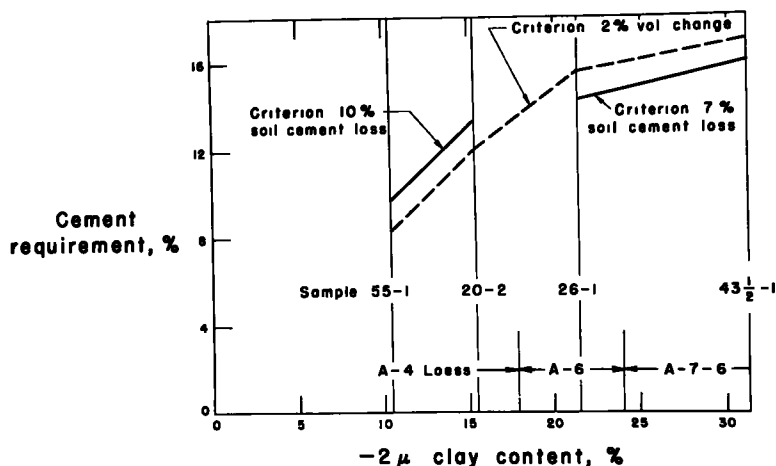
Figure 12. Relationship between cement requirement and clay content of southwestern Iowa loess. Cement requirements meet P.C.A. criteria for soil cement.
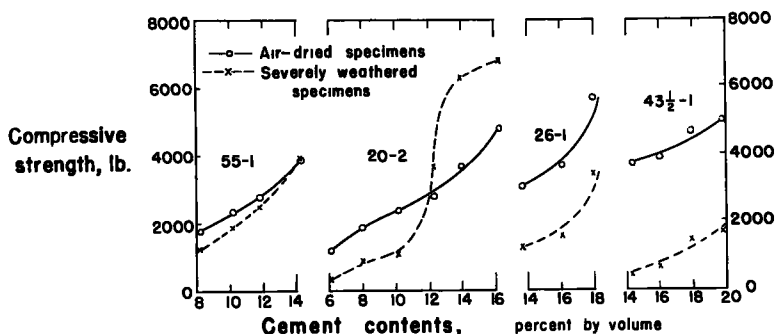

Figure 13. Compressive strengths of air-dry and severely weathered 2 inch by 2 inch soil cement cylinders containing varying amounts of cement. Air-dried specimens were moist-cured 7 days, air-dried 38 days, dried 1 day at 160 F., and tested. Severely weathered specimens were tested immediately after removal from the water bath.

Weathering of Soil Cement

Method. Compressive strengths of 2-by-2-inch-diameter cylinders of soil cement were measured by Ally (17) after severe weathering by a procedure developed by the Civil Aeronautics Administration (18). The specimens were first moist-cured for 7 days, then subjected to the following treatments: (1) three days of capillary absorption in a moist cabinet; (2) four days of total immersion in water; (3) twelve cycles of alternate freezing at -10F. for 8 hours and thawing by total immersion in water at room temperature for 16 hours; (4) twelve cycles of alternate wetting by total immersion in water at room temperature for 8 hours and oven-drying at 160 F. for 16 hours; and (5) seven days of total immersion. The specimens were tested while wet.

Results. Figure 13 shows compressive strengths of severely weathered 2-by-2-inch cylinders compared with strengths of un-weathered air-dried cylinders. Compressive strengths of both air-dried and severely weathered specimens increased with increasing cement contents. Not all specimens, however, lost strength as a result of severe weathering, and some showed a large gain. Since curves for the weathered specimens are on the basis of wet strength, and the other curves are on the basis of oven-dry strength, the gain is even greater than is apparent from the graph.

To determine if the weathering-strength gain is due to a curing effect in the weathering treatment, specimens were air-dried for periods of up to 45 days. Cement contents used were those which meet PCA minimum requirements. The strength gains with age are shown in Figure 14. Gains are greatest for Sample 20-2. This agrees with data from Figure 13 which indicate that only with Sample 20-2 was the cement content high enough to give a considerable strength gain after severe weathering. The conclusion is that the effect of severe weathering may be completely cancelled by a simultaneous cure.

The strength gain is odd in that it is most pronounced in the sample second lowest in clay (20-2). The same behavior was shown by the sample lowest in clay (55-1), but a higher cement content was necessary. Petrographic studies showed nothing out of the ordinary in any of the samples—only the fairly continuous com-
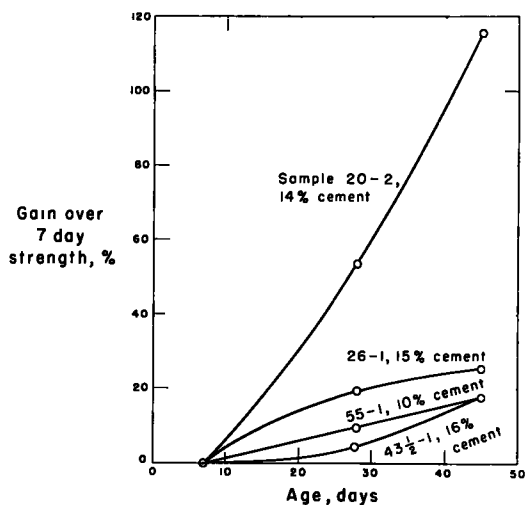

Figure 14. Gain in compressive strength of 2 inch by 2 inch soil cement cylinders by aging. The indicated age equals 7 days of moist curing plus days of air drying. Specimens were tested after drying 1 day at 160 F.

position change between samples according to clay content. The possible importance of the weathering strength gain justifies further study to find reasons for the gain. Knowing these, it should be possible to determine if and how the gain may be encouraged with lower cement contents or with less-susceptible loesses.

In the present paper the reason for the gain can not yet be stated with certainty because in the loess there are several variables which may be responsible. These variables will be pointed out and discussed, and have been used as a guide in the planning of testing programs now under way. Results of these tests should show if the possibilities pointed out in this paper are correct.

DISCUSSION

Mineral Surface Areas

In applying petrographic data to stabilization problems, it was reasoned that fine material in a soil has a greater effect on chemical stabilization, including soil cement, than a like amount of coarse material with the same composition, since with most methods of stabilization the grain surfaces are to be coated. Therefore, in this study mineral content was expressed on the basis of surface areas in addition
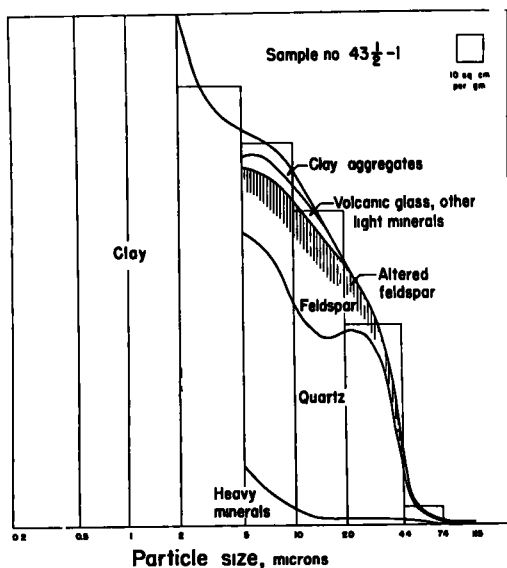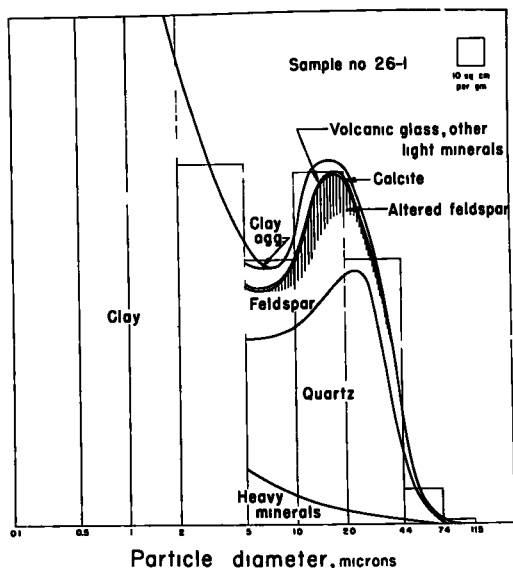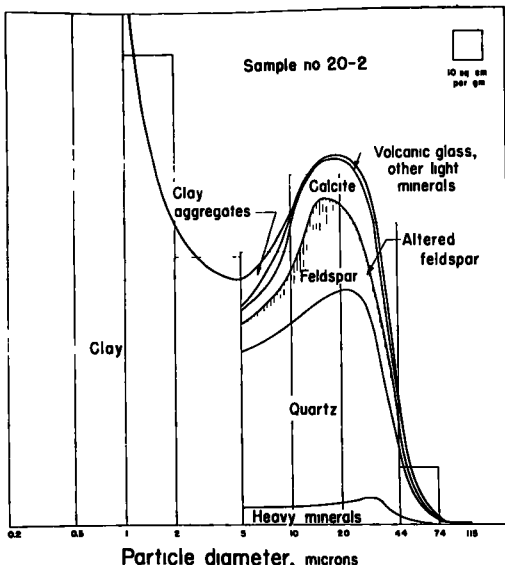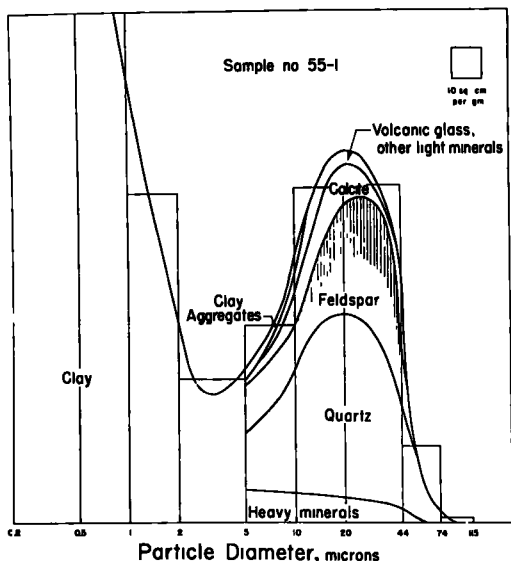
Figure 15. Mineral surface area diagram for samples. Areas in the diagram represent grain surface areas contributed by each mineral.

to the usual weight or volume percentages. Figure 15 shows histograms modified to show surface areas of grains of various minerals in each size fraction. By comparison with the standard histograms showing volume percentages (Figure 4). the new emphasis on the finer fractions may be noted. Since the composition of each sample varies in various size fractions, it is necessary to calculate and add together surface areas of the various minerals in each size fraction, thus obtain-

ing the total area of each mineral in a sample. The areas may then be reduced to percentages, as given in Table 8. A discussion of the surface area calculations and errors involved is given in Appendix C.

Application to Southwestern Iowa loess

It may be noted in Table 8 that the contribution of quartz to the surface area increases with increasing clay content.

Figure 16 shows the silt surface areas in the four loess samples, and an increase with increasing clay content may be noted. This is due to decreasing particle size of the silt.

TABLE 8

MINERAL SURFACE AREAS IN THE PLUS 5 MICRON FRACTION OF EACH LOESS SAMPLE

(Values given are percentages of the total sand-silt surface area)

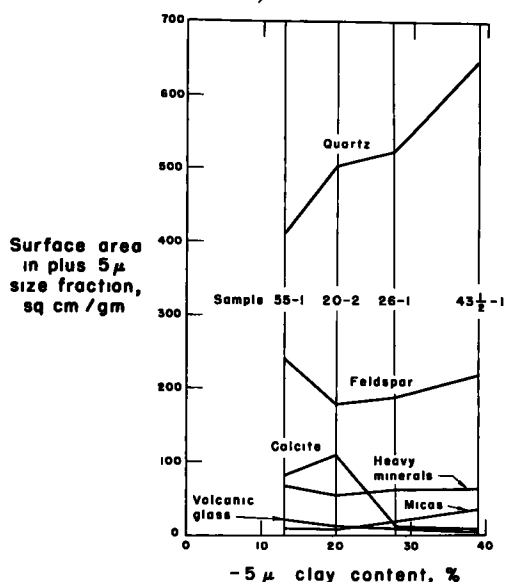| Sample No	55-1	20-2	26-1	43½-1
Quartz	49 3	58 3	64.0	66 0
Feldspar	29 0	20.5	23 0	24 0
Calcite	9 7	12.8	1.2	0 8
Volcanic glass	2 5	1 4	1 1	0.4
Heavy minerals	8 3	6.3	7 6	6 3
Other minerals	1 2	0.8	2 8	2 5

content is to reduce the effective nonclay surface areas available for cementing, and it is highly possible that the effective quartz surface area approaches an optimum in Sample 20-2. This is illustrated in Figure 17, where a cube function of clay content has been subtracted from the quartz surface areas. Such a relationship has the scientific status of a guess approximated, but it may point out the correct trend. Calcite is another major variable which will be investigated, perhaps by leaching some Sample 20-2 loess and testing for a strength gain on curing. Testing to determine accurate reference curing curves is now under way.

Figure 16. Mineral surface areas in the plus-5-micron-size fraction as related to the minus-5-micron clay content.
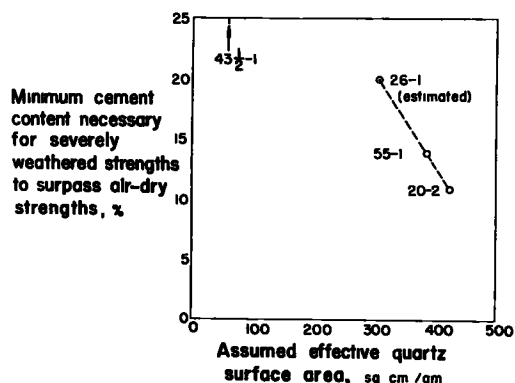

Figure 17. Cement contents at which severely weathered strengths equal air-dry strengths as related to an effective surface area calculated for quartz.

Sample 20-2, which behaved so uniquely in the CAA weathering test, displays some major differences from the other samples: It has the highest calcite (CaCO₃) and lowest feldspar areas, and it has a rather high quartz area in relation to its clay content.

While it is possible that the variations in weathering strength gain for different loess samples are related to reactions which affect the setting of the cement paste, the strength of the cement bonds to the loess grains appears to be the major factor, especially since loess grains are mostly clay coated. One effect of high clay

STABILIZATION OF SOIL CEMENT WITH TREATMENTS OR ADMIXTURES

Monionic Treatments

Loess Sample 20-2 was treated by Nicholls (19) to obtain a monoionic soil material. The treatments used are those of Winterkorn et. al. (20): the loess is first treated with HCl and washed to obtain a hydrogen clay, and the various metallic hydroxides added either in solution or colloidal suspension and time allowed for the reaction to take place. The effects of these treatments on portland-cement stabilization of Sample 20-2 are summarized in Table 9. The most notable effect is that of iron. This may be due to replacement of the exchangeable cations by iron, or quite possibly by an excess of iron hydroxide, either coating the loess grains and making a poor bond, or coating the cement

clinker grains and retarding the set (<u>21</u>, p. 479).

As pointed out by Grim (<u>22</u>, p. 143), the treatment to obtain a hydrogen clay may result in partial decomposition of the clay mineral and movement of aluminum ions to exchange positions. In such a case the aluminum would remain at the exchange position through the subsequent hydroxide treatment, and a monoionic clay would not be obtained.

Admixtures

A number of additives to soil cement were investigated by Nicholls (<u>19</u>). The effects of these additives on portland-cement mixtures with Samples 20-2, 26-1, and 43½-1 are summarized in Table 10. Major changes were noted only with the addition of calcium chloride, which increased the rate of hardening, and with sodium methyl siliconate (a silicone), powdered Vinsol resin, and an asphalt emulsion sold commercially as a waterproofing agent for concrete. The silicone, the resin, and the asphalt emulsion all resulted in higher strengths of immersed specimens, probably due to waterproofing. The waterproofing effect may be considerably aided by the adsorptive power of clay mineral coatings for organic complexes.

The effects of calcium chloride are illustrated by data from Ally (<u>17</u>) on Samples 20-2 and 43½-1. As shown in Figure 18, with Sample 43½-1 the addition of 2 percent of calcium chloride increased 7-day and 45-day strengths about proportionally, indicating an overall strength gain instead of an acceleration effect. The same proportional increase is also shown in specimens subjected to the CAA weathering test. Whether the strength gain is due to an improvement in the character of the clay minerals, of the cement gel, or of the bonding action, is not yet known.

TABLE 9

EFFECTS OF SOME MONOIONIC TREATMENTS ON PORT-
LAND CEMENT STABILIZED LOESS
(Based on data from exploratory tests (19))

Cation Used in Treating Natural Soil	Effect of Treatment on Properties of Soil Cement Mixtures
Aluminum	Effect not obvious
Iron	Large decrease in strength
Potassium	Slight decrease in strength
Sodium	Slight decrease in strength

The addition of calcium chloride to Sample 20-2 with portland cement results in somewhat different trends than those mentioned above. The addition of 4 percent calcium chloride increased 7-day strengths but there was no further gain in
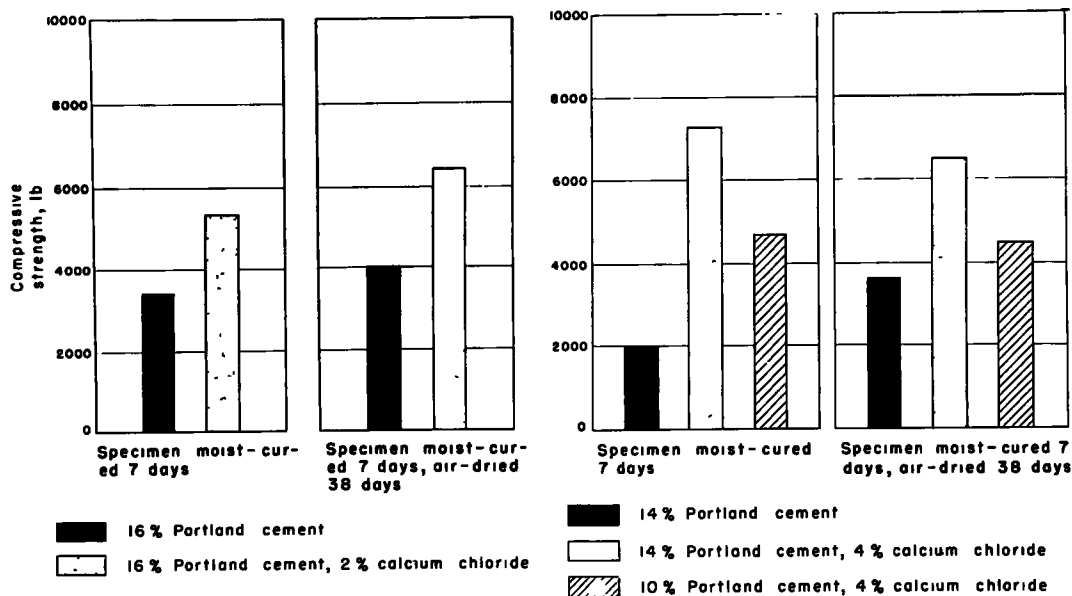

Figure 18. Effect of calcium chloride on the compressive strength of soil-cement with Sample 43½-1 (left) and Sample 20-2 (right). Specimens were dried 1 day at 160 deg. F. before testing.

TABLE 10

EFFECTS OF VARIOUS ADMIXTURES ON PORTLAND CE-
MENT STABILIZED LOESS

(Based on data from exploratory tests (19)).

Admixture[a] Added to Soil-Cement Mixture	Range of Admixture Tested, Percent[b]	Effect of Admixture on Soil-Cement Mixture
Calcium chloride	0 1-6 0	Increase in rate of hardening
Aluminum chloride	0 2-1 0	Effect not obvious
Ferric chloride	0 2-1 0	Effect not obvious
Lime, hydrated	0 1-2 0	Effect not obvious
Fly ash	0. 2-7 0	Effect not obvious
A calcium salt of vinyl acetate-maleic acid polymer	0. 02-0 05	Effect not obvious
Silicone A	0 05-0 25	Effect not obvious
Silicone B	0 1-0 5	Effect not obvious
Sodium methyl siliconate	0. 5-2 5	Increase in immersed strength
Ethyl sodium silanolate	0 05-0 5	Effect not obvious
A fatty amine acetate	0. 2-3 0	Effect not obvious
Vinsol resin, pulverized	02-1 00	Increase in immersed strength
Paraffin	1	Effect not obvious
A commercial waterproofing agent	1-4	Increase in immersed strength
Commercial admixture A	0. 02-0 30	Effect not obvious
Commercial admixture B	0 01-0 15	Effect not obvious
Commercial admixture C	0 04-0 15	Effect not obvious
Commercial admixture D	0 04-0 60	Effect not obvious
Bentonite	0 2-2 0	Effect not obvious

[a] Source and commercial name of admixtures are given in Appendix D

[b] Percentage of admixtures tested are expressed as percent of oven-dried weight of soil

strength with 38 days of additional curing. This would indicate that the major effect with Sample 20-2 is one of acceleration. Specimens without calcium chloride and subjected to the CAA weathering test showed a high strength gain, as has been discussed. With calcium chloride added, the specimens gave much lower strengths after severe weathering, indicating that the acceleration in cure may have been at the expense of the high strengths eventually obtained with cement alone. The effects of additions of smaller amounts of calcium chloride are now being studied in this relation.

SUMMARY

Petrographic information on a soil material is essential for an understanding of stabilization involving cementing reactions, and methods have been presented by which petrography may be applied to stabilization problems. Possible explanations for various questions involving stabilization of loess with portland cement alone and with admixtures have been suggested. The validity of these interpretations will be determined by experimental work now in progress. When reactions between cement and the ingredients of soil are thus more fully understood, further work to find improvements may be carried on more efficiently. In other words, the approach might be labeled Big Think and Little Do. This can grow into Big Do. It is preferable to the ever-popular Big Do and Little Think, which is equivalent to the proverbial shot in the dark and may result in a large expenditure of ammunition for benefits obtained.

ACKNOWLEDGEMENT

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Appendix A

A RISING-CURRENT ELUTRIATOR FOR FRACTIONATING SILTS

Separation of soils and sediments into size fractions prior to analysis is an important petrographic technique resulting in more-accurate and more-complete mineral-composition data. Coarse materials are usually separated by sieving, and materials too fine to sieve are separated by sedimentation procedures.

A rising-current elutriator is a separation device consisting of a vertical tube in which the sample is placed. Through this tube a fluid, usually water or air, flows upward at a measured velocity.

Particles in the sample which are large enough and heavy enough to settle faster than the velocity of the current remain in the elutriator; smaller particles are washed or blown out at the top. The particle size at which the separation occurs may be varied by regulating the velocity and viscosity of the suspending medium.

A multiple-tube water elutriator (Figure A) was used to fractionate silts in the loess. Successive tubes have double diameters, decreasing the water velocity in each case by a factor of one fourth. Ac-

cording to Stokes' Law, the settling velocity of a sphere is proportional to the square of its radius: $V = Kr^2$. Since at the critical particle size in each tube the up-current equals the settling velocity, decreasing the settling velocity by one fourth means

$$V' = \frac{V}{4} \dashv Kr'^2 = \frac{Kr^2}{4}$$

$$r' = \frac{r}{2}.$$

That is, doubling the tube diameter results in halving the particle size at which separation will occur. In the present study three tubes were used, and the water velocity was regulated so that material

Dispersion of Sample

Because of flocculation, water elutriation has previously been reported as unsuccessful for separating materials finer than 10 microns, and various stirring devices have been built into the elutriation tubes (23). In the present study the loess was first dispersed as for a mechanical analysis, using a high-speed stirrer and sodium metaphosphate. Because the first tube in the elutriator is small in diameter and would have to be disproportionately long to accommodate the dispersed sample, a sample loading chamber (Figure A) was added to the apparatus.

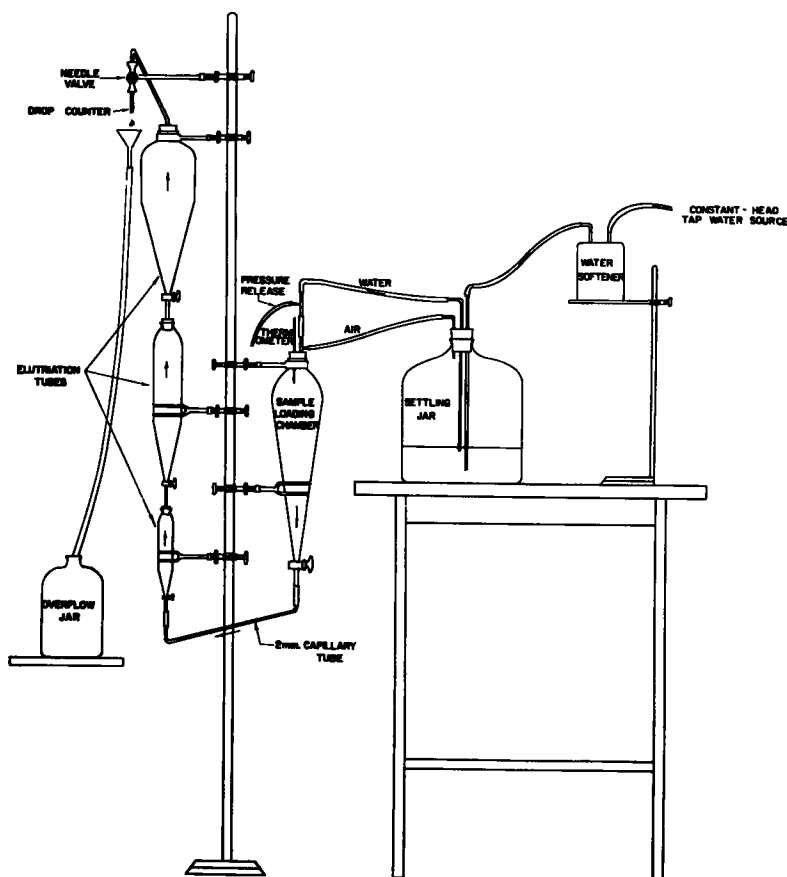The use of tap water in elutriation caused

Figure A.   Rising-current elutriator for the size separation of fine materials.

coarser than 20 microns diameter was retained in the first tube. This automatically resulted in material 10 to 20 microns being retained in the second tube, material 5 to 10 microns being retained in the third tube, and minus-5-micron clay being carried out in the overflow.

flocculation to occur in the third chamber and presented complete separation of the minus-5-micron clay. Distilled water also caused flocculation, probably because of dilution of the dispersing agent. The use of tap water with a small zeolite water softener was successful, the sodium in

the softened water being sufficient to maintain dispersion. A settling jar was then necessary to prevent solid impurities from the water softener from entering the sample.

Apparatus

The three elutriation tubes are made from glass stock $\frac{15}{16}$ inches, $1\frac{7}{8}$ inches, and $3\frac{3}{4}$ inches inside diameter, and were patterned somewhat after those of Kopecky and Anderson. The length of the taper on the tubes is about twice the inside diameter to prevent turbulence and to promote an even cross-sectional velocity. The tubes are arranged in a vertical column to prevent plugging of the connecting tubing. The sample charger is a 1000-ml. separatory funnel. It is connected to the first chamber by an inclined tube of capillary size to hold the velocity high and discourage plugging.

A 5-foot or 6-foot constant pressure head measured above the overflow is sufficient for elutriation. Flow is regulated with a needle valve located in the overflow line to maintain pressure throughout the system and to hold air in the water in solution. Otherwise air bubbles may restrict flow and cause a stoppage.

Procedure

1. About 20 grams of soil free from material larger than 1 mm. diameter and preferably all passing the no. 325 sieve is dispersed as for a mechanical analysis, using a proportional amount of dispersing agent. If necessary, the material may be first sieved to prevent clogging the elutriator.

2. The water softener is recharged if necessary, the elutriator filled with water, and the system checked for leaks or pressure failure.

3. The input head is blocked with a hose clamp and the sample flushed into the loading chamber. Pressure is again applied and hose clamps are so arranged that air from the settling bottle is forced into the sample-loading chamber. When the chamber is almost empty, the hose clamps are changed to force water from the settling bottle into the loading chamber. The loading chamber now serves only as a passage for water into the elutriator.

4. The temperature of the inflowing water is noted on the thermometer mounted in the sample-loading chamber. The rate of flow, which is regulated by means of the needle valve, may be calibrated in number of drops in a given time.

5. Elutriation is continued until the overflow runs clear. The three stopcocks on the three elutriation tubes are then turned off simultaneously. The pressure is turned off, and the tubes are disassembled to remove the samples.

6. A complete elutriation requires about 16 hours and about 3 gallons of overflow. If at any time a pressure failure occurs, the separated materials in the tubes will settle into the lower tubes, and a longer time will be required. The process may be interrupted at any time by turning the three tube stopcocks, but it is usually more satisfactory to continue the process uninterrupted to avoid difficulties in restarting. The elutriator tends to slow down rather than to speed up after adjustment, due to air coming out of solution at the needle valve. The velocity must therefore be checked occasionally.

Calculations

The velocity of flow may be calculated as in the following example:
From Stokes' Law,

$$V = \frac{2(d_1-d_2)\, gr^2}{9\,\eta} = \frac{L}{T},$$

where V is the settling velocity of the particles in cm. per sec., g is the acceleration of gravity, r is the particle radius in cm., η is the viscosity of the settling medium, d_1 and d_2 are the specific gravities of the particles and the medium, respectively, and L is the settling distance in time T. In elutriation the time is measured with a stopwatch for a given volume of overflow. If the overflow is 10 ml., the settling distance in the large tube with radius $r_e = 1\frac{7}{8}$ inches is

$$L = \frac{10}{r_e^2} = \frac{10}{71.1} = 0.1408 \text{ cm.}$$

The time T for a 10 ml. overflow is then

$$T = \frac{9\,\eta\,(0.1408)}{2(d_1-d_2)\,980r^2}.$$

If the separation in the large tube is to be made at 5 microns, the particle radius $r = 0.0025$ mm. At 68 F., $\eta = 0.01009$ poise.

If $d_1 = 2.65$, $T = \dfrac{9(0.01009)(0.1408)}{2(2.65-1.00)(980)(0.0025)}$

$= 63.1$ seconds for 10 ml. overflow.

For convenience in timing, the rate of overflow may be converted to time necessary for 5 drops, and suitable graphs relating overflow to temperature prepared.

Appendix B

CENTRIFUGAL SEPARATION OF HEAVY FROM LIGHT MINERALS

The separation of minerals into light and heavy fractions by immersing grains in bromoform (sp. gr. 2.87) is a useful technique in mineral identification. Bromoform separation of silts presents special problems, such as the necessity for speeding separations by centrifuging.

Apparatus

The inner and outer centrifuge tubes (Figure B) are described by Jeffries and Jackson (24). The outer tube is a 40-ml. double-thickness Pyrex centrifuge tube to withstand breakage.
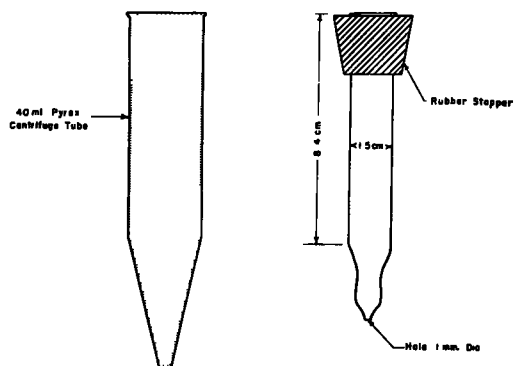

Figure B. Centrifuge tube apparatus for the bromoform separation of silts into heavy and light minerals.

Procedure

1. A thoroughly oven-dried (110 C.) size fraction obtained by sieving or elutriation is quartered to obtain a representative sample. The fine fractions require further prolonged drying at 150 to 200 C. to prevent flocculation in the bromoform.

2. The outer centrifuge tube is filled about half full with bromoform, and the inner tube is inserted. About 1 gram of sample is placed in the inner tube and stirred. The assembled apparatus is stoppered and centrifuged at speeds not to exceed 1500 rpm. to lessen breakage. After sufficient centrifuging heavy minerals will have settled through the hole in the inner tube into the outer tube. The sample in the inner tube is stirred and centrifuged three times, or until no more heavy particles settle out.

3. The mineral separates are rinsed into filtering crucibles with bromoform, and the bromoform is filtered off with the aid of a partial vacuum. The bromoform is saved for future separation.

4. Grains in the filtering crucibles are rinsed with acetone, oven-dried at 110 C., brushed out and weighed on an analytical balance. Heavy mineral percentages may then be calculated. As the separation is seldom perfect, the percentages may be corrected after microscopic examination of the light fraction.

Appendix C

DERIVATION OF AN EXPRESSION FOR THE SURFACE AREA OF GRAINS IN A SIZE FRACTION

Surface areas of various minerals in a soil material may be calculated to aid in interpretation of stabilization data, since this emphasizes the finer, more active material.

The basic assumption used in the derivation of Stokes' Law is also applied here with the particles assumed to be spherical. Abrading is not common in silt-size mate-

rial, so sphericities do not vary greatly between size fractions or between samples, and the error is more or less constant.

The mass of a spherical particle is

$$M = \frac{\pi D^3 \rho}{6}$$

where D is its diameter in cm. and ρ its density in grams per cubic cm. In one

gram there would be $\dfrac{6}{\pi\,D^3 \rho}$ particles, each with a surface area of πD^2. The surface area in one gram of particles is therefore

$$S = \frac{6}{\pi\,D^3\,\rho} \times \pi D^2$$

$$= \frac{6}{D\rho}$$

If d is in microns, S will be in square meters per gram.

In standard histograms the distribution of particles may be considered to be uniform throughout the size range of each bar. This is illustrated in Figure C; the narrower the bars of the histogram, the more accurate the assumption. The relationship between specific surface and particle diameter is shown in Figure D. If D_1 and D_2 are the limits of the size fraction, S_m is the mean specific surface of the fraction. In Figure D,

$$S_m\,(D_2 - D_1) = \int_{D_1}^{D_2} S\,dD$$

$$= \frac{6}{P} \int_{D_1}^{D_2} \frac{1}{D}\,dD$$

$$= \frac{6}{P}\,\ln\frac{D_2}{D_1}\,.$$

$$S_m = \frac{6\,\ln D_2/D_1}{P\,(D_2 - D_1)}\,.$$

Example: With a 10 - to - 20 -micron fraction of loess with a density of 2. 70 gm. per cu. cm.,

$$S_m = \frac{6\,\ln\left(\dfrac{20}{10}\right)}{2.70\,(20-10)}$$

$$= 0.154\ \text{sq. m./gm.}$$

$$= 1540\ \text{sq. cm./gm.}$$

The surface areas thus calculated for the various size fractions separated in the loess are given in Table A.
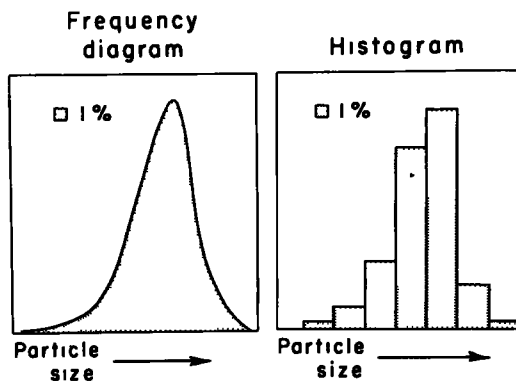
Figure C. Assumption of a uniform distribution of grain sizes in a size fraction. At left is the true representation of a soil; the graph at the right shows the assumption made.

TABLE A

SURFACE AREAS OF GRAINS AS RELATED TO PARTICLE SIZE

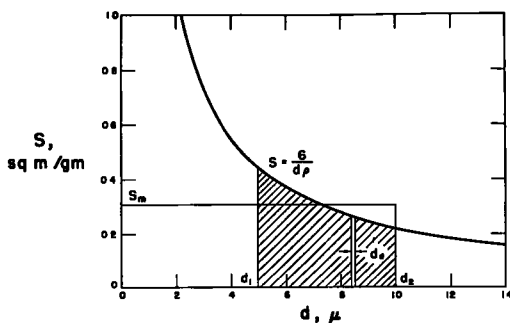| Particle Diameter, microns	Surface Area in 1 gm of Material, sq cm
0 2-0 5	67, 900
0. 5-1 0	30, 800
1. 0-2.0	15, 400
2 0-5 0	6, 790
5 0-10	3, 080
10-20	1, 540
20-44	714
44-74	384
74-115	237

Figure D. Relationship between specific surface and particle diameter, showing integration to obtain the mean surface area of a size fraction.

Appendix D

SOURCE OF ADMIXTURES USED IN PORTLAND CEMENT STABILIZATION OF LOESS

Material	Source
Portland cement, Type I	Hawkeye Portland Cement Company, Des Moines, Iowa
Lime, hydrated	Linwood Stone Products Company, Buffalo, Iowa
Flyash	Louisville Gas and Electric Company, Louisville, Kentucky
A calcium salt of vinyl acetate-maleic acid polymer, known as "Krilium No. 6 (CRD-186)"	Monsanto Chemical Company, St. Louis, Missouri
Silicone A, known as Linde silicone SF99	Linde Air Products Company, Tonawanda Laboratory, Tonawanda, N.Y.
Silicone B, known as Linde silicone C25	Same as above
Sodium methyl siliconate	General Electric Company, Chemical Division, Waterford, N.Y.
A fatty amine acetate, known as "Armac T"	Armour and Company, Chicago, Illinois
Vinsol resin, pulverized	Hercules Powder Company, Wilmington, Del.
A commercial waterproofing agent known as "Hydropel"	American Bitumuls Company San Francisco, California
Commercial admixture A, known as "Pozzolith No. 2"	Master Builders Company Cleveland, Ohio
Commercial admixture B, known as "Pozzolith No. 8"	Same as above
Commercial admixture C, known as "Stearolith"	Same as above
Commercial admixture D, known as "Omicron Mortarproofing"	Same as above