

# DISPERSION OF LOESS FOR MECHANICAL ANALYSIS

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## SYNOPSIS

THIS PAPER reports results of a special study made in connection with the investigation of the Wisconsin (Peorian) loess of Southwestern Iowa, which is being carried on jointly by the Engineering Experiment Station of Iowa State College and the Iowa State Highway Commission, to develop a tailored procedure for dispersing Wisconsin loess for mechanical analysis. The principle of dispersion is reviewed to clarify the function of each step in existing procedures and also to serve as a background and guide for the development of a dispersion procedure for Wisconsin loess.

In addition, experiments were performed to: (1) evaluate several deflocculating agents, *viz.*, sodium metaphosphate, sodium pyrophosphate, sodium oxalate, and sodium silicate; (2) determine the need for hydrogen-peroxide and hydrochloric-acid treatments; and (3) evaluate a rubber stirring paddle and determine stirring time requirements, using the conventional high-speed malted-milk-type mixing apparatus.

Among the deflocculating agents compared experimentally, sodium metaphosphate is the most efficient in dispersing Wisconsin loess. The amount of this chemical adopted for dispersing Wisconsin loess samples for mechanical analysis determinations is 60 ml. of 0.5 N solution.

Wisconsin loess samples having plasticity indices greater than 20 percent do not need to be treated with hydrogen peroxide, as specified in ASTM and AASHTO methods, providing the organic matter content is low. If sodium metaphosphate is used as the deflocculating agent, the hydrochloric acid treatment is not advantageous or desirable for Wisconsin loess samples having high carbonate contents.

To avoid frequent replacement of the standard metal stirring paddle due to wear, the rubber paddle used by the soils laboratory of the Iowa State Highway Commission can be used without loss of stirring efficiency (there may even be a slight increase in the stirring efficiency). A stirring period of 4 min. with the high-speed stirring apparatus is considered adequate for Wisconsin loess samples.

● LOESS is a fine-grained soil material whose origin, whether aqueous or aeolian, has been a subject of debate among geologists for many years. As a result of this controversy, no single definition of loess has received general acceptance. Flint (1) give a comparatively broad definition as follows:

Loess, then, is a buff-colored, nonindurated sedimentary deposit consisting predominantly of particles of silt size. Commonly it is nonstratified, homogeneous, calcareous, and porous, and it may possess a weak vertical structure resembling jointing.

In recent years, most geologists have been won over to the aeolian concept, which contends that the bulk of the loess was transported and deposited by wind action. Fine

sediments exposed without vegetative cover to the winds in desert basins, river flood plains, glacial drift and outwash surfaces are considered to be the major sources of loess.

Surface deposits of loess cover extensive areas in the United States (2) as well as in other parts of the world (3). Thicknesses of the deposits vary from a few feet to over 1,000 ft., and the loess is not usually intermixed with soil materials of different origin. Approximately two thirds of the State of Iowa has a surface covering of Wisconsin loess of variable thickness. This loess, also called Peorian loess in the geological literature, blankets older loesses and pre-Wisconsin Pleistocene deposits and is named after the Wisconsin glaciations from which the loess presumably originated. It has been estimated that, if all the Wisconsin loess in Iowa were

evenly distributed over the state, it would average about 10 ft. in depth. The deepest sections of Wisconsin loess are found in counties bounding the western side of the state, along the east valley wall (first bluff line) of the Missouri River. Here, where the wide flood plain and persistent sand bars of the great river have maintained an ever-replenished supply of dust and silt, the loess has accumulated to thicknesses of from 60 to over 100 ft.

Because of the abundance of loess in Iowa and its importance as a soil material on which and with which roads and other engineering structures are built, an investigation of the deep Wisconsin loess in southwestern Iowa has been included in a project established at the Engineering Experiment Station of Iowa State College. The project (No. 283-S) is entitled *An Investigation of the Loess and Glacial Till Materials of Iowa*; it is sponsored by the Iowa Highway Research Board and is financed by funds appropriated by the Iowa State Highway Commission and the U. S. Bureau of Public Roads. This paper reports the results of a special study made in connection with the southwestern Iowa loess investigation, which had as its purpose the development of a tailored procedure for dispersing Wisconsin loess for mechanical analysis.

The AASHO (T88-49) and ASTM (D422-39) standard methods of mechanical analysis (4, 5) recommend definite procedures for dispersing soil. These procedures were developed for use with a wide variety of soils, however; and after an extensive review of the literature, it seemed probable that they would not give optimum dispersion for loess soils, especially those containing large amounts (up to 18 percent  $\text{CaCO}_3$  by weight) of carbonates.<sup>1</sup> Since many investigators in the fields of agriculture, ceramics, geology, and engineering have studied the problems of soil dispersion, a review of present knowledge of the subject is given herewith before further discussion of the Wisconsin loess study.

#### PRINCIPLE OF SOIL DISPERSION

An accurate and reproducible mechanical analysis is dependent upon the complete and stable dispersion of a soil sample. In other

words, all soil aggregates (secondary particles) must be separated, as nearly as possible, into primary particles, and these particles must then be kept in a dispersed state throughout the mechanical analysis. It is necessary to understand, therefore, not only the factors causing aggregation in soils but also those which may cause the flocculation of primary particles once they have been separated.

#### *Causes of Aggregation*

Soil aggregates are formed primarily as a result of the presence in soil of the colloidal cementing agents, such as clay, organic matter, and oxides of iron and alumina. The cementation effect of colloidal clay is believed to be the result of dehydration of the clay particles. When the moisture content of a soil is fairly high, the water films surrounding clay particles are so thick that the cohesive forces between adjacent particles are not significant. As the moisture content decreases, the water films become thinner and thinner, and the cohesive forces increase accordingly. In the air-dry condition such water films have tremendous bonding strength; rehydration, however, tends to destroy the bond.

Colloidal organic matter has long been recognized as a cementing agent in soils, but there apparently is no exact knowledge of the mechanism of the cementation effect. In discussing the explanations that have been offered by various investigators, Baver (6) concludes that the majority of the evidence points to some type of oriented adsorption of organic molecules on the surfaces of clay particles; such adsorption appears to be stabilized by subsequent dehydration of the adsorbed colloidal organic matter. Since rehydration is extremely slow, this process causes a tenacious cementing influence which is responsible for aggregate formation.

Dehydrated oxides of iron and alumina are responsible for aggregate formation in lateritic soils. The cementation effects of these oxides appear to be principally due to the irreversibility of the colloids upon dehydration.

In addition to the cementing agents described above, it has been noted in some soils that carbonates may also contribute a weak cementation effect. Carbonates may be present in soil as a mixture with clay; they may also occur as secondary concretions

<sup>1</sup> Throughout this paper carbonate contents are expressed as percentages of calcium carbonate ( $\text{CaCO}_3$ ) by weight.

or as silt-size powders (7). It appears that the cementation effect is largely related to the carbonates mixed with clay and will probably become insignificant when the clay is hydrated.

### *Causes of Flocculation*

In the dispersion of a soil sample, the elimination of cementation effects is not sufficient to insure a stable soil suspension,<sup>2</sup> since the separated primary particles may again coalesce into floccules or aggregates. The magnitude of the electric charge (zeta potential) of clay particles, which is predominately negative, is considered the determining factor in the stability of a soil suspension. If the zeta potential is sufficiently high, the clay particles will repel each other when they collide during their constant haphazard (Brownian) movement in the suspension. If the zeta potential is low, there will be little repulsion, and the particles will coalesce as a result of a collision and settle out as a floccule.

The magnitude of the negative electric charge depends largely upon the combination of the mineralogical nature of the clay particle or mineral and the kind of adsorbed cations (positive ions). For that reason, with each type of clay mineral, the kind of adsorbed cations has an important bearing on the degree of dispersion obtained. The dispersive power of the following monovalent cations decreases according to the series: lithium (Li) > sodium (Na) > potassium (K), and ammonium (NH<sub>4</sub>). These cations cause the zeta potential of clay particles to be high, and thus may be classified as deflocculating cations. Common flocculating cations, which cause the zeta potential to be low, are hydrogen (H), calcium (Ca), and magnesium (Mg), all found in natural soils.

### *General Dispersion Procedure*

The essential requirements for dispersing a soil may be summarized as: (1) the elimination of cementation effects and (2) the prevention of flocculation. The removal of the cementing influences of the irreversible or slowly reversible oxides of iron and alumina is a difficult problem for which no generally acceptable solution has been found. But these oxides

occur in significant amounts only in lateritic soils; therefore their binding effects are usually assumed as negligible in most soils of the United States. The elimination of the cementation effects of the other soil colloids, organic matter and clay, can be accomplished by oxidation of the organic matter followed by rehydration of the clay particles. Flocculation of dispersed clay particles is prevented by replacing flocculating cations with deflocculating cations.

Organic matter in soils can be removed by oxidation with hydrogen peroxide; boiling soils with 6-percent hydrogen peroxide is a generally accepted method. This treatment is highly effective and produces only carbon dioxide and water as the byproducts of oxidation. The oxidation step in the dispersion procedure can be omitted with engineering soils of low organic content.

The rehydration of clay particles is accomplished by soaking the soil sample in distilled water for a specified period of time, followed by mechanical agitation<sup>3</sup> of the soil-water mixture. The more important methods of mechanical agitation are: stirring (4, 5, 8), shaking (9), boiling (10), rubbing (10), and bubbling air through the soil suspension (4, 11). High-speed stirring with apparatus patterned after the electric malted-milk mixing machine (4, 5) is perhaps the most extensively used method in engineering laboratories; the Wintermeyer apparatus (4, 11) is also used.

The prevention of flocculation in a soil suspension is more complicated than the removal of cementing influences. Because of the variable nature of soils, no specific method has been developed which is entirely satisfactory for all kinds. Techniques which are used attempt to control the kind of cations adsorbed on the clay particles so that the zeta potential of the particles will be high. As has been pointed out, clay particles with a high negative charge will repel each other upon collision, and the soil suspension will be in a stable condition.

To control the type of adsorbed cations it is necessary to accomplish two things, the cations which cause flocculation must be replaced with cations that promote deflocculation and the replaced cations and other free cations which might later interfere with

<sup>2</sup> In the mechanical analysis of soil, the term *soil suspension* refers to the soil-water mixture, which actually is a combination of a colloidal solution and a suspension of noncolloidal particles.

<sup>3</sup> A deflocculating agent is added before agitation.

the dispersion must be rendered ineffective. Both of these may be done by the one-step method of adding a deflocculating agent, or by a two-step method which involves a preliminary treatment of the soil sample before addition of the deflocculating agent.

In the one-step method, adsorbed flocculating cations are replaced by cations from the dissociated deflocculating agent; the replaced flocculating cations, together with other free cations in the soil suspension, are rendered ineffective either by precipitation as insoluble compounds or by the formation of soluble but undissociated compounds. This method is simpler and faster than the two-step method, and it has proved satisfactory for many types of soil.<sup>4</sup>

acid treatment because it requires additional expensive equipment (12). In the acid treatment, 0.2N hydrochloric acid is commonly used to leach the soil sample. The sample then must be washed free of electrolytes (13). The complete removal of chlorides is of special importance.

Table 1 summarizes the general procedure used for dispersing soil for mechanical analysis. The steps to be included will depend upon: (1) the nature of the soil, (2) the kind of deflocculating agent used, and (3) the fundamental objective of the mechanical analysis.

#### Deflocculating Agents

Sodium compounds, which dissociate to furnish sodium cations, are the most-exten-

TABLE 1  
GENERAL PROCEDURE FOR DISPERSING SOIL FOR MECHANICAL ANALYSIS

Step	Operation	Principal Purpose	Remarks
1	Hydrogen peroxide treatment	To remove organic matter	May be omitted for soils having low organic matter contents
2	Acid treatment (followed by washing)	To replace basic cations with hydrogen and to remove carbonates	Necessary only for some soils with certain deflocculating agents
3	Soaking in water	To rehydrate clay particles	Soaking period of at least 18 hours
4	Addition of deflocculating agent	To prevent flocculation by increasing the zeta potential	Deflocculating agent is added in solution form
5	Mechanical agitation	To effect thorough dispersion of the primary soil particles	High-speed stirring is the conventional method

A more complete dispersion may be obtained for some types of soil, with certain deflocculating agents, by use of the two-step method.<sup>5</sup> In general, soils benefited by the two step method are those containing appreciable amounts of carbonates. The predominant basic cations present in such soils are the strongly adsorbed divalent calcium and magnesium cations. The first step or preliminary treatment in this method involves replacing adsorbed basic cations with hydrogen cations and removing replaced cations and salts, such as carbonates, from the soil. The second step consists of replacing the hydrogen cations with deflocculating cations. The preliminary treatment can be accomplished either by electrodialysis or by leaching with dilute hydrochloric acid. The electrodialysis technique is not used as extensively as the

sively used deflocculating agents; the higher cost of lithium compounds makes them impractical to use.

After comparing the efficiency of several deflocculating agents for the dispersion of soil samples from four different states, Thoreen (14) concluded that sodium silicate is the most satisfactory deflocculating agent. In the ASTM and AASHTO standard methods of mechanical analysis, 20 ml. of 3-deg. Baumé sodium silicate solution<sup>6</sup> is used for the purpose of deflocculation.

Olmstead and his associates (13) found that sodium oxalate ( $\text{Na}_2\text{C}_2\text{O}_4$ ) is a satisfactory deflocculating agent for many types of soil. After extensive investigations of various deflocculating agents, Vinther and Lasson (15) concluded that sodium pyrophosphate ( $\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$ ) is the best for dispersing several types of clay.

<sup>4</sup> The ASTM and AASHTO methods of mechanical analysis utilize a one-step method.

<sup>5</sup> The International Society of Soil Science has adopted the two-step method.

<sup>6</sup> Prepared by dissolving sodium metasilicate crystals ( $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$ ) in distilled water.

The use of sodium metaphosphate ( $\text{Na}_4\text{P}_2\text{O}_{10}$ ) as a deflocculating agent for soil was investigated by Tyner (16). He obtained maximum dispersion for a wide variety of soils with this deflocculating agent. The effectiveness of sodium metaphosphate was explained as due to a more or less complete replacement of flocculating cations, particularly calcium, by sodium cations from the deflocculating agent. Since the exchange of cations results in the formation of a soluble but highly undissociated sodium calcium metaphosphate complex, the activity of calcium in a soil suspension is eliminated and soil particles will remain in a dispersed state. If calcium-carbonate colloids are present in a soil suspension, the dispersion of these colloids will also be stabilized due to the influence of sodium metaphosphate. Tyner believed that this stabilization is

TABLE 2  
PROPERTIES OF WISCONSIN LOESS SAMPLES  
FROM SOUTHWESTERN IOWA

Sample designation	A	B
Location of sample	Ridge at West Boundary	Hilltop at East Boundary
Liquid limit, %	30.9	48.3
Plastic limit, %	25.2	21.7
Plasticity index, %	5.7	26.6
Organic matter content, % by weight	0.30	0.21
Carbonate content, % $\text{CaCO}_3$ by weight	15.0	1.6

probably due to the coating of a gelatinous substance over the calcium-carbonate colloids.

#### DEVELOPMENT OF DISPERSION PROCEDURE FOR LOESS

The review of the principle of dispersion served as a background and guide for the development of a dispersion procedure especially adapted to the Wisconsin loess of southwestern Iowa. In addition, experiments were performed: (1) to evaluate several deflocculating agents, (2) to determine the need for hydrogen-peroxide and hydrochloric-acid treatments, (3) to evaluate a rubber stirring paddle and to determine stirring-time requirements. Before discussing the dispersion experiments, a brief description will be given of the loess samples used.

As a part of the southwestern Iowa loess investigation, more than 100 loess samples have been taken in the deep Wisconsin loess

area, which includes the whole or parts of twenty counties (17). This wind-blown soil material is thought to be associated with the glaciations of the Wisconsin stage. Major sources of supply were perhaps the raw surfaces of the newly deposited drifts in northwestern Iowa and the flood plains of the Missouri River and its tributaries which drained the drift areas.

The Wisconsin loess thicknesses in the southwestern Iowa area vary from over 100 ft. along the west boundary (east valley wall of the Missouri River) to about 17 ft. on hilltops near the arbitrary east boundary. In addition to the decrease in thickness with increasing distance from the valley wall, the loess becomes finer textured and its carbonate content decreases from a high of 18 percent ( $\text{CaCO}_3$  by weight) along the west boundary to a low of 1 percent at the east.

For the purpose of the dispersion experiments, two loess samples were selected to represent the extreme ranges in physical and chemical property variations; Sample A was taken at the west boundary and Sample B at the east. Both samples were obtained at a depth of 2 to 3 ft. below the top of the C horizon; Sample A was secured in the Hamburg pedological series and Sample B in the Marshall series. Properties which may influence the dispersion of the two samples are given in Table 2, which shows that the organic matter content of both samples was low. Particle-size distribution curves are shown in Figure 1. Differential thermal analyses on the samples indicate that both samples contain the same general kinds of clay minerals, apparently of the illite and montmorillonite types. Further mineralogical studies are being made to determine more definitely the mineralogy of the Wisconsin loess.

#### Evaluation of Deflocculating Agents

Mechanical analyses were performed for loess Samples A and B using solutions of sodium silicate,<sup>7</sup> sodium oxalate, sodium pyrophosphate and sodium metaphosphate<sup>8</sup> in

<sup>7</sup> The sodium silicate solution was prepared from sodium metasilicate crystals.

<sup>8</sup> The sodium metaphosphate solution was prepared from the commercial product Calgon, made by Calgon, Inc., Pittsburg, Pa. In Calgon, the sodium metaphosphate is buffered with a small amount of sodium carbonate to make it stable.

varying amounts as deflocculating agents. All four of these chemicals have been used by other investigators and have been found promising as deflocculating agents (14, 13, 15, 16). The concentrations used in this experiment are shown in Table 3. The 3-deg. Baumé solution of sodium silicate is specified by ASTM and AASHTO. A concentration of 0.5N was chosen for the other three chemicals for purposes of direct comparison and convenience of preparation.

2. The deflocculating agent was added in solution form.

3. The soil-water combination was mixed for 4 min. in a high-speed stirring apparatus of the malted-milk-mixer type.

This procedure is essentially the same as that specified in the ASTM standard method (D422-39), with three modifications: (1) the stirring time was increased from 1 to 4 min.; (2) a rubber stirring paddle was substituted for the conventional metal one;

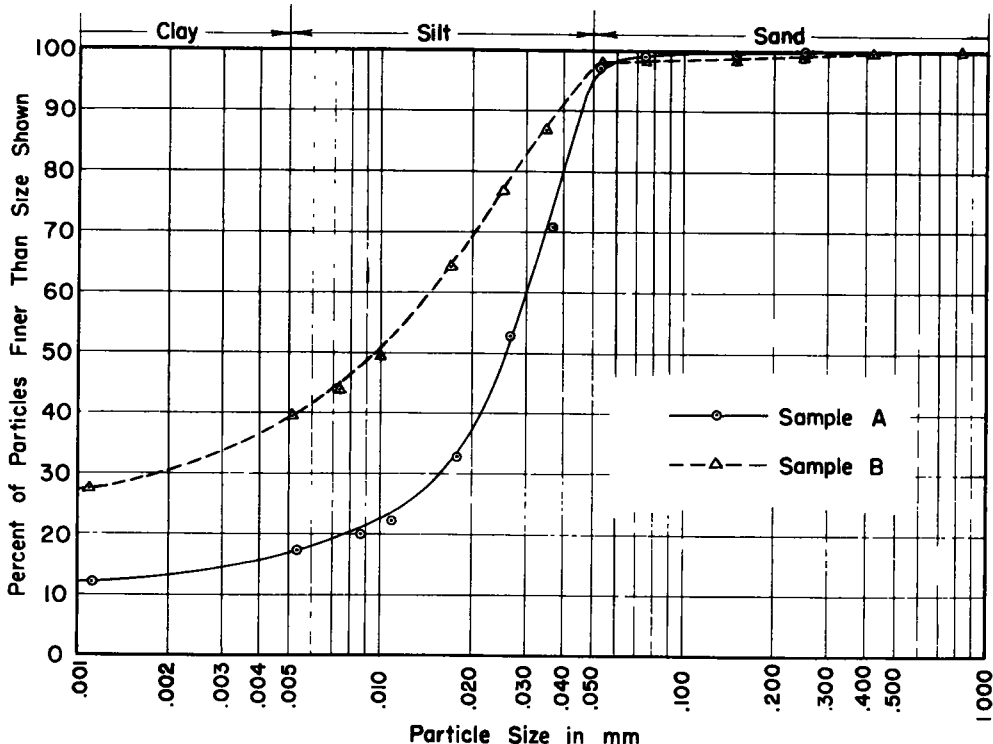


Figure 1. Particle-size accumulation curves for Samples A and B.

The only variable in the mechanical analyses was the type and amount of deflocculating solution added to the soil-water mixture prior to high-speed stirring. The hydrometer method was used for determining percentages of 0.005-mm. and 0.001-mm. clay. The efficiency of the deflocculating agents was rated on the basis of the magnitude of these clay size percentages.

The dispersion procedure adopted in this experiment was the following:

1. A 65-gram air-dry loess sample was soaked in distilled water for eighteen hours.

and (3) the hydrogen-peroxide treatment was omitted for Sample B, which had a plasticity index greater than 20. These modifications will be discussed later.

The hydrometer test was performed essentially according to the standard ASTM method, except that a correction was applied to the hydrometer reading to compensate for the direct influence of the deflocculating agent on the reading.

The effect of type and amount of deflocculating agent on the efficiency of dispersion of Samples A and B is shown in Table 3. The

table also shows the amounts of 0.005-mm. and 0.001-mm. clay measured without use of a deflocculating agent (other test steps were the same). The 0.005-mm.- and 0.001-mm.-clay-size data in Table 3 show a similar trend; this is illustrated graphically by Figure 2, which shows the relation between amount of sodium metaphosphate and degree of dispersion for loess Sample B. For this

oxalate solution greater than 20 ml. Sodium metaphosphate rated highest as a deflocculating agent for Sample A, with optimum dispersion being obtained with about 60 ml. of 0.5 N solution.

In the case of Sample B (Fig. 4), sodium silicate was decidedly inferior to the other three chemicals. Sodium metaphosphate rated slightly higher than sodium pyrophosphate

TABLE 3  
EFFECT OF TYPE AND AMOUNT OF DEFLOCCULATING AGENT ON THE EFFICIENCY OF DISPERSION

Deflocculating Agent			Sample A		Sample B	
Type	Conc. of Solution	Amount <sup>a</sup> (ml.)	Percent Finer Than		Percent Finer Than	
			.005 mm.	.001 mm.	.005 mm.	.001 mm.
Sodium silicate	3° Baumé	10	12.0	4.8	32.7	17.2
		20	12.2	5.8	34.3	18.0
		30	12.2	6.0	33.0	17.6
		40	12.0	6.0	33.3	18.8
		50	12.6	6.0	32.8	18.0
Sodium oxalate	0.5 N	10	14.0	7.6	35.0	20.0
		20	14.1	8.0	36.6	24.0
		30	14.2	Flocculated	37.8	26.0
		40	12.2	Flocculated	38.4	27.0
		50	10.2	Flocculated	39.0	27.5
		60			39.4	27.4
		70			39.8	26.4
Sodium pyrophosphate	0.5 N	10	13.6	8.0	34.6	20.2
		20	16.0	10.0	36.5	23.5
		30	17.1	10.8	38.2	25.2
		40	17.4	10.8	39.2	26.1
		50	16.9	10.5	39.6	26.6
		60	16.0	10.1	39.6	26.9
		70			39.2	27.0
		80			38.3	26.8
Sodium metaphosphate <sup>b</sup>	0.5 N	10	14.2	8.6	34.8	20.6
		20	16.4	10.0	36.4	23.0
		30	17.2	10.6	37.3	24.8
		40	17.5	11.3	38.1	26.0
		50	17.4	11.9	38.8	26.8
		60	17.0	12.0	39.2	27.4
		70	18.0	10.6	39.6	27.9
		80			39.8	28.1
		90			39.7	28.0
		100			39.4	27.9
		110			39.2	27.8
		120			39.8	27.8
		No deflocculating agent			11.7	1.6

<sup>a</sup> Refers to the amount of deflocculating solution used in dispersing a sample of 65 grams into a one liter soil suspension.

<sup>b</sup> In preparing the 0.5 N solution of sodium metaphosphate, *Calgon* was considered to have the gram-molecular weight of pure sodium metaphosphate.

reason, the deflocculating agents are compared graphically in Figures 3 and 4 on the basis of only the 0.001-mm.-clay measurements.

In Figure 3 the efficiency of the four chemicals as deflocculating agents for Sample A is compared. Sodium metaphosphate and sodium pyrophosphate were superior to sodium oxalate and sodium silicate; the latter chemical showed the least promise. Flocculation occurred with additives of sodium

and sodium oxalate, with 80 ml. of 0.5 N sodium-metaphosphate solution giving optimum dispersion. However, if 60 ml. of solution is used instead of 80 ml., only a slight loss in dispersive power is experienced, the decrease in the percent of 0.001-mm. clay measured being only 0.7 percent (see Fig. 4).

In consideration of the moderate degree of reproducibility of the hydrometer analysis, and in order to develop a uniform dispersion

procedure, it was decided to adopt 60 ml. of 0.5 N sodium-metaphosphate solution as the deflocculating additive for mechanical analyses of the Wisconsin loess. Results of mechanical analyses of Samples A and B using this sodium-metaphosphate additive are plotted in Figure 1.

Figures 3 and 4 also show that additives of sodium-silicate solution in excess of 20 ml. do not significantly improve the degree of dispersion of the two loess samples. Twenty milliliters is the amount of sodium silicate solution recommended in the ASTM and AASHTO dispersion procedures.

### Hydrogen-Peroxide Treatment

The main purpose of the hydrogen-peroxide treatment is, as mentioned previously, to

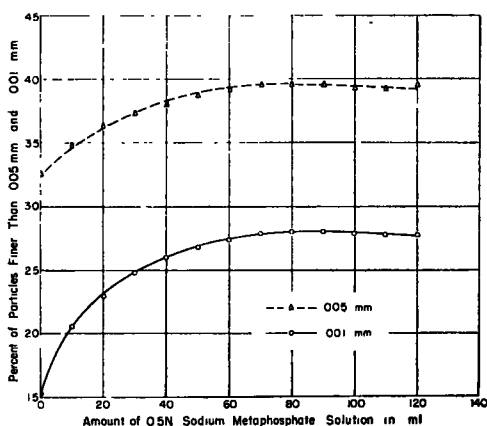


Figure 2. Relation between amount of sodium metaphosphate and degree of dispersion for Sample B.

remove the cementing influences of organic matter from soil. Since the organic content is low in all Wisconsin loess samples taken from southwestern Iowa, this treatment appeared to be unnecessary. However, it was considered desirable to determine experimentally the effect of the hydrogen-peroxide treatment on Sample B (P.I. = 26.6 percent), because the ASTM dispersion procedure specifies that soils having a plasticity index greater than 20 percent shall be treated with hydrogen peroxide (6 percent  $H_2O_2$ ) for the purpose of aiding dispersion, rather than for the removal of organic matter. The deflocculating agents used for this experiment were sodium metaphosphate (60 ml. of 0.5 N solution) and

sodium silicate (20 ml. of 3-deg. Baumé solution). Sodium silicate was used because it is the deflocculating agent specified by the ASTM procedure.

The hydrogen-peroxide treatment of Sample B was performed in accordance with ASTM directions. Hydrometer analyses to determine

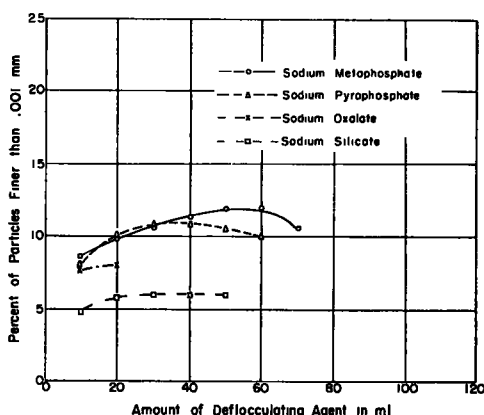


Figure 3. Comparison of efficiency of deflocculating agents for the dispersion of Sample A.

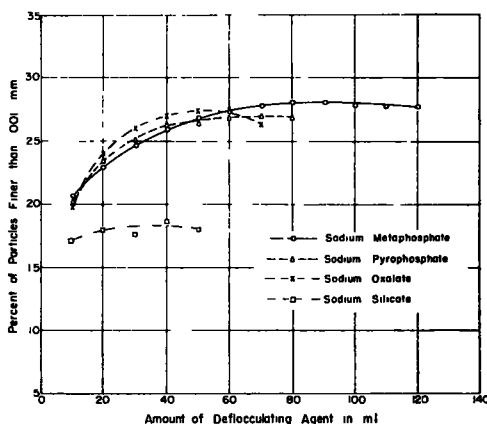


Figure 4. Comparison of efficiency of deflocculating agents for the dispersion of Sample B.

the percent of particles finer than 0.005 mm. and 0.001 mm. were made as previously described in connection with the evaluation of deflocculating agents. The test data given in Table 4 indicate that the hydrogen-peroxide treatment did not improve the degree of dispersion of Sample B, as reflected in the magnitude of the 0.005-mm.- and 0.001-mm.-clay determinations. For that reason this



treatment is omitted in the dispersion procedure for Wisconsin loess samples which have high plasticity indices but low organic-matter contents.

### *Acid Treatment*

The value of the hydrochloric-acid treatment as an aid to more efficient dispersion was investigated by experiments with highly calcareous ( $\text{CaCO}_3 = 15.0$  percent) loess Sample A. The purpose of the acid treatment has previously been discussed and is summarized in Table 1.

One hundred grams of air-dry loess was soaked in 1,650 ml.<sup>9</sup> of 0.2 N hydrochloric acid, with occasional stirring and shaking, for 24 hr. At the end of the soaking period, the acid-soil mixture was poured onto filter

TABLE 4  
EFFECT OF HYDROGEN-PEROXIDE  
TREATMENT ON DEGREE OF  
DISPERSION OF LOESS  
Sample B

Deflocculating Agent	Hydrogen Peroxide Treatment	Percent Finer Than	
		.005 mm.	.001 mm.
60 ml. 0.5 N sodium metaphosphate solution	Yes	38.6	27.2
	No	39.2	27.4
20 ml. 3° Baumé sodium silicate solution	Yes	33.0	16.8
	No	34.3	18.0

paper in a Buchner funnel, and the acid was drawn through by means of an aspirator. The soil was then washed with distilled water until complete removal of chlorides had been accomplished, as indicated by testing the leachate with silver nitrate; chlorides are considered eliminated when the leachate shows no cloudiness when a few drops of silver nitrate are added. After the removal of the chlorides, the washed sample was air-dried prior to preparing 65 grams for the mechanical analysis.

The validity of using the acid treatment in engineering mechanical analyses of calcareous soils is questionable, since the carbonates, an inorganic constituent of the soil, are removed and are not represented in the particle-size-distribution determination. With a highly calcareous soil, such an omission would

<sup>9</sup> Milliliters of 0.2 N HCl = (1.1) (% $\text{CaCO}_3$  in soil) (grams of soil). This equation provides an excess of HCl over that needed to remove the carbonates.

misrepresent the soil's texture. This fact, coupled with the added time required for the acid treatment, makes it advantageous to use a deflocculating agent whose dispersive power is not impaired by the presence of carbonates. The data in Table 5 indicate no significant evidence of increased dispersion with the acid treatment when sodium metaphosphate is used as the deflocculating agent. It was, therefore, considered inadvisable to include the acid treatment in the dispersion procedure for Wisconsin loess.

### *Type of Stirring Paddle*

The high-speed electric stirring apparatus described in the ASTM and AASHTO standard methods of mechanical analysis utilizes a replaceable metal paddle which is subject to quite rapid wear by the abrasive action of

TABLE 5  
EFFECT OF ACID TREATMENT ON DEGREE OF  
DISPERSION OF LOESS  
Sample A<sup>a</sup>

Acid Treatment	Percent of Particles Finer Than	
	.005 mm.	.001 mm.
Yes	17.2	13.0
No	17.0	12.0

<sup>a</sup> 60 ml. of 0.5 N sodium-metaphosphate solution was used as the deflocculating agent.

hard soil grains in the soil-water mixture. Frequent replacement of the paddle is necessary in order to insure a standard intensity of stirring. Because of the wear factor, the soils laboratory of the Iowa State Highway Commission has for several years been using a wear-resistant rubber paddle with highly satisfactory results. The rubber paddle, cut from an automobile inner tube, is  $\frac{7}{8}$  in. square and  $\frac{1}{8}$  in. thick and is mounted on the stirring shaft in the same manner as the metal paddle.

A series of mechanical analyses were performed on Samples A and B in order to compare the stirring efficiencies of the rubber and metal paddles. Sodium metaphosphate (60 ml. of 0.5 N solution) was the only deflocculating agent used. Table 6 shows the effect of type of stirring paddle on the magnitude of the 0.005-mm.- and 0.001-mm.-clay determinations. The data indicate that the rubber paddle compares favorably with the

metal paddle and possibly may give even slightly better dispersion. As a result of this experiment, the rubber paddle was adopted for use with the Wisconsin loess.

### Length of Stirring Period

The length of the stirring period used with the high-speed malted-milk-mixer-type apparatus is an important part of the dispersion procedure. The optimum stirring time should give maximum dispersion of the soil-water mixture on the one hand, and minimum degradation<sup>10</sup> on the primary soil particles on the other. A long stirring period is desirable in achieving a high degree of dispersion, but it is objectionable if degradation occurs.

TABLE 6  
EFFECT OF TYPE OF STIRRING PADDLE AND LENGTH OF STIRRING PERIOD ON DEGREE OF DISPERSION<sup>a</sup>

Type of Stirring Paddle	Stirring Period (min.)	Sample A		Sample B	
		Percent Finer Than		Percent Finer Than	
		.005 mm.	.001 mm.	.005 mm.	.001 mm.
Standard metal paddle	1	14.0	9.3	36.3	24.5
	4	15.5	10.6	37.4	25.7
Square rubber paddle	1	14.8	9.6	38.0	25.5
	4	17.0	12.0	39.2	27.4
	10	17.2	12.0	39.5	27.9
	16	17.4	12.8	39.6	28.7
No stirring	0	0	0	7.0	0

<sup>a</sup> 60 ml. of 0.5 N sodium-metaphosphate solution was used as the deflocculating agent.

The amount of degradation that occurs during a given stirring period depends largely upon both the soil texture and the hardness of the individual particles. Wintermeyer (11) found the coarse sand fraction (2.0 mm. to 0.42 mm.) to be most affected by degradation during the stirring operation. The coarse sand fraction in Wisconsin loess (see Fig. 1) is small enough to be negligible, and since quartz is believed to be the predominant mineral in the nonclay sizes, the soil particles most subject to degradation are likely to be extremely hard. Because of this, the action of degradation is probably of minor importance in the case of loess.

The effect of length of stirring period on the degree of dispersion is shown in Table 6

<sup>10</sup> The term *degradation* refers to the break-up of the primary soil particles into smaller ones.

and in Figure 5. It will be noted that for both Samples A and B the increase in the percentage of particles finer than 0.005 mm. and 0.001 mm. is very slight for increases in stirring time above four minutes.

A 1-min. stirring period is specified in both the ASTM and AASHTO dispersion procedures, presumably to avoid the degradation which may result from a longer stirring time. Since the action of degradation appeared to be of minor importance in stirring loess, especially with a rubber paddle, a stirring period of 4 min. was adopted for use with the Wisconsin loess. The soils laboratory of the Iowa State Highway Commission uses the 4-min. stirring period with all types of Iowa soils.

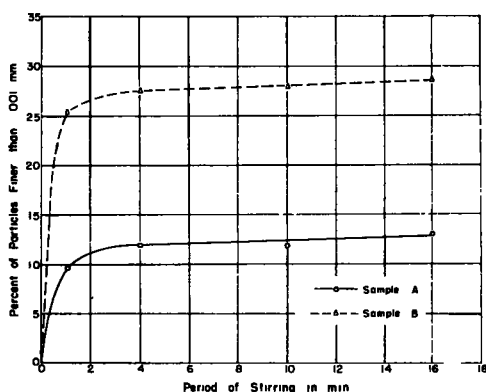


Figure 5. Relation between length of stirring period and degree of dispersion.

### CONCLUSIONS

The following are some of the more important conclusions drawn from the investigation reported in this paper:

1. The use of deflocculating agents other than sodium silicate, which is specified in the ASTM and AASHTO standard methods of mechanical analysis, results in a more complete dispersion of the Wisconsin loess of southwestern Iowa.

2. Among the deflocculating agents which were compared experimentally, sodium metaphosphate is the most efficient in dispersing the Wisconsin loess. The amount of sodium metaphosphate adopted for dispersing Wisconsin loess samples for mechanical analysis determinations is 60 ml. of 0.5 N solution.

3. Wisconsin loess samples having plasticity

indices greater than 20 percent do not need to be treated with hydrogen peroxide, providing the organic matter content is low.

4. If sodium metaphosphate is used as the deflocculating agent, the hydrochloric-acid treatment is not advantageous or desirable for Wisconsin loess samples having high carbonate contents.

5. To avoid frequent replacement of the standard metal stirring paddle due to wear, the rubber paddle used by the soils laboratory of the Iowa State Highway Commission can be used without loss of stirring efficiency; there may even be a slight increase in stirring efficiency.

6. A stirring period of 4 min. with the high-speed malted-milk-mixer-type stirring apparatus is considered adequate for Wisconsin loess samples.

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