

SEPARATION, FRACTIONATION AND MINERALOGY OF CLAYS IN SOILS

JAMES H. HAVENS, *Research Chemist*, JAMES L. YOUNG, JR., *Research Geologist* AND R. F. BAKER, *Research Engineer, Kentucky Department of Highways*

SYNOPSIS

This paper describes a working method for separation, fractionation and identification of colloid and near colloidal clay minerals in soils. The technical information presented pertains to super-centrifugation, electron-microscopy and X-ray diffraction. On the basis of the techniques described, twenty-two samples have been investigated in conjunction with a soil study of pumping pavements.

Soils were first dispersed and separated by gravity sedimentation. Fractionation was accomplished by controlled super-centrifugation. The separated fractions were purified and then analyzed by X-ray diffraction. Computed size fractions were checked by shadow castings and lineal dimensions on electron micrographs.

Results include identification of the mineral or minerals present and the properties of the natural sample from which the colloidal fractions were extracted.

These methods furnish a basis for more extensive research relating the behavior of the clay minerals and their contributions to the properties of soils.

Many of the problems in soil mechanics confronting the engineer are essentially problems in chemistry, geology, and mineralogy. This implies the need for further investigation of the constituents of soils and their contributions to the properties of the material as a whole.

Many of the general trends and relationships in soil science concentrate attention on the fine particles. Coarse-grained constituents are largely inert and provide inactive bulk to the soil. Surface phenomena are slight because of the limited amount of surface exposed. Logic indicates a progressive increase in the reactivity, particularly in surface phenomena, with progressive decrease in particle size. For illustration, imagine a 1-cm. cube having a surface area of 6 sq. cm. cut into cubes 0.001 micron on an edge. The particles would then expose areas totaling $1\frac{1}{2}$ acres. It is obvious that surface effects would be greatly accentuated.

The words "clay" and "clay mineral" may be used interchangeably throughout this paper to identify known or unknown mineral suites, but not with the intent to specify size. In general, "clay" is a term referring to any or all of the naturally occurring finely divided hydrated aluminum silicates. The "clay minerals" are a known series of mineral suites, all falling in the monoclinic system. They may be separated into three major groups

on the basis of the properties which they exhibit.

In Table 1, several isolated properties of the clay mineral groups are listed in order to emphasize some of their inherent characteristics, and their influence in corresponding soil mixtures. There are other miscellaneous clay minerals which are not listed here.

Table 1 presents the most common and the most important groups of the clay minerals. The properties of these groups infer a trend in reactivity. That is, kaolinite should be the most stable and the most inert, while montmorillonite should be very reactive and least stable. Illite should exhibit properties intermediate between kaolinite and montmorillonite.

For any clay mineral, there are definite size-behavior relationships. Base-exchange, for instance, is a function of the surface area exposed; and Johnson and Davidson (4)¹ describe an equation for the relation:

$$X = (2 \times 10^{-5})S \quad (1)$$

where X = exchange capacity—m.e. per 100 g.

S = specific surface—sq. cm. per g.

¹ Italicized figures in parentheses refer to the list of references at the end of the paper.

Theoretically this property is a manifestation of unsatisfied bonds and the number of these broken bonds is greatly multiplied by each degree of subdivision.

Montmorillonite has a high affinity for water and will expand its lattice in order to accommodate the water. In contrast, kaolinite has only a very slight affinity for water. Further, the plasticity of kaolinite is greatly altered by the addition of small amounts of montmorillonite.

Clay minerals occur in the minus 5-micron fraction of soils and dominate the minus 1-micron fraction. It has been postulated by some that different clay minerals in a soil will exist within discrete size levels. That

engineering soil tests, as well as the CBR test, were made. Thus, the physical properties and some indication of the behavior of each soil were known prior to the analysis for clay mineral content. In Table 2 there is a list of the physical properties of the 22 samples thus far analyzed for mineralogic characteristics.

PROCEDURES

Pre-Treatment—Two to three hundred grams of soil passing the No. 40 sieve were taken as a sample. It was dispersed in a 4-liter beaker of distilled water by adding 5 cc. of concentrated ammonium hydroxide to the soil and water and mixing for 15 min. with an electric

TABLE 1
MAJOR CLAY MINERAL GROUPS AND SOME OF THEIR PROPERTIES

Clay Mineral	Properties of Clay Minerals			Properties They Contribute to Soils		
	Base Exchange Capacity ^d	Height of Unit Cell ^a	Plasticity Index for Pure Clay ^b	Plasticity Index for Soil ^c	Relative Static Compaction ^d	Relative Permeability ^d
Kaolinite Group 4 members	3-15	7.2 Angstroms	29.6	6.2	Compacts a small amt. rapidly	Very permeable
Illite Group 1 member	20-40	10.0 Angstroms	No figures available	No figures available	Intermediate	Intermediate
Montmorillonite Group 3 members	60-100	12-26 Angstroms	91.1 (Calcium Base) 428 (Sodium Base)	34.7 (Calcium Base) 103.8 (Sodium Base)	Compacts a large amt slowly	Impermeable

^a Hanawalt Card File (1).

^b After Endell, Loos, and Breth (2).

^c Based on a (Quartz) 7:3 (Clay) mixture after Endell, Loos, and Breth (2).

^d R. E. Grim, "Clay Minerals in Soils and Their Significance" (3).

Italicized figures in parenthesis refer to the list of references at the end of the paper.

is, in an illite-kaolinite mixture, kaolinite is more abundant in the 1-micron to 0.2-micron fraction and practically extinct in the minus 0.1-micron fraction, while illite will exist throughout the smaller fractions. For this reason the minus 1-micron fractions of the soils tested in this study were separated into discrete sub-fractions.

MATERIALS TESTED

The soils analyzed for clay mineral content were selected from samples obtained by the laboratory for a study of the pumping action of rigid pavements. These samples were taken from beneath the pavement and from locations of known performance. In conjunction with the pumping study (5), all routine

mixer. The material was left in the beaker and after 24 hours, the top 10 cm. containing particles approximately 1 micron and smaller were siphoned off. The time required for particles to fall 10 cm. through the water was computed and a graph plotted, as shown in Figure 1. The process of sedimentation was repeated until a 5-gal. bottle was filled. The material was then fractionated by centrifugation.

Fractionation—Fractionation of small particles was based on sedimentation principles described as Stoke's Law:

$$\frac{D}{2} = \sqrt{\frac{9/2n \, dx/dt}{(dp - dm)g}} \quad (2)$$

where, D = effective diameter of particle

n = viscosity of medium
(in poises)

dp = density of the particle
(assume 2.65)

dm = density of the medium

x = distance of fall

t = time to fall x distance

g = acceleration of gravity

ern centrifuge methods make it possible to multiply the effective force several thousand times. By adjusting the force, optimum time intervals may be obtained for the desired particle size.

Fortunately, clay minerals exhibit colloidal properties to such an extent that they can be dispersed and suspended in water, but still retain their "matter in mass" identities to such extent that they can be treated as discrete particles or crystals, exhibiting known surface phenomena, characteristic of so many finely divided materials and lyophobic colloids.

TABLE 2
LIST OF SAMPLES AND THEIR PHYSICAL PROPERTIES

Sample Number	Geologic Area	CBR	Vol. Change %	Maximum Density	Opt. M.C.	Specific Gravity	L.L.	P.L.	P.I.	Sand %	Total Sample			Col-loids (100% Passing No. 4) %	Col-loids (100% Passing No. 40) %	PRA Classification
											Silt %	Clay and Col-loids %	Col-loids %			
206	Trenton	3	10.5	96.6	24.3	2.74	50	29	21	20	20	60	19	20	23	A-7
293	Trenton	3	1.4	93	29	2.81	62	27	35	18	27	55	34	37	37	A-6
340	Pottsville	5	4.3	105	20	2.74	39	21	18	28	36	36	18	19	21	A-5
341	Pottsville	1	7.2	104	21	2.76	44	28	16	23	20	48	16	18	19	A-5-7
228	St. Louis Ls.	1	0.1	98	23	2.72	63	59	4	21	26	53	33	35	38	A-5
325	St. Louis Ls.	2	10.0	105	19	2.68	37	16	21	6	40	45	24	24	24	A-4
209	Loess	12	3.3	114	14	2.69	23	17	6	29	44	27	13	14	16	A-4
232	Loess	18	2.7	112	16	2.66	29	20	9	9	62	29	12	12	13	A-4
270	Alluvium	4	4.2	113	15	2.66	34	20	14	33	30	37	18	20	20	A-5-7
201	Alluvium	10	5.2	101	22	2.68	37	26	11	6	48	46	20	20	5	A-5
203	Alluvium	6	3.4	100	23	2.72	48	26	22	4	40	56	5	5	22	A-7
229	Glacial	5	8.2	103	20	2.67	40	24	16	12	43	45	22	22	20	A-5
258	Glacial	8	3.3	108	19	2.70	37	20	17	14	46	40	19	19	20	A-6
256	Salurian	5	5.4	111	16	2.70	32	19	12	22	44	33	16	16	17	A-5
287	Salurian	12	2.3	107	18	2.70	35	21	14	22	44	34	17	19	20	A-5
260	Devonian	1	8.7	100	23	2.75	62	29	33	8	33	59	33	34	36	A-7
264	Devonian	3	1.4	97	24	2.70	60	20	40	24	27	49	26	26	32	A-6
281	Eden	2	7.1	107	21	2.74	45	26	19	19	31	50	25	29	30	A-5-7
202	Eden	2	11.5	112	17	2.76	33	18	15	12	55	53	28	28	29	A-5-7
239	Cynthiana	1	12.2	104	21	2.77	53	33	20	6	29	66	34	34	35	A-5-7
351	Maysville	2	7.1	105	19	2.75	49	27	22	31	29	40	18	23	24	A-5-7
204	Maysville	5	4.5	102	21	2.73	47	27	20	12	36	52	25	25	27	A-7

Thus, if the rate of fall of spherical particles is measured;² and the viscosity of the liquid, the difference in density between the particle and the liquid, and the constant of the gravitational field are known, the particle size can be calculated. This statement is true for sedimentations in centrifuges as well as ordinary sedimentations. Accordingly, by adjusting conditions a process can be set up which will yield particles of any desired size.

Larger particles may be isolated by sedimentation under the force of gravity, but as the particle size decreases, the time factor becomes infinite as shown in Figure 1. Mod-

² It is believed that plate-like particles cut a sphere during sedimentation (4).

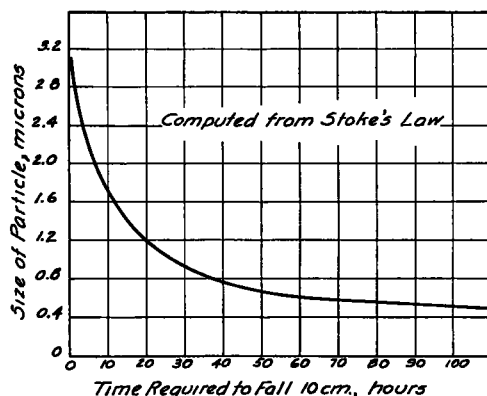


Figure 1. Particle Size Versus Time Required to Fall 10 cms. through Water

It was first decided to separate four fractions from the sample. The conditions of centrifugation were adjusted for recovery of particles 1 to 0.2 microns, 0.2 to 0.1 microns, 0.1 to 0.05 microns, and below 0.05 micron in diameter. Later the last two fractions were combined to contain the minus 0.1-micron sizes. A Sharples super centrifuge was used for the separation.

Schachman (6), Hauser and Lynn (7) and Seay (8) give a set of working equations for the use of the Sharples super centrifuge:

$$Y = \frac{18QK_1N6}{\pi(R_2^2 - R_1^2)D^2W^2P} \cdot \left[\frac{R_2}{2} \ln \frac{R_2}{X_0} - \frac{R_1^2}{2} \ln \frac{R_2}{X_0} + \frac{X_0^2 - R_2^2}{4} \right] \quad (3)$$

where,

Y = vertical distance from bottom of centrifuge bowl to a point where the particle comes to rest on the bowl wall

R_1 = radius of air column in operating centrifuge bowl (cms.)

R_2 = inside radius of centrifuge bowl (2.22 cm.)

X_0 = distance from axis of rotation of bowl at which particle begins to settle (cm.)

Q = rate of feeding sol. into centrifuge bowl (cc. per sec.)

$$K_1 = \frac{R_2^2 - R_1^2}{\frac{3}{4}R_1^4 + \frac{R_2^4}{4} - R_1R_2 - R_1^4 \ln \frac{R_1}{R_2}} = 1.109$$

N = viscosity of dispersion medium (poises)

D = diameter of clay particle (cm.)

W = angular velocity of rotation (radians per sec.)

P = difference in density of dispersed and dispersing phases.

The solution of equation (3) is difficult, requiring a family of curves, or solution by determinates. Simplifying the equation by substituting numerical values for constants and rearranging, the equation becomes:

$$Q = \frac{112.7D^2P}{N} \quad (4)$$

The terms inside the brackets of equation (3) are the centrifuge factor and are called C for simplification. Actually C is a constant, and the plot of C versus Y used by Seay and Schachman was adopted since the dimensions of the bowls used were the same.

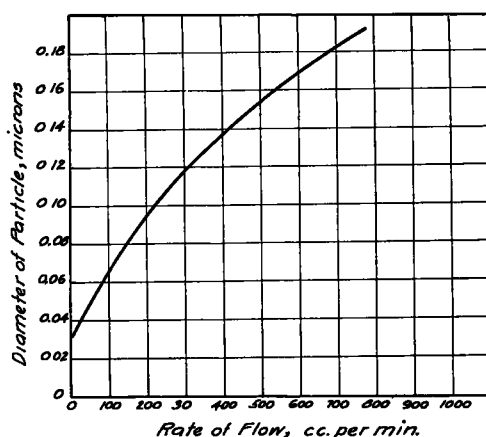


Figure 2. Calculated Particle Size Versus Rate of Flow of Sol

Conditions were adjusted such that the minimum size retained was 0.2 micron at $Y = 20$ cm., particles below 0.2 micron remained in suspension and passed through. (Larger fractions are contaminated to some extent by the smaller sizes.) After recovery of the fraction and sol, the sol was run through again under more restricted conditions such that 0.1 micron was the minimum size retained at $Y = 20$ cm. until fractions greater than 0.2 micron, 0.2 to 0.1 micron, and 0.1 to .05 micron had been obtained. Particles smaller than 0.05 micron were finally obtained by precipitation.

A plot of Q versus D is advantageous for quick reference. See Figure 2.

Control of conditions was completed by calibration of rate of flow through appropriate drags, nozzles, or orifices against various pres-

sure heads, using suitable means of maintaining a constant head for each height. See Figure 3 for calibration curves and Figure 4 for a diagram of apparatus.

Eighteen liters of soil were collected, in which the maximum particle size was 1 micron. A 0.003-in. cellulose acetate liner was inserted into the centrifuge bowl to collect the fraction. The head was adjusted to give the desired size. The speed of the centrifuge was maintained at 25000 R.P.M. by use of a voltage regulator. The liner was removed and a new one inserted after each fraction was collected. The fraction was dried on the acetate sheet after which the particles were easily chipped off. As indicated in Fig-

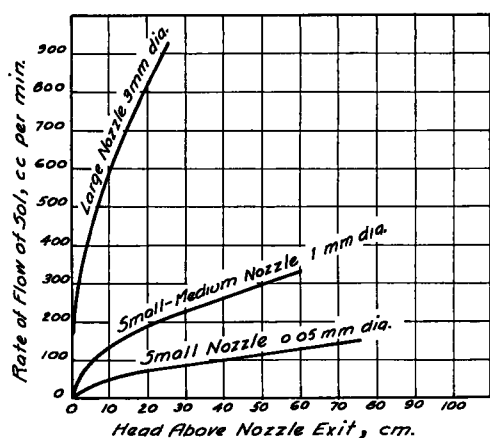
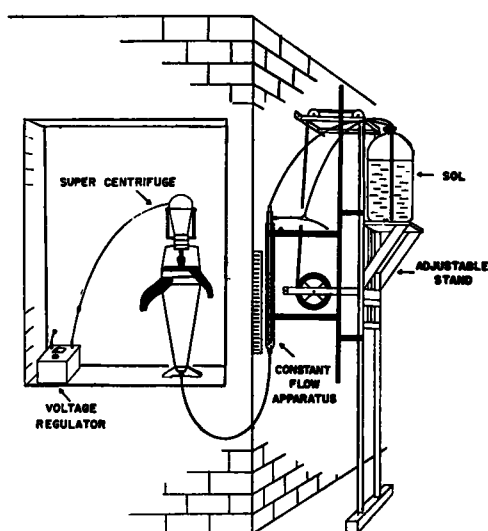


Figure 3. Calibration of Flow Rates

ure 2 it is impractical to separate fractions below 0.05 micron centrifugally. This final fraction is precipitated by adding NaCl to the sol from the last fraction.

Flow Regulation—Flow of sol was controlled by means of the flow regulator apparatus shown in Figure 4. The sol was forced out of the bottle by blowing into the system of rubber tubing and starting the siphoning action. The sol flowed into the constant flow apparatus and filled the outer tube until it reached the bottom of the inner tube. As the sol continued out a partial vacuum was set up in the air space of the bottle and the sol stopped flowing out of the bottle. The sol in the outer tube then flowed out to the centrifuge until the level dropped below the bottom of the inner tube. At this time an air

bubble entered the bottom of the inner tube and rose to the air space in the bottle, thus releasing the vacuum and allowing more sol to flow into the outer tube and raising the level again to the bottom of the inner tube. The cycle was repeated again and again and created a constant head of sol at the bottom of the inner tube which varied about 1 mm. due to bubbling of air. The height of this head was changed by adjusting the inner tube so that it was level with the desired height measured on a scale marked on the wall beside the apparatus.



APPARATUS FOR CONTROLLED FRACTIONATION OF A CLAY SOL

Figure 4. Flow Regulator Apparatus

Post Treatment—Treatment of the fraction after separation involved leaching out the soluble material such as iron and carbonates with HCl and mild heat, followed by H_2O_2 with boiling to remove organic materials, finally washing, centrifuging down with acetone and drying.

Electron Microscopy—In order to check the size of separated fractions for complete assurance of the accuracy of separation, it was decided to observe the sizes by means of the electron microscope.

Optical microscopy is limited by the wave length of visible light—that is, even by ultra-violet light, 0.36 micron or 3600 Å—whereas particles were separated into fractions smaller

than 0.05 micron. Obviously these particles are beyond the limit of optical observations. The electron microscope utilizes the shorter wave-lengths of an electron beam in addition to the advantage of higher magnification. Fortunately, clay minerals demonstrate sufficient opacity to the electron beam to provide good detail.

Specimen mounts were prepared using one part U.S.P. collodion (containing 24 percent alcohol) and four parts amyl acetate, filtered and allowed to age 24 hours. Mounting films were prepared by dropping the collodion from an eye-dropper held 1 in. above the surface

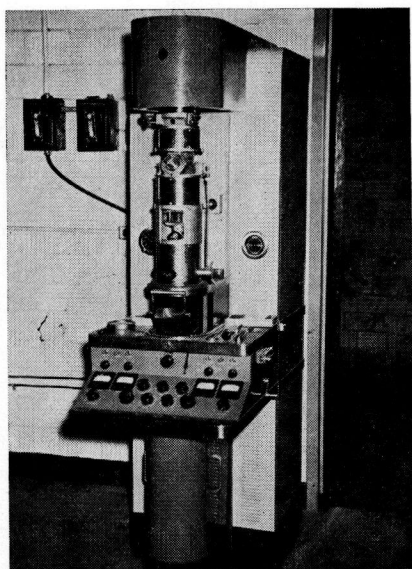


Figure 5. Electron Microscope

of water in a container having a diameter of 12 in. or greater. The film was allowed to stand until the diffraction patterns disappeared. Prepared screens, approximately 3.5 mm. in diameter, of 275-300 wire mesh were dropped onto the film (burred edges upward so as not to puncture the film), then a clean glass microscope slide was cautiously placed onto the floating film, covering the screen. The film was split with a scalpel on each long side of the slide extending radially outward to the edge of the container. A pair of crucible tongs was inserted into the slits and the slide was held to the surface of the water while excess film was lapped over and

onto the top of the slide. Then the slide was raised from the surface, inverted, and the excess film removed from the bottom edge with the fingers. The slide was placed on end and dried. Thus, the screen supported a microfilm and was held to the slide until the specimen was mounted and ready for observation.

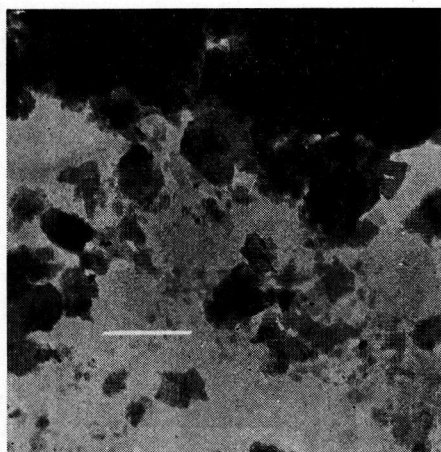
The clay sample to be observed was re-dispersed and re-suspended in water (only a few cubic centimeters are necessary and must be of such concentration that only a very slight trace of opacity is apparent); and, with the aid of a micro-pipette, a small drop of the sol was placed onto the screen and allowed to dry. Inspection of the sample thus prepared can be made to a good advantage under an optical microscope. In this manner it is possible to determine the distribution and concentration of the particles in order to assure good results and facilitate observation.

In this study films were prepared varying in thickness from 3 drops to 10 drops. It was found that in some instances 3, 4, and 5-drop films were strong enough to withstand the force of the electron beam, but 7, 8, 9, and 10-drop films gave better strength and showed less boiling due to heat generated in the particle by the beam. These thicker films were sufficiently transparent to give good contrast. Fractions over 0.2 micron, 0.2 to 0.1 micron, 0.1 to .05 micron, and below 0.05 micron were prepared and observed. Electron micrographs were taken at a magnification of approximately 7,300 diameters. On this basis 0.73 cm. on the negative represents 1 micron. This dimension is shown as a white line on photographs shown in Figures 6 and 7.

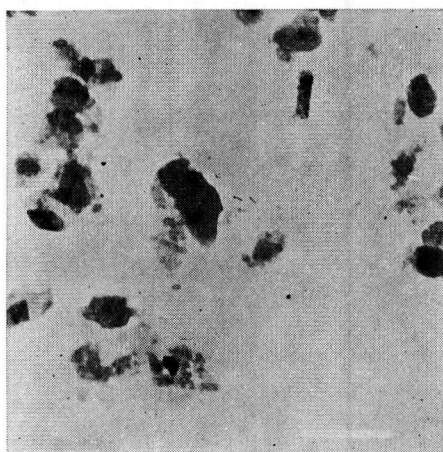
In order to estimate the vertical dimensions, the 0.2- to 0.1-micron fraction was shadow-cast. In this process, a companion specimen was used. The shadow was cast by plating chromium, vaporized from a hot filament situated so that the angle of incidence caused the particles to shadow areas such that the horizontal lengths of the shadows were five times the vertical dimensions of the particles. This specimen was observed again in the scope and photo-micrographs made. See Figure 7.

The photo-micrographs in Figure 6 substantiate the validity of the procedure used

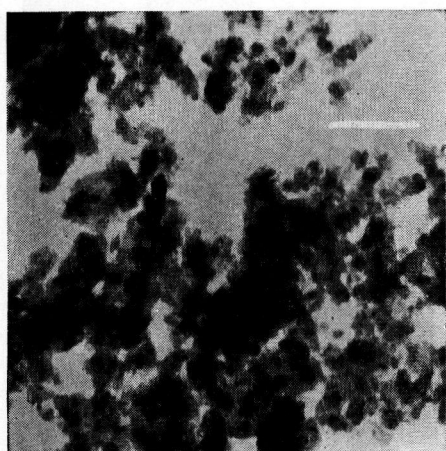
in fractionation of the particles on the basis of sizes. It is to be noted that smaller sizes this with 10 Angstrom units which is the height of the unit cell for Illite.



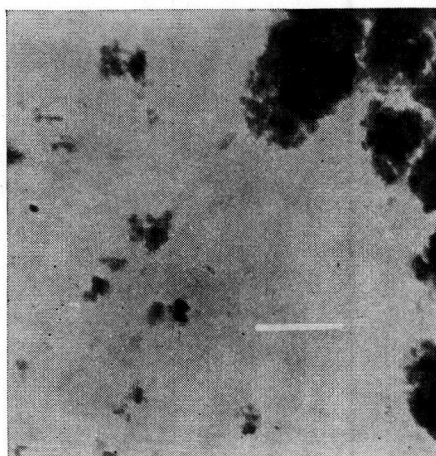
A. Greater than 0.2 micron



B. 0.2 to 0.1 micron



C. 0.1 to 0.05 micron



D. Less than .05 micron

Figure 6. Electron Photomicrograph of Fractions from Soil No. 202. White line in each instance represents a distance of 1 micron in the sample

contaminate the larger fractions, but that the larger sizes conform to the specified limits. The photographs show considerable aggregation of particles, but many isolated particles appear as flakes or thin sheets.

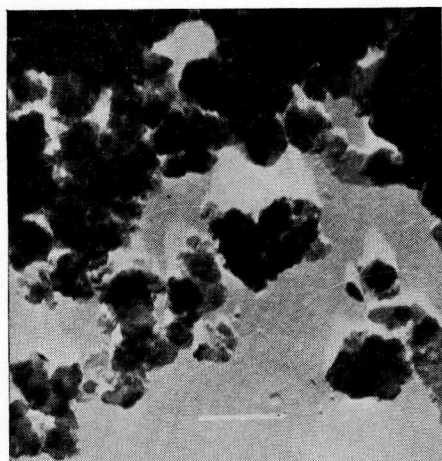
The shadow-graphs in Figure 7 show some isolated particles which cast shadows only 0.2 micron horizontally. The corresponding vertical dimension approximates 400 Angstrom units, and it is of interest to compare

X-ray Diffraction—One of the latest methods for analysing macroscopic to sub-microscopic substances is based on the Bragg (9) method of X-ray analysis.

This method utilizes the short wave lengths of X-rays and the principles of diffraction gratings. In X-ray crystallography the wave lengths approximate the magnitude of the atomic interstices separating the planes of symmetry within the crystal. Since each of

the planes of symmetry is a reflecting plane and the incidence angle approaches the critical point, some rays are scattered and constructive interference results, provided the fundamental equation is satisfied. These conditions are described in equation (5).

The specimen holder was then placed in a Debye-type cylindrical camera, (Figure 8), film was clamped around the inner periphery, the camera sealed, and the exposure started. Two hours of exposure was usually sufficient using 40 kilo-volts and 15 milli-amperes, cop-



A



B

Figure 7. Electron Photomicrographs of Shadow-Cast Fraction 0.2 to 0.1 Micron—Soil No. 202

$$n \lambda = 2 d \sin \theta \quad (5)$$

where, λ = wave length of X-ray (Cu. = 1.54 Angstrom units)

n = whole number (small multiple)

d = distance between planes of symmetry (in Angstrom units)

θ = angle of reflection where constructive interference occurs

The clay minerals lend themselves readily to the powder diffraction method. The advantage of the powder method is in the fact that a fine powder orients all planes to the beam and produces a continuous arc registered on the film. The distance is a characteristic constant for any crystalline identity. The constants used are given in the Hanawalt Index (1).

The samples were first ground in a mullite mortar to pass a No. 325 pigment sieve. Samples that were moist had to be dried to assure disaggregation. The powder was pressed into a wedge-shaped specimen holder, piling until a sharp line of the powder was obtained.

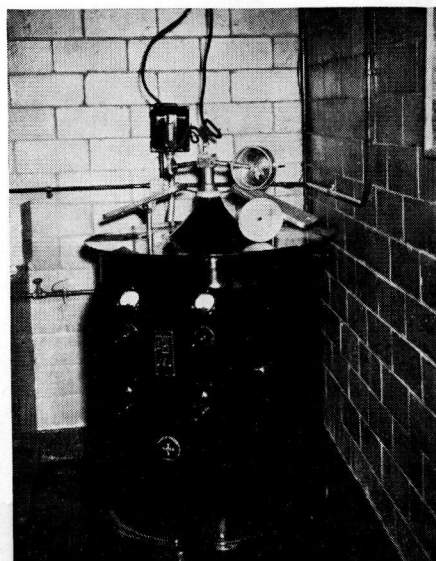


Figure 8. X-ray Spectrograph with Diffraction and Microradiographic Cameras in Position.

per radiation, and nickel foil filter. The film was processed and patterns such as those shown in Figure 9 were obtained.

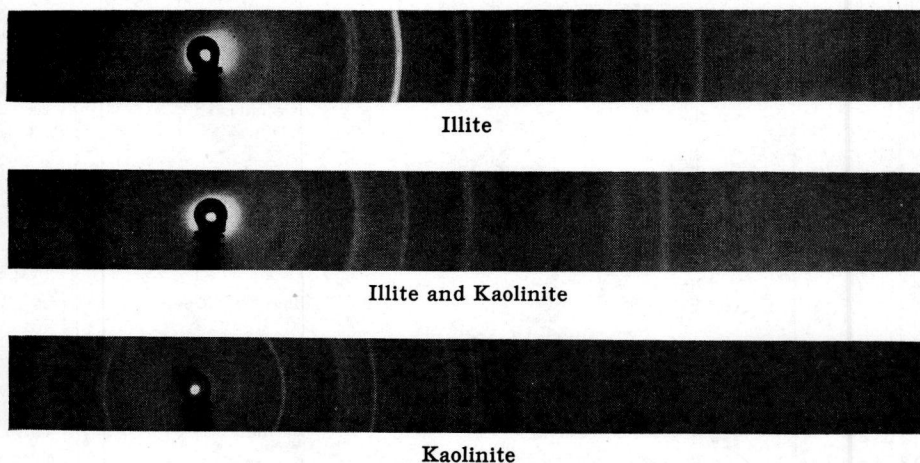


Figure 9. X-ray Diffraction Patterns

RESULTS

The purpose of this study was primarily to test the procedures and practical applications of clay mineralogy to soils research. As a matter of course some indications of trends resulted. Because of the extremely limited number of samples analysed, fundamental limitations of the physical soil tests, and the lack of control over extraneous influences such as exchangeable bases, organic matter, soluble silica, the pH of the soils, and oxides; it is impossible to arrive at any definite correlation between the clay mineral content and the routine soil tests. Nevertheless, the trends that were developed establish definite lines for future investigations and survey work.

The 22 soils which were analysed for clay mineral content are listed in Table 3. The "Relative Percent of Clay Mineral Occurrence" is given for each fraction analysed. This figure is based on an estimate of the relative intensities of the lines on the X-ray diffraction patterns. In the next column the "Relative Quantitative Distribution" appears. This value, based on the volumes of recovered fractions, was derived by assigning the lowest size fraction a value of one and relating the other sizes to it in whole numbers.

With the intent of measuring the percentage of frequency of the clay minerals with relation to the soil as a whole, a percentage value was derived using the "Relative Percent of Clay Mineral Occurrence" and the "Quantitative

Distribution (Relative)". This value called "Percent of Clay Minerals", is moderately accurate and is a basis proposed for future analysis.

In the final column of Table 3, the "Percent Colloids" is shown, based on the total soil. This value was derived from a sieve and a hydrometer analysis of each soil sample. The fact that the hydrometer analysis is not 100 percent accurate, coupled with the fact that unknown quantities of organic matter, oxides, and other extraneous materials appear in the "colloid" fraction, produces considerable magnitude of error in this value, if it is to be used as a criteria for quantity of clay minerals. As the analysis is of a limited nature, it is felt that a fair indication of the quantity of clay mineral can be derived from "Percent of Clay Minerals" and "Percent Colloids of the Total Sample".

The premise that any admixture of clay minerals will show varying percentages in different micro-fractions is substantiated by the results obtained. In general, kaolinite does not reduce easily to smaller sizes. Illite reduces more readily to smaller sizes and is usually dominant in any illite-kaolinite mixture below the 0.2 micron fraction. Montmorillonite when present might be expected to dominate the lowest fractions. The single occurrence of this mineral is not enough to show this.

Some good correlation is shown between geologic derivation and clay mineral content

TABLE 3
RESULTS OF THE CLAY MINERAL ANALYSIS

Soil Number	Fraction in Microns	Relative Percent of Clay Mineral Occurrence	Quantitative Distribution (Relative)	Percent of Clay Minerals	Percent Colloids (Total Sample)
201	> .2 .2 - .1 .1 - .05	Illite 60 Kaolinite 40 Illite 70 Kaolinite 30 Illite 80 Kaolinite 20	20 2 1	Illite 62 Kaolinite 38	20
202	> .2 .2 - .1 .1 - .05	Illite 100 Illite 100 Illite 100	4 2 1	Illite 100	28
203	> .2 .2 - .1 .1 - .05	Illite 100 Illite 100 Illite 100	25 5 1	Illite 100	5
204	> .2 .2 - .1 .1 - .05	Illite 100 Illite 100 Illite 100	6 3 1	Illite 100	25
206	> .2 .2 - .1 .1 - .05	Illite 100 Illite 100 Illite 100	6 2 1	Illite 100	19
209	> .2 < .2	Illite 10 Kaolinite 90 Illite 25 Kaolinite 75	4 1	Illite 13 Kaolinite 87	13
228	> .2 .2 - .1 .1 - .05	Illite 60 Kaolinite 40 Illite 60 Kaolinite 40 Illite 60 Kaolinite 40	6 3 1	Illite 60 Kaolinite 40	33
229	> .2 .2 - .1 < .1	Illite 50 Kaolinite 50 Illite 60 Kaolinite 40 Illite 90 Kaolinite 10	20 4 1	Illite 53 Kaolinite 47	22
232	> .2 .2 - .1 < .1	Illite 60 Kaolinite 40 Illite — Kaolinite — Illite 50 Kaolinite 50	8 4 1	Illite 59 Kaolinite 41	12
239	> .2 .2 - .1 < .1	Illite 100 Illite 100 Illite 100	8 4 1	Illite 100	34
256	> .2 .2 - .1 < .1	Illite 100 Illite 100 Illite 100	24 1 1	Illite 100	16
257	> .2 .2 - .1 .1 - .05	Illite 30 Kaolinite 20 Illite 90 Kaolinite 10 Illite 60 Kaolinite 40	15 3 1	Illite 80 Kaolinite 20	17
258	> .2 .2 - .1 < .1	Illite 75 Kaolinite 25 Illite 90 Kaolinite 10 Illite 90 Kaolinite 10	16 2 1	Illite 78 Kaolinite 22	19
260	> .2 .2 - .1 .1 - .05	Illite 100 Illite 100 Illite 100	9 1 1	Illite 100	33
264	> .2 .2 - .1 < .1	Illite 10 Kaolinite 90 Illite 50 Kaolinite 50 Illite — Kaolinite —	10 10 10	Illite 10 Kaolinite 90	26
279	> .2 .2 - .1 < .1	Illite 90 Kaolinite 10 Illite 90 Kaolinite 10 Illite — Kaolinite —	9 2 1	Illite 90 Kaolinite 10	18
281	> .2 .2 - .1 < .1	Illite 100 Illite 100 Illite 100	20 4 1	Illite 100	25
293	> .2 .2 - .1 < .1	Illite 100 Illite 100 Illite 100	12 3 1	Illite 100	34
325	> .2 < .2	Illite 33 Kaolinite 33 Montmorillonite 33	33 33 33	Illite 33½ Kaolinite 33½ Montmorillonite 33½	24
340	> .2 .2 - .1 < .1	Illite 40 Kaolinite 60 Illite 10 Kaolinite 90 Illite 75 Kaolinite 25	10 3 1	Illite 36 Kaolinite 64	18
341	> .2 .2 - .1 < .1	Illite 60 Kaolinite 40 Illite 60 Kaolinite 40 Illite 90 Kaolinite 10	12 2 1	Illite 62 Kaolinite 38	16
351	> .2 .2 - .1 < .1	Illite 100 Illite 100 Illite 100	20 4 1	Illite 100	25

in Table 4. However, it is nowhere conclusive. In the case of the soils derived from the Silurian and Devonian formations, some difficulty in correlation might be explained by the absence of any positive geological identification, due to the type of locality. These samples all came from the same vicinity. This might be cleared up by analyzing soils from similar but more widely scattered areas.

The fact that only one clay mineral occurs in a soil can very well indicate a mature soil. This has been demonstrated by the Ordovician soils in Table 4, all of which contain illite exclusively. This does not imply that geologic age has anything to do with the maturity. It merely represents a soil in which nature has established a balance.

The fact that some soils show wide variations of percentages of clay mineral constituents might be attributed to several factors. Local relief might influence this, such as in the Pottsville sandstone, a resistant layer which in some areas determines the relief. Depending on the relief, combinations of weathering products of several formations might form the resultant soil. This has produced tremendous variations in the samples classed as Pottsville. Further, general differences in climatic conditions are known to influence clay mineral formation, so that under one set of influences kaolinite might form and under another set of influences montmorillonite might form from the identical parent rock. Information of this phase is not complete enough to use, but it is thought worthy of mention. It is also reported that soil conditions of acidity or alkalinity will influence the clay mineral formed.

In the interpretation of X-ray diffraction patterns, several pertinent things were learned. Below 0.05 microns, the clay mineral tends to become a minor fraction, dominated by the organic material and silica gel. If this fraction is to be analysed, post-treatment with intent to destroy the organic matter is necessary. Samples not so treated merely produce broad bands across the film under X-radiation, obscuring the clay mineral lines.

Iron and aluminum oxides occur in every fraction of most soils. When copper X-radiation is used the iron oxide fluoresces so as to obscure the first lines on a pattern, often hopelessly confusing the pattern. Treatment to remove the iron or the use of some other wave length of radiation becomes necessary.

Fractions above 1 micron in size almost invariably contain quartz. From 1 to 5 microns, fractions are either mixtures of quartz and some clay mineral or are dominated by quartz. This limits the clay minerals to a dominate position in the 1 to 0.05 micron sizes for this group of soils.

TABLE 4
CLAY MINERAL ANALYSES GROUPED
GEOLOGICALLY

Soil Number	Geologic Formation	Clay Mineral
		%
		Ordovician
206	Trenton	Illite 100
203	Trenton	Illite 100
239	Cynthiana	Illite 100
202	Eden	Illite 100
281	Eden	Illite 100
351	Maysville	Illite 100
204	Maysville	Illite 100
		Silurian
256	Silurian	Illite 100
257	Silurian	Illite 80 Kaolinite 20
		Devonian
260	Devonian	Illite 100
264	Devonian	Illite 10 Kaolinite 90
		Mississippian
228	St. Louis	Illite 60 Kaolinite 40
325	St. Louis	Illite 33½ Kaolinite 33½ Montmorillonite 33½
		Pennsylvanian
340	Pottsville	Illite 36 Kaolinite 64
341	Pottsville	Illite 62 Kaolinite 38
		Loess
209	Loess	Illite 13 Kaolinite 87
232	Loess	Illite 59 Kaolinite 41
		Alluvium
279	Alluvium	Illite 90 Kaolinite 10 (Ohio River)
201	Alluvium	Illite 62 Kaolinite 38 (Ohio River)
203	Alluvium	Illite 100 (Kentucky River)
		Glacial
229	Glacial	Illite 53 Kaolinite 47
258	Glacial	Illite 78 Kaolinite 22

CONCLUSIONS

The limited number of samples practically excludes any specific conclusions. However, some general trends have been established from the foregoing results.

(1) The work indicates that, although different fractions show varying abundance of clay identities, fractionation is not necessary for soil survey work since no mineral becomes extinct in any fraction.

(2) There is some indication that clay mineral identities characterize geologic areas of limited regional extent.

(3) Organic matter and silica gel usually appear in the minus 0.05 micron sizes. Oxides of aluminum and iron appear in considerable abundance in all fractions and seem to be closely associated with the clay minerals and to influence their behavior. The influence of silica gel is a matter for further study.

In view of the indiscriminate variations in the properties of any clay identity, as indicated by the lack of correlation with routine soil tests, the influences of exchangeable bases and accessory materials must provide the source of the variations. In the future, attempts will be made to further define the influences of these accessory factors.

ACKNOWLEDGMENTS

The authors wish to thank Dr. Martin E. Weeks of the University of Kentucky Agricultural Experiment Station for his advice and assistance on equipment and soil preparation, Mr. William Seay for use of procedures and information developed in his M.S. thesis, Dr. Morris Scherago, Dr. O. F. Edwards and Mr. D. C. McMurtry, of the Department of Bacteriology at the University of Kentucky for their work and instruction in Electron Microscopy.

The authors also wish to express appreciation to the staff of the Laboratory for their assistance and work related to this study, and to Mr. L. E. Gregg, Associate Director of Research, for his interest and advice in the preparation of this paper.

REFERENCES

1. Hanawalt Card File, A.S.T.M. Committee E-4.
2. Endell, K., Loos, W., and Breth, H., "Relation Between Colloid Chemical and Soil Physical Characteristics of Soils and Frost Action", *Forschungs-Arbeiten a.d. Strassenwesen*, 16, 53 pp., 1939.
3. Grim, R. E., "Clay Minerals in Soils and Their Significance", Illinois State Geological Survey, Circular No. 65, Urbana, Illinois, 1941.
4. Johnson, A. L., and Davidson, D. T., "Clay Technology and Its Application to Soil Stabilization", *Proceedings, Highway Research Board*, Vol. 27, p. 418 (1947).
5. Baker, R. F., "A Study of the Relationship Between Subgrade California Bearing Ratios and Pumping of Rigid Pavements", Preliminary Report on Kentucky Project SWHP 1 (9), Kentucky Department of Highways and Public Roads Administration, February, 1948. (Unpublished)
6. Schachmam, H. K., (1939), S. B. Thesis, Massachusetts Institute of Technology.
7. Hauser and Lynn (1940), "Separation and Fractionation of Colloidal Systems", *Ind. and Engr. Chemistry*, Industrial Ed. XXXII.
8. Seay, W. A., "Physical and Chemical Properties of Fractionated Clays from Two Kentucky Soils", Unpublished Masters Thesis, University of Kentucky (1947).
9. Bragg, W. L., "Atomic Structure of Minerals", Cornell University Press (1937).