Petrography of two Iowa loess materials

Frederick Leicester Cuthbert

Iowa State College

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PETROGRAPHY OF TWO IOWA LOESS MATERIALS

by

F. Leicester Cuthbert

A Thesis Submitted to the Graduate Faculty for the Degree of

DOCTOR OF PHILOSOPHY

Major Subject Geology and Soils

Approved:

Signature was redacted for privacy.

In charge of Major work

Signature was redacted for privacy.

Head of Major Department

Signature was redacted for privacy.

Dean of Graduate College

Iowa State College
1940
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ABSTRACT

Two samples of loess material, selected by the Iowa State Highway Commission as being significant in highway construction, were investigated by several methods with the purpose of determining their mineralogical constitution. Although the materials are nearly similar as far as standardized highway laboratory tests are concerned, one affords a stable highway foundation while the other gives considerable difficulty, causing the slab to buckle and dip. Field examination revealed that the materials differed in their relation to a heavy gumbotil; one being located immediately above, and the other about ten feet above the gumbotil. The samples were fractionated by sedimentation and by a supercentrifuge. X-ray, optical, and chemical studies including base-exchange determinations were made on each of the colloidal separates obtained. The results show that one of the samples contains as its principal clay mineral, montmorillonite, while the other contains mixtures of kaolinite and illite. Evidence points to the conclusion that highway engineers must take into consideration both the geological positions of the materials to be used in subgrades and their clay mineral content.
INTRODUCTION

Highway engineers have 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 discovering 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 (5).

Two samples, which were as much alike in physical properties as it was possible to obtain but different as regards behavior in the subgrade, were selected for study by Mr. L. W. Wood of the Iowa State Highway Commission. 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 mineral-
ogical composition. X-ray, optical, and chemical studies including base-exchange determinations were made.*

Sample 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 1/4 NW 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. The soil of this area is the Tama Silt loam. Sample 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 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 1/2 feet above a chocolate-brown gumbotil. The age of the loess is Peorian and the gumbotil, Kansan. The soil of this area, although the county is not mapped, very likely is the Marshall Silt loam.

---

* The initial project was cooperative between the Departments of Geology and Chemistry and supported by the Research Council. The chemical analyses were made by workers under the direction of Dr. J. A. Wilkinson, and the remaining work was under the supervision of Dr. J. T. Lonsdale.
REVIEW OF LITERATURE

The literature referring to clay minerals, methods applicable to their determination, and of their influence on the behavior of materials is of a diversified nature. Articles have been found in geological journals, soil journals, ceramic, and chemistry journals. Many of these have no particularly close application to the problem at hand and consequently will not be mentioned in this review. For the benefit of any reader interested in clay mineralogy and in its literature which is not reviewed here, the reader is referred to the work of R. E. Grim (10) who has recently published the most complete survey of literature and bibliography to date.

Up to about the last ten years mineralogists recognized one common clay mineral, kaolin or kaolinite. The last edition of Dana's Manual of Mineralogy, a widely used standardized text, lists this one clay mineral. Within the last ten years our knowledge of the minerals composing soils, clays, and loess materials has increased many fold. Thus, clay mineralogy is young and has all the promise of youth. The present data are by no means complete and in the future still further developments can be expected.

The development of new techniques of study and their adaptation to investigations concerning clay mineralogy has
been the greatest single factor in the growth of this science. In this country it is common practice to use as many methods as possible to determine the nature of the clay mineral constituents of any material. These methods, at present, include fractionation, chemical analysis, microscopic determinations, x-ray powder diffraction analysis, base-exchange determinations, and dehydration curves. In Germany many determinations are made on the basis of x-ray analysis alone (11). Largely as a result of the improved methods of study we now recognize three distinct groups of clay minerals: the kaolin group, the montmorillonite-beidellite group, and the latest addition, the illite group (13).

Ross and Kerr have determined and summarized the properties of the kaolin group of clay minerals in two papers. The first of these is on kaolinite, dickite, and nacrite (21), the second on halloysite and allophane (22).

The association of the montmorillonite-beidellite group of clay minerals in bentonite, a rock of wide commercial use, has led to a natural interest in this group. Ross and Shannon (23) define bentonite as "a rock composed essentially of a clay-like mineral formed by devitrification and the accompanying alteration of a glassy igneous material, usually a tuff or volcanic ash; and it often contains variable proportions of accessory crystal grains that were originally
Montmorillonite, another member of the montmorillonite group.

The two (16) of these, the result of the difference in hydration between
Wark's montmorillonite, it exhibits distinctly different properties.

It appears to be formed under somewhat the same circumstances as mont-
the opal and chalcedon data. While this material is thought
calergos, belite, and fayalite as determined
Tarsen and Reilly (17) named a new mineral from Belding,
rocks and sphenocryst (24) determined
almost unreasonably acceptable.

true of montmorillonite and the work of Hofmann is now
(17) and Kelley (16) have published on the crystal stix- 
both selenites. N. Hofmann and co-workers (17) Marshall
base-exchange capacity has interested both mineralogists and
the crystalline lattice structure of the mineral and to its
phenomenon of montmorillonite in water and its relation to
penetration of such a permeable value (2). The swelling
these minerals, particularly montmorillonite, that makes
ofteren belite. It is the peculiar physical behavior of
and if it is usually the mineral montmorillonite, but less
and usually a texture inherited from volcanic tuffs or ash.
and usually a texture inherited from volcanic tuffs or ash.
atmosphere. The charactertistic clay-like material has
(17) are typical of
common orthoclase and orthoclase quartz.
pronounced in the volcanic glasses. These are referred
was originally described by Weinshenck (25) as the ferric equivalent of kaolinite. Subsequent investigations by Larsen (17) and Gruner (14) have shown that montmorillonite is closely related, structurally, to montmorillonite and belongs to this group of clay minerals rather than to the kaolin group.

Saponite, the remaining member of this group, was first clearly described by Palache (20) in connection with a study of the Keweenawan copper deposits. X-ray studies of this mineral revealed a pattern indicative of a material isomorphous with montmorillonite. Foshag and Woodford (6) also showed the isomorphous relation of this high magnesium content clay.

The last group of clay minerals that is recognized at the present time is the illite group (13). Numerous workers have reported the occurrence of a mica-like clay mineral. Hydromica, glimmerite, and sericite-like (1) are names that have been given to this material. In 1937 Grim and his co-workers proposed the name, illite, for this material, specifically as a group name and with the understanding that as data were accumulated by various men it might be subdivided into more than one mineral. As further evidence, Grim and Bradley (12) described a clay from the Goose Lake area in Illinois which apparently belongs to this group yet whose properties differ somewhat from those ordinarily
associated with the so-called mica-like clay minerals. By using the term, illite, further confusion in the nomenclature will be avoided and at the same time it will afford a common name for the mica-like clay minerals.

The application of clay mineralogy to problems of the type dealt with in this paper was first suggested by the work of Ekblaw and Grim (5). The relationship of the clay mineral constitution of various materials and their physical properties is made evident and it seems apparent that the knowledge accumulated about clay minerals and their identification is sufficient to warrant investigations of a practical nature.
EXPERIMENTAL

Method of Procedure

Determination of physical properties

The research project, as set up, was in conjunction with the Iowa State Highway Commission with the cooperation of Mr. L. W. Wood. Mr. Wood chose the two samples to be studied after making a careful comparison of the physical properties of many whose behavior in the subgrade varied. Two samples were finally chosen and the standardized highway laboratory tests showed them to be quite similar. These tests include mechanical analysis by sieving and by the Bouyoucos method (3) and the determination of shrinkage factors, density, moisture content, and plasticity.

Fractionation

A fractionation or mechanical analysis of material of this nature is essential for a study with the purpose of ascertaining the mineralogical composition. The fractionation has three main objectives:

1. To obtain an idea of the size grade distribution of particles particularly in the colloidal (less than one micron) ranges.
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 pure minerals.

The latter point is brought about by the fact that certain of the clay minerals tend to be more or less concentrated in the various size fractions (4).

The two samples were fractionated according to Bray, Grim and Kerr (4), 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 300 gram portions with 1000 cc. of 1 N CH₃COOH and 1000 cc. of 0.1 N HCl, then washing with 1000 cc. of 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 NH₄OH solution (2 cc. of conc. NH₄OH in 1500 cc. of distilled water). Each suspension was shaken manually for five minutes in 2½ 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 NH₄OH 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. Aliquot samples of 100 c.c. were used for tests.

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.

Chemical analysis

Chemical analyses were made of the original samples before the dispersion treatment and fractionation 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. This work was done by Gurney and King under the direction of Dr. J. A. Wilkinson of the Department of Chemistry, Iowa State College.
**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 (9). 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. A set of index liquids was made up and used according to Jewell Class (8), varying in steps of 0.005.

**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 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 B-Naphthylamine (7) 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 change the position of its most characteristic diffraction line as to make it easily recognizable if present. Results were computed and tabulated as d values from the following relations:

\[ D = \text{distance in centimeters from zero point to middle of diffraction line} \]

\[ r = \text{radius of camera, 6.34 cm.} \]

\[ \lambda = \text{wave length, 1.935 Å} \]

\[ \frac{D}{r} = 2 \theta \text{ in radians} \]

\[ \lambda = 2d \sin \theta \]

\[ d = \frac{\lambda}{2 \sin \theta} \]

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\((\text{C}_2\text{H}_3\text{O}_2)_2\) was washed five times with 70 per cent alcohol, and again centrifuged five times with neutral 1 N NH\(_4\)C\(_2\)H\(_3\)O\(_2\). Fifty cc. of each reagent were used for each treatment. All of the supernatant liquid from each of the last five treatments was saved and calcium determined volumetrically.

Results

**Determination of physical properties**

The physical properties, determined by the highway laboratory, reveal very little difference between the two samples. Table I gives the results of these tests. The most significant variations are the moisture content and the plasticity. In both, it is sample number 1316 which is high. This will be discussed later.
Table I.*--Physical Properties of Samples 1316 and 1263

<table>
<thead>
<tr>
<th>Sample: Number:</th>
<th>#4</th>
<th>#8</th>
<th>#10</th>
<th>#40</th>
<th>#60</th>
<th>#100</th>
<th>#270</th>
<th>.005 mm.*</th>
<th>.001 mm.*</th>
</tr>
</thead>
<tbody>
<tr>
<td>1316</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>99.8</td>
<td>99.0</td>
<td>28.3</td>
<td>14.8</td>
<td></td>
</tr>
<tr>
<td>1263</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>99.0</td>
<td>97.0</td>
<td>95.0</td>
<td>94.0</td>
<td>25.5</td>
<td>15.1</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Shrinkage Factors</th>
<th>Density Test</th>
<th>Moisture Content %</th>
<th>Material Passing #40</th>
</tr>
</thead>
<tbody>
<tr>
<td>1316</td>
<td>19.5</td>
<td>1.73</td>
<td>2.71</td>
</tr>
<tr>
<td>1263</td>
<td>16.9</td>
<td>1.84</td>
<td>2.69</td>
</tr>
</tbody>
</table>

* Analyses by Iowa State Highway Commission Laboratory at Ames, Iowa.
** Determined by Bouyoucos Method.
Fractionation

Table II gives the results of the fractionation or mechanical analyses by both sedimentation and centrifugal methods. The weights given are all on an air-dry basis.

The similarity between the two samples is striking. There has been an increase in the total colloidal content of both samples over that determined by the highway laboratory. Sample 1263 shows a greater loss labeled due to either leaching or handling. As the amount lost in handling was the same in both instances and comparatively small, the greater amount lost by sample 1263 is caused largely by other factors.

Chemical analysis

The chemical analyses of the bulk samples and of each of the four fractionated separates are presented in Table III. Significant points brought out by the chemical analyses are:

1. The increase in the amount of silica in the residue fraction over the bulk sample.
2. The gradual decrease in silica as the particle size diminishes.
3. The changes in the amounts of iron and aluminum and their relation to the amount of silica as shown by the relative amounts of each and by the silica-
### Table II. -- Resume of Mechanical Analyses by Sedimentation and Centrifuge

<table>
<thead>
<tr>
<th>Sample</th>
<th>#1263</th>
<th>#1316</th>
</tr>
</thead>
<tbody>
<tr>
<td>Original weight: 1500 gms.: Per cent: 1500 gms.: Per cent</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Residue</td>
<td>1071.81</td>
<td>71.45</td>
</tr>
<tr>
<td>Coarse colloid</td>
<td>105.08</td>
<td>7.00</td>
</tr>
<tr>
<td>Fine colloid</td>
<td>37.67</td>
<td>2.51</td>
</tr>
<tr>
<td>Superfine</td>
<td>104.20</td>
<td>6.94</td>
</tr>
<tr>
<td>Total colloids</td>
<td>246.95</td>
<td>16.45</td>
</tr>
<tr>
<td>Total colloids and residue</td>
<td>1318.76</td>
<td>87.90</td>
</tr>
<tr>
<td>Lost in leaching and handling</td>
<td>181.24</td>
<td>12.08</td>
</tr>
</tbody>
</table>
Table III.--Chemical Data*

<table>
<thead>
<tr>
<th></th>
<th>1263 Marshall County</th>
<th>1316 Cass County</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>68.69 81.93 54.12 46.89 49.36 69.92 76.15 54.37 48.66 45.62</td>
<td></td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>9.95   8.79 21.31 22.26 20.09 11.88 11.33 18.36 19.22 15.51</td>
<td></td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>2.94   1.93 7.25 8.77 9.60 4.45 5.04 5.25 10.65 10.60</td>
<td></td>
</tr>
<tr>
<td>FeO</td>
<td>0.40   trace  trace  trace  trace  0.44  trace  trace  trace  trace</td>
<td></td>
</tr>
<tr>
<td>MgO</td>
<td>2.34   0.70 2.47 2.72 2.69 1.40 1.02 2.01 2.55 2.49</td>
<td></td>
</tr>
<tr>
<td>CaO</td>
<td>4.17   0.96 1.32 1.91 1.82 1.13 0.74 0.57 0.45 0.46</td>
<td></td>
</tr>
<tr>
<td>Na₂O</td>
<td>2.86   2.23 1.77 1.18 1.64 1.33 2.52 1.60 1.71 1.18</td>
<td></td>
</tr>
<tr>
<td>K₂O</td>
<td>1.63   1.87 1.87 1.47 0.86 1.88 2.45 2.55 1.75 1.06</td>
<td></td>
</tr>
<tr>
<td>TiO₂</td>
<td>0.82   0.67 1.13 0.75 0.41 0.66 0.65 0.97 0.66 0.31</td>
<td></td>
</tr>
<tr>
<td>P₂O₅</td>
<td>0.10   0.04 0.14 0.09 0.36 0.21 0.04 0.17 0.17 1.53</td>
<td></td>
</tr>
<tr>
<td>CO₂</td>
<td>4.01   trace  trace  trace  trace  0.22  trace  trace  trace  trace</td>
<td></td>
</tr>
<tr>
<td>MnO</td>
<td>0.03   0.01 0.09 0.03 0.02 0.09 0.11 0.17 0.07 0.03</td>
<td></td>
</tr>
<tr>
<td>CaO₄</td>
<td>2.40   1.69 3.24 3.44 10.35 2.84 3.12 7.23 8.66 9.14</td>
<td></td>
</tr>
<tr>
<td>H₂O</td>
<td>1.68   trace  trace  trace  trace  2.99 6.19 3.91 2.70 trace 7.99 3.43**</td>
<td></td>
</tr>
</tbody>
</table>

|        | 101.40 101.02 102.70 102.70 101.31 98.71 101.17 101.79 101.02 100.55 |
|--------|----------------------|------------------|
| SiO₂/R₂O₅ | 9.38 15.67 3.54 2.98 5.18 8.08 9.74 3.78 3.24 3.54 |

* Analyses by Gurney and King of Iowa State College.
** Oil from centrifuge.
sesquioxide ratio. It is slightly higher in the case of sample 1316.

4. There is a greater percentage of calcium carbonate in sample 1263. This is shown by the amounts of calcium oxide and carbon dioxide.

5. The titanium oxide tends to be concentrated in the coarse colloid fraction in both samples. This cannot be accounted for at the present time.

X-ray analysis

The x-ray patterns obtained are shown in Plate I and Plate II. Many of the diffraction lines are quite faint and consequently do not show up well in reproduction. The position and presence of lines characteristic for any mineral are marked. Table IV gives the calculated d values in Ångström units and the observed intensity. Those lines marked as clay are not to be considered diagnostic of any particular clay mineral.

Line number one in the fine colloid fraction of sample 1316 and the superfine colloid of sample 1263 are marked questionable. This is due to the haze at that end of the film. These two samples, as mentioned in the procedure, were immersed in a solution of B-Naphthylamine and again exposed. In both cases the unquestionable presence of montmorillonite was noted.
Table IV.--X-ray Data*

<table>
<thead>
<tr>
<th></th>
<th>1316 c</th>
<th>1316 S.F.</th>
<th>1316 c</th>
<th>1316 P</th>
<th>226 P</th>
<th>236 P</th>
<th>d values</th>
<th>A</th>
<th>Mineral</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>M</td>
<td>?</td>
<td>S</td>
<td>?</td>
<td></td>
<td></td>
<td>13.64</td>
<td></td>
<td>Montmorillonite</td>
</tr>
<tr>
<td>2</td>
<td>W</td>
<td>W</td>
<td>W</td>
<td>M</td>
<td>M</td>
<td>M</td>
<td>9.85</td>
<td></td>
<td>Illite</td>
</tr>
<tr>
<td>3</td>
<td>W</td>
<td>W</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>7.25</td>
<td></td>
<td>Kaolinite</td>
</tr>
<tr>
<td>4</td>
<td>W.W.</td>
<td>W</td>
<td>W</td>
<td>W.W.</td>
<td>W</td>
<td>W</td>
<td>4.94</td>
<td></td>
<td>Illite, B. quartz</td>
</tr>
<tr>
<td>5</td>
<td>S</td>
<td>S</td>
<td>S</td>
<td>S</td>
<td>S</td>
<td>S</td>
<td>4.49</td>
<td></td>
<td>Clay**</td>
</tr>
<tr>
<td>6</td>
<td>S</td>
<td>W.W.</td>
<td>S</td>
<td>W.W.</td>
<td></td>
<td></td>
<td>4.27</td>
<td></td>
<td>Quartz</td>
</tr>
<tr>
<td>7</td>
<td>W</td>
<td></td>
<td>M</td>
<td>W</td>
<td></td>
<td></td>
<td>5.55</td>
<td></td>
<td>Kaolinite</td>
</tr>
<tr>
<td>8</td>
<td>S,S.</td>
<td>S</td>
<td>S,S.</td>
<td>S</td>
<td></td>
<td></td>
<td>3.32</td>
<td></td>
<td>Quartz, Illite</td>
</tr>
<tr>
<td>9</td>
<td>M</td>
<td></td>
<td>M</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Calcite</td>
</tr>
<tr>
<td>10</td>
<td>S</td>
<td>S</td>
<td>S</td>
<td>S</td>
<td>S</td>
<td>S</td>
<td>2.59</td>
<td></td>
<td>Clay**</td>
</tr>
<tr>
<td>11</td>
<td>S</td>
<td></td>
<td>M</td>
<td></td>
<td></td>
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* Observed intensities
  S.S. = Very strong
  S  = Strong
  M  = Medium
  W  = Weak
  W.W. = Very weak

** Not diagnostic of any particular clay mineral.
Explanation of Plate I

Each of the diffraction lines used to indicate the presence of a mineral is indicated and numbered. These numbers correspond to those of the calculated data in Table IV.

The last two reproductions represent x-ray diffraction patterns of the same fraction, the superfine, and are identical with the exception of line one. This line, that shows in the last pattern, is due to montmorillonite and has been brought out by the Β-Napthylamine treatment described in the body of the thesis.
I. Coarse Colloid Fraction

II. Fine Colloid Fraction

III. Superfine Colloid Fraction

IV. Superfine Colloid Fraction with B-Napthylamine

Plate I. X-ray Diffraction Patterns of Sample 1263
Explanation of Plate II

Each of the diffraction lines used to indicate the presence of a mineral is indicated and numbered. These numbers correspond to those of the calculated data in Table IV.

The second and last reproductions represent x-ray diffraction patterns of the same fraction, the fine colloid, and are identical with the exception of line one. This line, that shows in the last pattern, is due to montmorillonite and has been brought out by the B-Naphthylamine treatment described in the body of the thesis.
I. Coarse Colloid Fraction

II. Fine Colloid Fraction

III. Superfine Colloid Fraction

IV. Fine Colloid Fraction with B-Napthylamine

Plate II. X-ray Diffraction Patterns of Sample 1316
The results show a definite tendency for the montmorillonite type of clay mineral to be predominant in sample 1316 and for illite and kaolinite in sample 1263. Quartz persisted through to the fine colloid fraction in diminishing amounts but not to the superfine fraction.

Base-exchange determinations

Table V contains the results of the base-exchange determinations. The great difference in the base-exchange capacity between the colloidal and non-colloidal fractions is striking. The superfine colloid of sample 1316 has a remarkably high value which can be accounted for only by a predominance of montmorillonite.

| Table V.--Total Base-Exchange Capacity Expressed as Milliequivalents per 100 Grams of Colloid |
|--------------------------------------------------|---------------------------------|-----------------|-----------------|-----------------|-----------------|
| :                                                | Coarse : Fine : Superfine       |
| : Residuum : Colloid : Colloid : Colloid         |
| Sample 1263                                      | 8.70 : 46.30 : 69.22 : 79.05 |
| Sample 1316                                      | 13.20 : 45.31 : 65.98 : 95.24 |

In order to completely understand the above results and their significance as regards the mineralogical composition of the two samples, the following presentation of results is given from the standpoint of the various frac-
tions of each of the samples. The optical data are also presented in these results.

Sample 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, 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 those 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 \( \text{SiO}_2 \) and more \( \text{Al}_2\text{O}_3 \) 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 was secured whose indices of refraction ranged from 1.554 to 1.574. The best oriented aggregates were biaxial, negative, 2V = five degrees, \( \alpha = 1.554, \gamma = 1.573 \). The x-ray diffraction pattern showed moderately strong lines of illite and questionable lines of kaolinite and montmoril-
ionite. An x-ray diffraction pattern of powder that had been immersed in \( \text{B-Naphthylamine} \) 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 1363, Table III. 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 mont-
morillonite 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, α = 1.542, γ = 1.569. The powder x-ray
diffraction pattern revealed a questionable line of mont-
morillonite and weak lines of illite, kaolinite, and quartz.
The x-ray pattern secured after treatment of the material
with E-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 montmoril-
onite 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, $2\theta$ varying
from 10 to 20 degrees, $\alpha = 1.559$, $\gamma = 1.578$. The base-
exchange capacity of this fraction was 95.24 milliequiva-
lents per 100 grams, the highest value obtained, and one
that can only be accounted for by a large amount of mont-
morillonite. The data indicate that this fraction is com-
posed chiefly of montmorillonite with a small amount of
illite.
DISCUSSION

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 I. As pointed out previously the moisture content and plasticity of sample 1316 is slightly greater than sample 1263. This would be expected if, for example, sample 1316 contained a predominance of montmorillonite. Whether or not this difference is significant would depend upon a study of many more samples similar to these.

Fractionation of the materials 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 II. Accordingly, it is impossible to forecast the behavior of these materials by a comparison of their colloidal content, although it is well known that in some circumstances, variations in the amount of colloidal material can be regarded as being diagnostic. That is, materials with a large percentage of colloidal particles behave differently from materials that contain negligible amounts.

The reaction of the leaching process which preceded the dispersion of the samples was noticeably different.
Sample 1263 effervesced with both acetic and hydrochloric acids, whereas sample 1516 gave practically no reaction. This indicates the difference in the carbonate content of the two materials and is also substantiated by the chemical analyses of the bulk and colloidal samples. It also accounts for the larger amount lost in the leaching process in sample 1263.

Both the sedimentation and fractionation by centrifugal action processes must be stopped at some arbitrary end point. In this work the separation of the colloidal and non-colloidal fractions was stopped when the amount of colloidal material being collected per week was less than 0.3 per cent of the original sample weight. The centrifuge operations were stopped for each fraction when the centrifugate from a given dispersion contained less than 0.2 per cent of the original sample weight. If one were to draw a curve plotting the amount recovered against the original sample weight, it would flatten perceptibly but never arrive at a complete endpoint. It should be possible then to continue centrifugal action on a clay suspension to a point where mechanical breakdown of the particles would always allow some finer particles to be separated.

The relative significance of the chemical, optical, x-ray, and base-exchange studies as means of determining clay minerals depends entirely on the degree that the various
minerals have been concentrated. If certain of the clay minerals are more or less concentrated in certain size fractions, then the analyses of that fraction should closely correspond to the analyses of the pure mineral. That this actually happens has been proven by Grim (4). If this was not the case the constants or characteristics of the fraction would not be those of any particular single clay mineral but would be the combined, resultant characteristics of two or more.

The chemical composition of all of the clay minerals, as summarized in Grim's work (10), shows that, chemically, the clay minerals differ very little. The difference is so slight as to make chemical analyses, alone, practically valueless as a means of determining the mineralogical composition unless one is positive he is working with pure minerals. However, the chemical analyses aid considerably when used with other methods of analysis.

The large decrease in silica from the bulk sample to the coarse colloid and the gradual decrease as the size of the fraction diminishes is caused by a decrease in the amount of quartz. This is also shown by the x-ray diffraction patterns. As quartz is more resistant to weathering and mechanical breakdown than any of the clay minerals and does not tend to weather to such small particles, this is to be expected. The changes in the amounts of iron and aluminum
and their relation to the amount of silica dioxide as shown by the silica-sesquioxide ratio is significant. It is slightly higher for sample 1316 than for sample 1263, and this is attributed to a concentration of clay minerals in the finer fractions, particularly of the montmorillonite type of mineral in sample 1316.

The results of the x-ray analyses show a definite tendency for the montmorillonite type of clay mineral to be predominant in sample 1316 and for illite and kaolinite in sample 1263 and offer the most conclusive proof of the mineralogical composition of the various fractions and when considered in conjunction with the other data afford well founded conclusions. Because of the difficulty in reproducing clearly many of the fine lines shown on the x-ray film, the plates may appear indistinct unless one knows what to look for and where. However, the last two reproductions of the fine colloid of sample 1316 and the superfine colloid of sample 1263 show remarkably well the results of immersing the samples in a solution of B-Naphthylamine to bring out the presence of the montmorillonite type of clay mineral. The position of the diagnostic lines on each of the films, which were used as evidence of the presence of minerals, are marked and numbered. These numbers agree with the calculated data in Table IV. It is also interest-
ing to note the presence of quartz in all fractions but the superfine.

The striking difference between the base-exchange capacity of the colloidal and non-colloidal fractions can only be explained by a comparatively good separation. The total base-exchange capacity of all of the colloidal fractions is notably high and probably indicates the presence of more montmorillonite than the other data suggest. Uniformity of aggregates and optical constants determined for the superfine colloid of sample 1316 substantiate the conclusion that the high base-exchange capacity of this fraction can only be accounted for by a large amount of montmorillonite.

The analytical results show that the two samples differ in the nature of their clay mineral content, 1316 being made up essentially of montmorillonite whereas sample 1853 contains illite and kaolinite as its principal clay mineral constituents. The difference 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 the 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. A study of the
field relations reveals that sample 1263 was taken from a horizon immediately above a relatively impervious gumbotil, while sample 1316 was taken from material 10 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 the intervening distance. 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.

This study is the first of its type on Iowa materials. For this reason it was thought desirable to gather as much data as possible in order to establish the fundamental characteristics. Because of the involved and lengthy procedures of analyses used, it was impossible to work with any more than two samples. It is recognized that a study of many more samples of different types of material would be desirable.

The plasticity test is suggested as a possible means of differentiating between materials of the nature of these
experimented upon in this work. Again a study of more samples will reveal whether or not this difference in plasticity should be considered significant.
CONCLUSIONS

1. At the present time it is impossible to predict the behavior of subgrade materials from a study of the physical properties as determined in the highway laboratory.

2. Fractionation by sedimentation and a supercentrifuge into several different size colloidal separates failed to reveal any significant difference in amounts of the several grade sizes.

3. Analytical methods, chemical, x-ray, optical, and base-exchange show that the samples differ in the nature of their clay mineral content, 1316 being made up essentially of montmorillonite, whereas sample 1363 contains illite and kaolinite as its principal clay mineral constituents.

4. The field relations of the two materials studied are such as to obscure any variation in the behavior of the materials that might be due to a difference in clay mineral content.

5. Highway engineers should take into consideration the possibility of differences of behavior being due to mineral differences, particularly of the colloidal fractions and the geological positions of the materials they plan to use in the subgrades of roads.
6. The plasticity tests (Table I) are slightly higher for the montmorillonite-bearing material and a study of more materials will perhaps reveal that this difference should be considered significant.

7. The possibility of using the great difference in the base-exchange capacity between the colloidal and non-colloidal fractions as a criterion of the perfection of separation of these fractions is suggested.

8. It is necessary to have as many different types of data as possible in order to determine the clay mineral constituents of materials of this nature.
ACKNOWLEDGMENTS

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LITERATURE CITED


