PETROGRAPHY OF TWO IOWA LOESS MATERIALS†

F. LEICESTER CUTHBERT,
Iowa State College, Ames, Iowa.

INTRODUCTION

The Iowa State Highway Commission has found from experience that certain soils, subsoils, and soil parent materials behave differently in the subgrades of roads. Some are stable as foundations, whereas others cause the road to buckle and drain poorly. The standard highway laboratory tests fail to distinguish between these materials, for their properties from the engineer's standpoint are very similar. The slight differences that do occur have not been considered diagnostic. It was with the purpose of affording some explanation of the difference in behavior of these materials that the following work was undertaken. Recent work in clay mineralogy indicates the possibility of applying knowledge of the clay-mineral constituents to practical problems of this nature (2).

The two samples selected for study were as much alike in physical properties as it was possible to obtain, but different as regards behavior in the subgrade. It was decided to investigate these two samples as completely as possible in order to determine their mineralogical character and any significant difference in their mineralogical composition. Chemical, *x*-ray, optical, and base-exchange studies were made.

TABLE 1.* PHYSICAL PROPERTIES OF SAMPLES NOS. 1316 AND 1263

	Mechanical Analyses—% Passing Sieve								
Sample Number	#4	#8	#10	#40	#60	#100	#270	.005 mm.**	.001 mm.**
1316	100	100	100	100	100	99.8	99.0	28.3	14.8
1263	100	100	100	99.0	97.0	95.0	94.0	25.5	15.1

	Shrinkage Factors		*Density Test*		*Moisture Content %*		*Material Passing #40*		
	Limit	*Ratio*	*Absolute*	*Standard*	*F.M.E.*	*C.M.E.*	*Lower Liquid Limit*	*Lower Plastic Limit*	*Plasticity Index*
1316	19.5	1.73	2.71	1.55	31.4	37.0	38.7	20.8	17.9
1263	16.9	1.84	2.69	1.66	27.7	26.72	32.2	18.3	13.9

* Analyses by Iowa State Highway Commission Laboratory at Ames, Iowa.

** Determined by Bouyoucos Method.

---

† Part of a Ph.D. thesis. Complete thesis on file at Iowa State College, Ames, Iowa.

Sample No. 1263, of the material that causes trouble in the subgrade, consists of a composite section from 12 to 15 feet deep taken from Marshall County, Iowa (SE. $\frac{1}{4}$ NW. $\frac{1}{4}$ sec. 22, T. 83 N., R. 19 W.). The material is gray loess, slightly mottled with iron oxide. It rests immediately upon Kansan till, the upper portion of which has weathered to gumbotil. The loess is Peorian in age. Sample No. 1316, of somewhat similar properties from the engineer's standpoint and which does not cause trouble in the subgrade, is of a composite section, 5 to 11 feet deep taken from Cass County, Iowa (S. $\frac{1}{4}$ sec. 20, T. 77 N., R. 37 W.). The material is brown loess, mottled with gray and with occasional iron-stained streaks. This material lies $10\frac{1}{2}$ feet above a chocolate-brown gumbotil. The age of the loess is Peorian and the gumbotil, Kansan.

EXPERIMENTAL PROCEDURE

Fractionation. A fractionation or mechanical analysis of material of this nature is essential mainly for three reasons:

1. To obtain an approximation of the size grade distribution of particles.
2. To separate the active or colloidal fraction from the relatively inert or non-colloidal fraction.
3. To facilitate microscopic study, *x*-ray analysis, chemical analysis, and base-exchange determinations by attempting to secure separates of relatively pure minerals.

The two samples were fractionated according to Bray, Grim, and Kerr (1), with certain adaptations. Every endeavor was made to treat the two exactly alike. One 1500 gram sample from each location was prepared for fractionation by leaching with dilute 1 N CH_3COOH and dilute 0.1 N HCl and washed with distilled water. The samples were then separated into two parts by sedimentation (one fraction containing all those particles greater than one micron in diameter, called the residue, and one fraction containing all those particles less than one micron, the colloidal fraction). This was effected by dividing the samples in two parts and dispersing each part in a very weak NH_4OH solution (2 cc. of conc. NH_4OH in 1500 cc. of distilled water). Each suspension was shaken manually for five minutes in $2\frac{1}{2}$ liter bottles, allowed to stand for 24 hours and the top eight centimeters then siphoned off. This represented the colloidal fraction and the remaining fraction constituted the residue. The bottles containing the residue were again filled up to the original level with the NH_4OH solution and the process repeated. This was done for a total of 63 times or until the total amount of solids being collected per week in the colloidal suspension was less than 0.3 of one per cent of the original sample weight.

The colloidal separate was then fractionated with a Sharples Supercentrifuge into three fractions: the coarse colloid, particles approximately

1 to 0.1 micron in diameter; the fine colloid, 0.1 to 0.06 microns; and the superfine colloid, all those less than 0.06 microns in diameter. These three colloidal fractions and the residue were air-dried and weighed (Table 2).

TABLE 2. RESUMÉ OF MECHANICAL ANALYSES BY SEDIMENTATION AND CENTRIFUGE

Sample	#1263		#1316	
Original weight	1500 gms.	Per cent	1500 gms.	Per cent
Residue	1071.81	71.45	1176.35	78.43
Coarse colloid	105.08	7.00	113.40	7.56
Fine colloid	37.67	2.51	46.36	3.09
Superfine	104.20	6.94	91.09	6.07
Total colloids	246.95	16.45	250.85	16.72
Total colloids and residue	1318.76	87.90	1427.20	95.15
Lost in leaching and handling	181.24	12.08	72.80	4.85

Chemical analysis. Chemical analyses were made of the original samples before the dispersion treatment, and of each of the four fractionated separates (the residue, the coarse colloid, the fine colloid, and the superfine colloid). Standardized procedures were used throughout (Table 3).

Microscopic study. The determination of optical constants of each of the colloidal fractions affords an additional means of supplementing the other data. In addition, separates of the residue ranging in particle size from one to three microns, three to five microns, five to ten microns, and all those particles greater than ten microns were made and studied microscopically. Due to the fact that the clay minerals are micaceous in habit and tend to orient themselves in aggregates, if allowed to settle slowly out of suspension, it is possible to determine several optical constants (4). Optical constants determined were indices of refraction, sign, and 2V. Well oriented aggregates gave good interference figures and permitted the rather accurate determination of the least (α) and greatest (γ) indices of refraction.

X-ray analysis. X-ray powder diffraction patterns were made of all of the colloidal separates of each of the samples. The *x*-ray laboratory of the University of Illinois was made available to the author by Dr. G. L. Clark of the Department of Chemistry and the work was done under the supervision of Dr. W. F. Bradley of the Illinois State Geological Survey.

The equipment used consisted of a Philips Metalix tube with FeKα radiation and what is commonly known as a circular camera. Exposures

Table 3. Chemical Data*

	1263					1316				
	Bulk	Residue	Coarse	Fine	Super-fine	Bulk	Residue	Coarse	Fine	Super-fine
SiO_2	68.69	81.93	54.12	48.89	49.36	69.92	76.15	54.37	46.66	46.62
Al_2O_3	9.93	8.79	21.31	22.26	20.09	11.88	11.33	18.66	19.22	15.51
Fe_2O_3	2.94	1.93	7.25	8.77	9.80	4.45	3.04	8.95	10.83	10.80
FeO	0.40	trace	trace	trace	trace	0.44	trace	trace	trace	trace
MgO	2.34	0.70	2.47	2.72	2.69	1.40	1.02	2.01	2.55	2.49
CaO	4.17	0.96	1.32	1.91	1.82	1.13	0.74	0.57	0.45	0.46
Na_2O	2.86	2.23	1.77	1.18	1.64	1.33	2.52	1.80	1.71	1.18
K_2O	1.63	1.87	1.87	1.47	0.86	1.88	2.45	2.53	1.75	1.06
TiO_2	0.62	0.67	1.13	0.75	0.41	0.66	0.65	0.97	0.66	0.31
P_2O_5	0.10	0.04	0.14	0.09	0.36	0.21	0.04	0.17	0.17	1.53
CO_2	4.01	—	—	—	—	0.22	—	—	—	—
MnO	0.03	0.01	0.09	0.03	0.02	0.09	0.11	0.17	0.07	0.03
H_2O+	2.40	1.89	8.24	8.44	10.35	2.84	3.12	7.23	8.66	9.14
H_2O-	1.68	—	2.99	6.19	3.91	2.70	—	4.36	5.29	7.99
										3.43**
	101.40	101.02	102.70	102.70	101.31	98.71	101.17	101.79	101.02	100.55
SiO_2/R_2O_3	9.88	13.87	3.54	2.98	3.18	8.08	9.74	3.78	3.24	3.54

* Analyses by Gurney and King of Iowa State College.

** Oil from centrifuge.

varied from six to ten hours. Two of the fractions, the fine colloid of sample 1316, and the superfine colloid of sample 1263 were treated with β-naphthylamine (3) to show the presence of the montmorillonite type of clay mineral when, because of the haze at the significant end of the film, it was impossible to tell whether or not this group was present. The large organic molecules spread the lattice of the montmorillonite and consequently so changed the position of its most characteristic diffraction line as to make it easily recognizable if present. Results were computed and tabulated as *d* values (Table 4).

TABLE 4. *X*-RAY DATA*

	1316 C	1316 F	1316 S.F.	1263 C	1263 F	1263 S.F.	*d* *values* Å	*Mineral*
1	M	?	S			?	13.64	Montmorillonite
2	W	W	W	M	M	M	9.85	Illite
3	W	W		M	W	?	7.25	Kaolinite, Illite
4	W.W.	W	W	W.W.	W	W	4.94	β quartz
5	S	S	S	S	S	S	4.49	Clay**
6	S	W.W.		S	W.W.		4.27	Quartz
7		W		M	W		3.55	Kaolinite, Quartz
8	S.S.	S		S.S.	S		3.32	Illite
9	M			M			3.00	Calcite
10	S	S	S	S	S	S	2.59	Clay**
11	S			M			2.45	Quartz
12	M			M			2.28	Quartz
13	W			W			2.24	Quartz
14	S			S			2.12	Quartz
15	M	W.W.		M	W.W.		1.98	Illite, Quartz
16	S			S			1.81	Quartz
17	W	W	W	W	W	W	1.66	Clay**, Quartz
18	S			S			1.53	Quartz
19	S	S	S	S	S	S	1.49	Clay**
20	S			S			1.37	Quartz

* Observed intensities

S.S. = Very strong
S = Strong
M = Medium
W = Weak
W.W. = Very weak

** Not diagnostic of any particular clay mineral.

Base-exchange determinations. Duplicate samples were prepared of each of the four fractions by powdering the material to allow complete and immediate reaction. Care was taken not to powder so vigorously as to destroy the original particle size. Three gram samples of the residue fraction were used, two grams of the coarse colloid, and one gram each of the fine and the superfine colloids. The samples were centrifuged five times with 1 N $Ca(C_2H_3O_2)_2$, washed five times with 70 per cent alcohol and again centrifuged five times with neutral 1 N $NH_4C_2H_3O_2$. The supernatant liquid from the last treatment was saved and Ca determined volumetrically (Table 5).

Table 5. Total Base-Exchange Capacity Expressed as Milliequivalents per 100 Grams of Colloid

	Residue	*Coarse Colloid*	*Fine Colloid*	*Superfine Colloid*
Sample 1263	8.70	48.30	69.22	79.05
Sample 1316	13.20	45.31	65.98	95.24

Experimental Results

Sample No. 1263

Residue. Samples representing different particle sizes were separated from this fraction by sedimentation. The coarsest of these, with particles larger than ten microns, contained 95 per cent angular quartz, a few rock fragments, some limonite and muscovite. The fraction containing particles between five and ten microns in size contained about 75 per cent quartz. The rest was mainly limonite and muscovite. Between three and five microns, the amount of quartz decreased to about 60 per cent and the amount of mica increased. A few fragments of kaolinite were also found in this particle range. The fraction containing particles ranging between one and three microns contained the same amount of quartz and mica as the preceding one, and a slightly greater amount of kaolinite. The small base-exchange capacity of this total residue fraction indicates a high degree of separation by the sedimentation process and consequently a general lack of clay minerals.

Coarse colloid fraction. Optical examination of this fraction revealed aggregates whose indices of refraction ranged from 1.569 to 1.574. No aggregates could be obtained with an orientation sufficiently perfect to yield additional information about the optical properties. The *x*-ray diffraction pattern of this fraction showed moderately strong lines of illite (5), kaolinite, calcite, and strong lines of quartz. There was a decided increase in the base-exchange capacity indicating a concentration of the clay minerals. The analyses suggest that this fraction is composed largely of quartz, kaolinite, illite, and calcite.

Fine colloid fraction. The indices of the aggregates ranged from 1.563 to 1.594. Aggregates of almost perfect orientation and hence showing good interference figures were common. The mineral of the aggregates was biaxial, negative, and 2V was about 5 degrees. Indices of refraction for these aggregates were: $\alpha=1.579$, $\gamma=1.584$. The x-ray diffraction pattern revealed moderately strong lines of illite, and weak lines of kaolinite and quartz. Chemical analysis showed less SiO_2 and more Al_2O_3 than the coarse colloid fraction. This is attributed to a smaller amount of quartz. The combined data show that this fraction is composed of illite with small amounts of kaolinite and quartz.

Superfine colloid fraction. A large number of well oriented aggregates were secured (4) whose indices of refraction ranged from 1.554 to 1.574. The best oriented aggregates were biaxial, negative, and $2V=5$ degrees, $\alpha=1.554$, $\gamma=1.573$. The x-ray diffraction pattern showed moderately strong lines of illite, and questionable lines of kaolinite and montmorillonite. An x-ray diffraction pattern of powder that had been immersed in β-napthylamine gave an identical pattern except for a moderately strong line of montmorillonite so moved in position that it could be identified positively. The silica-sesquioxide ratio at 3.18 and the base-exchange capacity at 79.05 were greater than with the fine colloid fraction. This also suggests the presence of montmorillonite. The complete data indicate that this fraction is composed largely of illite and montmorillonite, and that kaolinite is also probably present.

Sample 1316

Residue. About 75 per cent of the particles of the coarsest residue separate, that consisting of particles greater than ten microns in diameter, were quartz. The others were mainly limonitic material. Small amounts of muscovite and magnetite were also present. The other residue separates, those containing particles from five to ten, three to five, and one to three microns in size respectively, were not significantly different except that they had progressively less quartz and more mica, kaolinite, and limonitic material. The iron content of this sample was also higher than that of sample 1263 (Table 3). The base-exchange capacity was quite small, indicating a fairly good separation of the colloidal and non-colloidal materials. This was also true of the residue fraction of sample 1263.

Coarse colloid fraction. The x-ray diffraction pattern of this fraction showed moderately strong lines of montmorillonite and calcite, strong lines of quartz, and weak lines of illite and kaolinite. Indices of refraction ranged from 1.530 to 1.580. Further optical data could not be obtained because of the imperfect orientation. The evidence, chiefly x-ray, indicates that this fraction is composed essentially of montmorillonite, quartz, and calcite.

Fine colloid fraction. Optical examination showed indices of refraction varying from 1.540 to 1.573 and the following values were obtained from many aggregates having good interference figures; biaxial, negative sign, 2V about 10 degrees, $\alpha=1.542$, $\gamma=1.569$. The powder x-ray diffraction pattern revealed a questionable line of montmorillonite, and weak lines of illite, kaolinite, and quartz. The x-ray pattern secured after treatment of the material with β-naphthylamine revealed the unmistakable presence of montmorillonite. The combined data indicate that this fraction is essentially a mixture of montmorillonite, illite, kaolinite, and quartz.

Superfine colloid fraction. The x-ray diffraction pattern of this fraction showed a strong line of montmorillonite and a weak line of illite. The indices of refraction varied from 1.554 to 1.579. The better oriented aggregates gave biaxial interference figures, negative sign, 2V varying from 10 to 20 degrees, $\alpha=1.559$, $\gamma=1.578$. The base-exchange capacity of this fraction was 95.24 milliequivalents per 100 grams, the highest value obtained, and one that can only be accounted for by a large amount of montmorillonite. The data indicate that this fraction is composed chiefly of montmorillonite with a small amount of illite.

Discussion and Summary

The physical properties of the two materials under study, as determined by the highway laboratory through the use of standardized tests, are so similar that it is impossible to differentiate between them (Table 1). Fractionation of the material by sedimentation and a supercentrifuge into several different size separates also fails to reveal any significant difference in amounts of the several grade sizes (Table 2). Accordingly, it is impossible to forecast the behavior of these materials by a comparison of their colloidal content, although it is known that in some circumstances, variations in the amount of colloidal material can be regarded as being diagnostic.

The analytical results show that the samples differ in the nature of their clay mineral content, 1316 being made up essentially of montmorillonite, whereas sample 1263 contains illite and kaolinite as its principal clay mineral constituents. The differences in the characteristic behavior of these clay minerals, montmorillonite, illite, and kaolinite would lead one to suspect that the material containing montmorillonite in larger amounts would behave poorly in the subgrades of roads. However, in this situation the reverse is true and the montmorillonite-bearing material forms the better subgrade. Another condition must be taken into consideration. Sample 1263 was taken from a horizon immediately above a relatively impervious gumbotil, while sample 1316 was taken from material $10\frac{1}{2}$ feet above a similar gumbotil. This relatively impervious layer creates a temporary, perched water table, which in the former case can be expected to interfere with the drainage of the material immediately beneath the slab. In the case of sample 1316 the drainage of the material immediately beneath the slab is not influenced by the gumbotil. Because of this difference in the effect of the gumbotils upon the drainage of the two materials, any variation in the behavior of the materials due to a difference in clay mineral content is felt to be obscured.

A considerable difference in the clay mineral content of these two subgrade materials has been shown, and it is concluded that highway engineers should take into consideration the possibility of differences of behavior being due to such mineral differences, particularly in the colloid fractions, and the geological positions of the materials they plan to use in the subgrades of roads. Differences in the nature of the clay mineral content of materials can be expected to affect their behavior in the subgrade and these differences are apparently not indicated by the standard laboratory tests. The plasticity tests (Table 1) are slightly higher for the montmorillonite material, as would be expected, and a study of more materials will perhaps reveal that this difference should be considered significant.

The great difference in the base-exchange capacity between the colloidal and the non-colloidal fractions of both samples is striking (Table 5). The possibility of using this as a criterion of the perfection of separation between the colloidal and the non-colloidal fractions is suggested.

ACKNOWLEDGMENTS

Deep appreciation is expressed to those who by their assistance and cooperation have made this work possible. They are: Professor J. T. Lonsdale, Dr. L. M. Cline, and Dr. C. S. Gwynne of Iowa State College, Dr. R. E. Grim and Dr. W. F. Bradley of the Illinois State Geological Survey, and Mr. L. W. Wood of the Iowa State Highway Commission.

REFERENCES

1. Bray, R. H., Grim, R. E., and Kerr, P. F., *Bull. Geol. Soc. Am.*, **46**, 1909–1926 (1935).
2. Ekblaw, G. E., and Grim, R. E., *Ill. State Geol. Surv., Rept. of Investigations*, **42**, 11–12, (1936).
3. Gieseking, J. E., *Soil Sci.*, **47**, No. 1 (1939).
4. Grim, R. E., *Jour. Sed. Pet.*, **4**, No. 1, 45 (1934).
5. Grim, R. E., Bray, R. H., and Bradley, W. F., *Am. Mineral.*, **22**, No. 7, 816 (1937.)