# THE IDENTIFICATION AND PROPERTIES OF COLLOIDS EXTRACTED FROM CERTAIN SOIL SERIES FOUND IN OKLAHOMA

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By

## NEIL FARRIS

Bachelor of Science

Oklahoma Agricultural and Mechanical College

Stillwater, Oklahoma

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APPROVED BY:

unplu

Chairman, Thesis Committee

M.g. Pe

Member of the Thesis Committee

num Head of the Department

Dean of the Graduate School

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### TABLE OF CONTENTS

I.	Introduction
II.	Review of Literature
III.	Purpose of the Study
IV.	Experimental Methods
v.	Results 15
VI.	Discussion
VII.	Summary
VIII.	Bibliography

# THE IDENTIFICATION AND PROPERTIES OF COLLOIDS EXTRACTED FROM CERTAIN SOIL SERIES FOUND IN OKLAHOMA

#### INTRODUCTION

The clay minerals are of fundamental importance in the soil. The movement of water within and through the soil and its availability to plants are greatly influenced by the clay content. Clay also has a direct influence on soil erosion. The state of dispersion of the clay particles and the stability of the clay aggregates greatly influence the erodibility of soils.

The clays have great absorptive powers that are important in the prevention of the loss of valuable plant nutrients. At the same time, some of the nutrients are held by clay in a form that is available to plants while different clays have the ability to fix plant nutrients in an unavailable form. Soil clay has long been known to possess the property of base exchange. Potassium, calcium, and magnesium in soils generally and sodium in arid regions are absorbed by clay and held in the exchangeable form.

When any of the absorbed bases of clay are replaced by H ions, the clay tends to become acid. The intensity of the acidity, that is, the pH is determined by the percentage saturation with H ions. Calcium clay is spoken of as that clay whose absorbed and, therefore, replaceable ions are largely calcium ions; and acid clay means clay containing a preponderance of H ions.

Clay is an important consideration in soil classification, particularly as regards its vertical distribution in the soil profile. The classification of soils as to type is based largely on clay content.

According to Grim (10), since the clay minerals occur in flake-shape particles, it follows that most clays are essentially aggregates of very minute flakes or slates.

Montmorillonite, kaolinite, and mica-like clays have been found in soils of practically all types of climate, and each of them seems to have been formed from various kinds of parent material.

#### The Clay Minerals

Growther (8) states that considerable progress has been made during the last few years in the mineralogy of clays, and X-ray studies have demonstrated that the bulk of the inorganic colloids in soils is definitely crystalline. A few clay minerals--kaolinite, halloysite, and montmorillonite--have been recognized in soil colloids. The layer structures proposed for certain clay and related minerals suggest interesting possibilities for interpreting some of the vital properties of the clay fraction of soils, such as water absorption and base exchange; but it must be admitted that most of the proposals are based on analogies with the somewhat uncertain structures of a few clay minerals.

There are two main groups of clay minerals, with low and high baseexchange properties respectively. The kaolin group with the ideal formula  $Al_2O_3$ ,  $2SiO_2$ ,  $2H_2O$  includes kaolinite-anauxite, dickite, nacrite, and halloysite. In the kaolinite-anauxite series the  $SiO_2$  :  $Al_2O_3$  ratio may vary from 2 to 3 without perceptible change of properties. The four members of this group have distinct X-ray patterns with other minor differences in their physical properties, especially in their dehydration curves. They all undergo decomposition at temperatures just above  $500^{\circ}C$ . Halloysite, which is nearly isotropic optically, has often been regarded as a very finely divided or amorphous kaolinite; but Ross and Kerr (30) have shown that though halloysite is closely related to kaolinite, it has a distinct X-ray pattern and dehydration curve, the lattice breaking down at about  $60^{\circ}C$  lower than kaolin.

Gruner (11) states that the group of clay minerals with high baseexchange power includes the alumino-silicate montmorillonite. Some

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authorities write the formula (Mg. Ca) 0,  $Al_2O_3$ ,  $5SiO_2$ ,  $nH_2O$ , with the  $SiO_2 : Al_2O_3$  ratio varying considerably. It is assumed that an Al (OH)<sub>3</sub> layer is condensed with two  $SiO_2$  layers, so that each Si is surrounded by 4 0 in a tetrahedron as in all silicate structures and each Al by 2 OH and 4 0 in an octahedron. Sandwickes of these three layers are about 6.6 Å. thick, but are separated from each other by a comparatively large variable distance which depends on the content of water or other liquid. In the air-dry state the material contains 23% of water and the structural layers are separated by 3.4 Å., giving a total distance of 15.2 Å, before the pattern repeats.

The crystals swell and shrink at right angles to these layers as the liquid content changes. Kelley, et al., (19) have shown that dehydration curves of the different clay minerals show that OH lattice constituents pass off as water vapor at temperatures which are characteristic for each class.

Analyses of clays by X-ray, optical, chemical, and dehydration methods have shown that they are generally aggregates of extremely minute crystalline particles of one or more species.

Physical properties of clays are considered in relation to the structure of their constituent clay minerals and the character of the exchangeable bases present. In general, plasticity, bond strength, and shrinkage are relatively high for montmorillonite clays and low for kaolinite clays. Grim (10) writes the formula for kaolinite as  $(OH)_3$  Al<sub>4</sub>Si<sub>4</sub>O<sub>10</sub> and montmorillonite as  $(OH)_4$ Al<sub>4</sub>Si<sub>3</sub>O<sub>20</sub> · XH<sub>2</sub>O. Magnesium is practically always present.

#### Origin of Clay Minerals

Grim (10) found in the system  $A1_2O_3 - SiO_2 - H_2O - K_2O$  at  $300^{\circ}C$  with equal cation concentration, montmorillonite is formed in an alkaline

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solution, kaolinite in an acid solution.

Norton (25) has shown that kaolinite is produced from feldspar by the attack of a variety of acids including organic acid in soils. Mattson (21) has indicated that there is a suggestion that less siliceous material, that is, hydroxides, kaolinite, and halloysite, tend to form under warm climatic conditions; and more siliceous material, that is, the montmorillonite minerals tend to form under cooler conditions. Ross and Kerr (30) state that kaolinite is produced by surface weathering and by the action of sulphate or carbonate waters. Ross and Kerr (31) state that halloysite appears to be the result of supergene processes; and so far as is known, it never forms as a result of hypogene hydrothermal processes. It forms through weathering and by the action of cool sulphatebearing solutions on aluminous materials.

#### Sesquioxide Ratio

Holmes and Hearn (16) found that, generally, soils lacking good drainage showed a higher silica-sesquioxide ratio than those of better drainage development.

The low silica-sesquioxide ratios for the well-drained soils indicate a material alteration in the assumed composition of the original colloid complex.

It is reasonable to assume that the greater part of the clay content now present in these soils existed in the soil parent material at the time it was laid down. This assumption is supported by the fact that the residual material other than clay is essentially quartz. It is also reasonable to assume that the clay in this parent material, as it came from somewhat the same source, was rather uniform in chemical composition at the time of its deposition. If the two above assumptions are true, then differences in the composition of the colloids are the

result of the differences in the present active soil forming processes. Alexander, Byers, and Eddington (1) have shown that the morphological difference between the zonal group of soils is reflected in their chemical composition. Working with colloids of nine of the erosion station surface soils, Holmes and Hearn (16) found an immediate and definite relation between the composition of the colloids, as shown by their silicasesquioxide ratios and their settling volumes. The colloids of the lateritic type are in general of low settling volume and the high silicasesquioxide ratios have greatly increased water-saturation capacities.

The water-saturation capacity represents the greatest quantity of water which may remain in equilibrium with the soil. This quantity cannot, of course, be found in the field sample; but the condition represented by it must be approached when, under very heavy rain, the surface of a soil reaches the condition described by the term "quagmire".

Hough and Byers (17) have shown that both the laterites and lateritic soils are characterized by decreased silica content of the whole soil as compared with presumably parent lava, and the colloids by higher silica content than the soils.

#### Base-Exchange Capacity

Ross and Kerr (30) found base-exchange capacity in milliequivalents per 100 grams of montmorillonite to be 60-100 and for kaolinite a range of 3-15. Base-exchange is known to vary with the size of particles; however, this is only for a particular sample. Other results from investigators vary widely from these.

Benzidine Color Reaction as a Test for Montmorillonite

Hendricks and Alexander (15) have shown that in the absence of organic matter, benzidine or its hydrochloride gives a blue color with montmorillonite and that the reaction is specific enough to characterize

this mineral in the presence of other clay minerals and constituents of soils. Hauser and Leggett (13) observed a series of color reactions between various amines and different clays. They found that the color is specific to the amine and can be produced with all reactive types of clay.

Hendricks and Alexander (15) used a saturated aqueous solution of benzidine hydrochloride (after oxidation of organic matter with  $H_2O_2$  where necessary). They found that halloysite and kaolinite give negative results and montmorillonite gives a dark blue color.

As was pointed out by Hendricks and Alexander, the color developed is undoubtedly due to the formation of a semiquinone compound resulting from the partial oxidation of the diamine. These authors suggest that iron may be connected in some way with the color development.

Hauser and Leggett (13) suggest that the clay particle itself can act as the oxidant, accepting an odd electron from the adsorbed amine.

Page (26) later tried to disprove the benzidine hydrochloride test on the ground that various iron compounds could be added to any clay and positive results could be obtained.

#### Phosphate Fixing

Murphy (24) investigated the possible role of the mineral, kaolinite, in phosphate fixation. He found that ground kaolinite fixed large amounts of phosphate in a form unavailable to plants. In one instance 100 grams of ground kaolinite suspended in a solution of  $KH_2PO_4$  fixed 10.3 grams of  $PO_4$ , which corresponds to a fixation of 324 milliequivalents per 100 grams.

Stout (33) discloses a marked difference in the adsorption of phosphate and ammonia by montmorillonite as contrasted with the kaolinite and halloysite. From a normal ammonium acetate solution of pH 7, the montmorillonite adsorbed 81 milliequivalents of ammonia per 100 grams; the 3-day-ground kaolinite, 37 milliequivalents per 100 grams; and the

9-day-ground halloysite, 73 milliequivalents per 100 grams of the air-dry mineral. The adsorption of phosphate by the kaolinite and halloysite is thus many fold greater than the adsorption of phosphate by the montmorillonite at pH 7. An even greater distinction was shown by the adsorption of the three minerals when the acidity was increased to a pH of 3.

Murphy (24) points out that iron and aluminum hydrosols and hydrogels are known to have the ability to remove soluble phosphates from solution under certain conditions. Hydrolysis of the freshly prepared iron complexes occurs, however, and at pH 6 and above there is a considerable release of PO<sub>4</sub> even in the presence of an excess of iron. The fixation by kaolinite is extremely high for all concentrations of soluble phosphates in the reaction range of agricultural soils. The fixation is greatest at acid reactions. Then the exposed solution contains  $H_2PO_4$ ions, the fixation is great.

Colloids having a high silica-sesquioxide ratio have a lower capacity to fix phosphates, as shown by both chemical and greenhouse work. This was found to be generally true by other investigators.

Greenhouse results substantiate the laboratory data in showing that montmorillonite has a much lower capacity to fix phosphorus in an unavailable form for plant growth than does colloidal kaolinite.

Harper (12) shows that the fixation of phosphorus by soil is the conversion of the soluble phosphates to a less soluble form. The process is influenced largely by the pH of the soil and by active cations predominating in the colloidal complex.

#### Potassium

Results of Bray's (3) work indicated a decrease in content of  $K_20$  with decrease in particle size. X-ray analysis showed this to be due

to the decrease in mica content. Illite mineral was found in most shales as a  $K_20$ , MgO mica with a SiO<sub>2</sub> :  $R_2O_3$  ratio of 3.0. Illite was found to have relatively no base-exchange properties.

According to Coleman (7), fixation of phosphate by both montmorillonitic and kaolinitic clays is due largely to the free iron and aluminum in the clay; for when the free iron and aluminum were removed, the clay minerals lost most of their ability to absorb phosphate.

Ross and Kerr (30) have found that dehydration tests indicate that kaolinite loses the largest part of its water content at about  $450^{\circ}$ C to  $550^{\circ}$ C, and that illite and montmorillonite's water loss are more nearly constant with a temperature up to  $750^{\circ}$ C.

The purpose of this investigation was to obtain more fundamental data on the behavior of minerals present in soil colloids in relation to certain clay minerals in the pure form under a given set of conditions. During recent years it has been found that clay minerals are important constituents of soil colloids. Investigators have found that by subjecting a mineral to certain tests, a reaction occurs that is characteristic of that particular mineral.

By the use of suitable chemical analyses, color reaction tests, and dehydration tests, an effort was made to identify the predominating minerals found in certain soil colloids.

#### Source of Materials

The soils were collected from depths of from 0-7 inches in different parts of the state of Oklahoma. The county soil surveys were used in determining the soil series. The kaolinite was obtained from California, the halloysite from Missouri, and the montmorillonite from the American Colloid Company.

#### Method of Extraction of Colloid

One to four kilograms of soil were dispersed with 10 to 15 liters of distilled water in a 5-gallon stone jar. A small amount of ammonium hydroxide was added to aid in dispersion. Stirrings were made frequently to tear down the aggregates. The liquid was allowed to stand sufficiently long for the silt and sands to settle and this supernatant liquid decanted into another 5-gallon stone jar. This process was repeated until sufficient colloids remained in suspension. The liquid was filtered through porcelain Chamberland filters connected with a suction pump. After the filter was covered with colloids the suction was reversed in a fruit jar. This process was repeated until a sufficient quantity of colloids was obtained. A portion of the colloids was placed on a porcelain plate to air-dry. A portion of these was placed in a platinum dish in the oven to make moisture determination on an oven-dry basis.

The colloids were then placed in a desiccator for subsequent determinations.

#### Sodium Carbonate Fusion of the Colloids

One gram of oven-dry colloid was mixed on glazed paper with four grams of sodium carbonate. The mixture was placed in a platinum crucible and heated until fusion resulted. The mixture was heated an additional 10 minutes with an oblique flame. The contents were poured into a large platinum dish set in water. The crucible was placed in a wide 200 ml. beaker and covered with water, transferring the infused lump from dish to beaker. Fifteen cc. of concentrated hydrochloric acid were added, the beaker covered and placed on steam plate until contents disintegrated, after which they were placed back in the platinum dish and evaporated to dryness.

The residue was taken up in hydrochloric acid (1 to 10) and filtered through a 9 centimeter Buchner funnel with suction. It was then washed with hot water containing 1 to 2 cc. hydrochloric acid per liter. The filtrate and washings were collected in a casserole and dehydrated on a steam bath until the silica assumed a crystalline appearance. The silica was moistened with hydrochloric acid and dehydration repeated. Five cc. concentrated hydrochloric acid and 100 cc. of hot water were added, the residue filtered, washed, and then added to main portion of silica obtained from the first filtration. The combined filtrate and washings were made up to 500 cc. and saved for subsequent determinations.

#### Sesquioxide Determination

One hundred cc. of leachate of the above extraction were heated just to the boiling point; a small piece of pink litmus paper was added and the iron, aluminum, and phosphorus were precipitated by the slow addition and constant stirring in of 1 : 1 ammonium hydroxide, using just enough ammonia to turn the litmus paper blue. The solution was heated to the boiling point for 60 seconds to boil off excess ammonia and to flocculate the precipitate. The contents were filtered immediately through No. 30, 11 cm. paper and washed four times with distilled boiling water. The filter and precipitate were placed in an oven to

dry completely. Next, the filter and precipitate were placed in weighed porcelain crucibles and ignited to redness over flame for 20 minutes then cooled and weighed as sesquioxides, neglecting the small quantities of TiO<sub>2</sub>, P<sub>2</sub>O<sub>5</sub>, and Mn<sub>3</sub>O<sub>4</sub> which it may have possibly contained.

#### Determination of Iron and Aluminum Oxides

The ignited precipitate obtained from the sesquioxide determination was placed in a small beaker. Traces adhering to the crucible were removed by heating small portions of 6 N hydrochloric acid in it, finally pouring each portion into the beaker, using 20 milliliters of acid in all. The acid with the iron and aluminum oxides were heated at about  $90^{\circ}$ C until all iron had dissolved. When a clear solution was obtained, the beaker was removed and the contents were reduced carefully with stannous chloride, and the iron was determined by titration with .0547N potassium permanganate. The percentage of Fe<sub>2</sub>O<sub>3</sub> present was computed and a subtraction was made from the sesquioxides to get the per cent of Al<sub>2</sub>O<sub>3</sub>.

#### Phosphate Fixation

Two-gram samples of halloysite, kaolinite, montmorillonite, and ten Oklahoma soil colloids were shaken intermittently for seven days with 10 cc.  $KH_2PO_4$  solution containing 6.2 mg. of phosphorus. The colorimetric MoO<sub>3</sub> method for the determination of phosphorus was used with a photometer. A blank reading was made, and calculations were derived on that basis.

#### Base-Exchange Capacity at Various pH Values

Two and one-half grams of colloids were treated at  $70^{\circ}$ C for one hour with 25 cc. of anmonium acetate. The suspension was filtered and 5 cc. of one-tenth normal NH<sub>4</sub>Cl were leached through, then an additional 75 cc. more. The colloids were next leached with methyl alcohol until free from free NH3 and chlorides.

The filtrate was then distilled (Kjeldahl procedure) and NH<sub>3</sub> determined by the titration method. The base-exchange capacity was determined at three given pH values: 4.6, 7, and 9.1, respectively. Ammonium acetate has a pH of 7 at 1N. To increase the pH a base was added and to decrease the pH an acid was added. The pH of the three solutions was determined by the glass electrode.

#### Potassium

A one-gram sample of the colloid was digested on the steam plate with hydrochloric and perchloric acid. The usual perchloric procedure was used. The precipitate was weighed as KClO<sub>4</sub> and the per cent of potassium was derived from this formula.

#### Dehydration of Colloids and Minerals

Samples of the colloids and minerals were brought to a comparable moisture condition by placing them in a furnace at a temperature of 100°C for 24 hours. Due to the depletion of four samples of the colloid, only six samples of colloids were run in this experiment. In order to get a comparison of the behavior of the colloids, three pure minerals were investigated for dehydration at different temperatures.

The nine samples in platinum crucibles were placed in the furnace for 2 hours at each of the following temperatures:  $340^{\circ}$ C,  $400^{\circ}$ C,  $530^{\circ}$ C,  $630^{\circ}$ C, and  $750^{\circ}$ C. At each temperature the sample was cooled, weighed, and subtraction was made from previous weighings and calculations were made on a percentage basis.

#### RESULTS

The colloid analyses (Table 1) show generally a relatively high silica-sesquioxide ratio. There is, however, a wide range of ratios, for example, from 2.09 with the Miller series to 4.66 with the Kirkland series. This may be explained by the fact that the Miller series is an alluvial soil and the Kirkland series is a residual soil. As indicated in table 1, all the soil colloids examined exhibited a relatively small iron content. The silica-alumina ratios appear to have a fair correlation with the silica-sesquioxide ratio for all colloids.

The total potassium content of each of the ten colloids used is shown in table 2. The potassium content ranged from 1.00 per cent in the Kirkland colloid to 2.89 per cent for the Miller colloid. This can be attributed to perhaps a larger per cent of mica clay in the Miller colloid (3).

The effect of phosphorus absorption by different soil colloids and clay minerals is shown in table 3. It may be worthy to note that all the pure minerals absorbed more phosphorus than any of the soil colloids; however, the results of the minerals are in accord with other investigators (24, 33).

Generally, with the exception of montmorillonite, all colloids and minerals increased in base-exchange capacity with the increase in their pH values. At any rate, no wide difference was noticed except for the mineral kaolinite as shown in table 4.

A combination of colors was obtained by the montmorillonite test with the soil colloids. The results found in table 5 pertaining to the clay minerals are in accord with the findings of other investigators (15, 26).

As shown by the dehydration test in figure 1, the soil colloids and clay minerals vary greatly as to their water loss at the same temperatures. By the curves, it is obvious that all of the colloids are mixtures of minerals, since their curves fall between the mineral, montmorillonite, which loses 5.2% moisture at  $750^{\circ}$ C, and the mineral, halloysite, which loses 16.5\% moisture at  $750^{\circ}$ C.

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Sample	\$10 <sub>2</sub>	Oxides	Fe203	A1203	S102 R203	S102 Fe203	Si02 Al203
Gerald	.6082	.3475	.0255	.3220	3.05	63.74	3.21
Foard	.6141	.2900	.0315	.2585	3.76	52.21	4.03
Hanceville	.6964	.2750	.0245	.2505	4.45	75.85	4.72
Kiowa	.6129	.3745	.0250	.3495	2.85	65.48	2.98
Kirkland	.6694	.2520	.0225	.2295	4.66	79.68	4.46
Miller	.5907	.3415	.0256	.3159	2.09	61.53	3.17
Osage	.6253	.3205	.0310	.2895	3.43	53.99	3.67
Oswego	.5978	.3540	.0210	.3330	3.31	76.05	3.05
Randall	.6542	.2665	.0325	.2340	4.36	53.70	4.75
Vernon	•5540	•3525	.0300	.3225	2.75	49.37	2.92

Table 1. Chemical analysis of ten Oklahoma soil colloids.

## Table 2. Potassium analysis.

Sample	% Total K in one gm. colloid	% Total KCl04 in one gm. colloid
Gerald	2.13	.0757
Foard	1.62	.0574
Hanceville	1.41	.0501
Kiowa	1.44	.0510
Kirkland	1.00	.0353
Miller	2.89	.1023
Osage	1.70	.0626
Oswego	1.24	.0440
Randall	2.14	.0760
Vernon	1.04	.0368

Sample	Mgs. Unabsorbed Phosphorus	Fixed in 2 gm. colloid
Gerald	3.16	3.04
Foard	3.86	2.34
Hanceville	3.27	2.93
Kiowa	3.21	2.99
Kirkland	4.04	2.16
Miller	3.69	2.51
Osage	3.77	2.53
Oswego	3.94	2.26
Randall	3.47	2.73
Vernon	3.94	2.26
Halloysite	1.93	4.27
Kaolinite	2,11	4.09
Montmorillonite	2.67	3.43
Blank	6.2	0

Table 3. The fixation of phosphorus by soil colloids and pure clay minerals.

Table 4. Base-Exchange in milliequivalents per 100 grams of clay minerals and soil colloids at various pH values.

Sample	pH 9.1	pH 7	рН 4.6
Gerald	43.2	43.6	41.6
Foard	66.4	65.6	64.0
Hanceville	36.4	34.8	36.4
Kiowa	66.6	64.6	64.6
Kirkland	43.2	42.0	42.8
Miller	43.9	43.2	41.9
Osage	50.8	50.0	48.4
Oswego	70.4	68.0	70.0
Randall	44.0	43.6	43.2
Vernon	50.4	50.4	50.0
Halloysite	43.6	38.8	40.8
Kaolinite	28.0	18.0	17.6
Montmorillonite	108.0	110.0	112.0

Sample		Color
Gerald		Bluish black
Foard		Blue
Hanceville		Dark blue
Kiowa		Bluish green
Kirkland		Dark blue
Miller		Greenish blue
Osage		Lead blue
Oswego	LUC States	Lead blue
Randall	Service and Service	Light blue
Vernon	the second second	Bluish red-purple
Halloysite		Colorless
Kaolinite		Colorless
Montmorillonite		Dark blue

Table 5. Benzidine hydrochloride test for montmorillonite.



#### DISCUSSION

The Gerald colloid was obtained from soil in northern Oklahoma near the Chilocco Indian School. The silica content of the colloid is 60.82% as shown in table 1. The silica-sesquioxide ratio is 3.05 which denotes that only medium weathering has occurred (28), and that it probably has a large percentage of minerals other than kaolinite as shown by the silica-sesquioxide ratio. The potassium content of 2.13% (table 2) is high, which would indicate considerable mica minerals to be present. The Gerald colloid fixed more phosphorus than any of the colloids investigated (table 3), which, according to some investigators (24), may indicate also a high percentage of the kaolin group. In table 4 the base-exchange capacity is given in three columns at various pH values. The possible explanation of its relatively low base-exchange is that the illite mineral, in the mica group, has a very small baseexchange capacity (18). Table 5 shows the color for the montmorillonite test to be bluish black. This test indicates the colloids contain a considerable amount of montmorillonite.

The Foard colloid contains slightly more silica than does the Gerald colloid. A great deal of difference is noted in their silicasesquioxide ratio, the Foard colloid being .71 higher. This is due to less weathering of the Foard colloid and indicates that it may contain a high per cent of montmorillonite. As shown in table 2, the potassium content is just fairly high with 1.62%. The amount of phosphorus fixed, as shown in table 3, is definitely on the low side. The exceptionally high base-exchange capacity (table 4) at various pH values seems to warrant a comparison of this colloid with montmorillonite. The montmorillonite base-exchange capacity is given in the last line of table 4.

Its blue color is another positive test to consider (15).

The Hanceville soil is found in eastern Oklahoma. The data in table 1 show the silica content of the colloid from this soil to be higher than any of the other colloids that were investigated. It has a silica-sesquioxide ratio of 4.45. Table 1 shows the colloid to be fairly low in potassium. This may be attributed to a low percentage of micas or potassium-bearing minerals. The Hanceville colloids fixed 2.93 milligrams of phosphorus in two grams of colloids. This is fairly high in comparison with the other nine colloids. The exceedingly low base-exchange of this colloid may be caused by the lack of time for its reaction with the ammonium ion. The ion was in contact with the colloid for only one hour, and this colloid may have been slower to react than the other colloids. The benzidine hydrochloride test as shown in table 5 gives a positive test for montmorillonite. In figure 1, the dehydration curve shows the Hanceville colloid to break down about 100°C before montmorillonite and to lose about 13% more moisture than montmorillonite at 750°C.

Soil of the Kiowa series was collected in Kiowa County, Oklahoma. The colloid from this soil contains 61.29% silica (table 1). As shown in column 5 of table 1, it has a low silica-sesquioxide ratio. This may be caused by the percentage of iron and aluminum lower in the profile. The potassium content is 1.44% as shown in table 2. The colloid fixed 2.99 milligrams of phosphorus in a two-gram sample, which may indicate considerable kaolinite present. The base-exchange capacity at various pH values shows this soil to be comparable to the montmorillonite type of clay as shown in table 4. The bluish green color as shown by the benzidine hydrochloride test for montmorillonite would indicate a mixture of minerals.

The Kirkland colloid was from Kirkland loam collected in Payne County, Oklahoma. The silica content is high with 66.94% as shown in table 1. As shown, it has the highest silica-sesquioxide ratio of all the colloids investigated. This would indicate that very little weathering has occurred. As shown in table 2, the potassium content is very low. This would lead to the assumption that the potassium bearing mineral, illite, is absent from the colloids or present in a very small quantity. This colloid fixed only 2.16 of phosphorus (table 3) in a two-gram sample of colloids. According to some workers (24), this particular soil would have very little kaolinite. The base-exchange capacity in milliequivalents per 100 grams ranges from 42 to 43.2 at various pH values. The benzidine hydrochloride test as presented in table 5 indicates a positive test for montmorillonite. In the dehydration curve (figure 1), the Kirkland colloid correlates fairly closely with that of the montmorillonite elay.

The Miller colloid contains 59.07% silica. Its silica-sesquioxide ratio of 2.09 is the lowest of all the colloids examined. It has the highest total potassium - 2.39 - as shown in table 2 and fixed only 2.51 mg. of phosphorus in a two-gram sample of colloid. The baseexchange capacity is similar to that of Kirkland as shown in table 4. The benzidine hydrochloride test with a greenish-blue color shows this colloid to be a possible mixture of kaolinite, montmorillonite, and mica minerals. This seems to be logical since this is an alluvial soil.

The Osage colloid has 62.53% silica. It has a silica-sesquioxide ratio of 3.43. The relatively high silica-sesquioxide ratio indicates that little weathering has occurred. Table 2 shows the potassium content to be fairly high. This may indicate the presence of mica minerals. Two grams of the Osage colloid fixed 2.53 mg. of phosphorus. The

base-exchange capacity in milliequivalents ranges from 48.4 to 50.8. This indicates, in relation to the other colloids, that it has a medium base-exchange capacity. The benzidine hydrochloride test showed a leadblue color. This indicates that the colloid contains considerable kaolinite (15).

The Oswego colloid contains 59.75% silica and has a silicasesquioxide ratio of 3.31. The potassium content (table 2) is 1.24. The phosphorous fixed is 2.26 mg. in a two-gram sample of colloid. The base-exchange capacity in milliequivalents per 100 grams of colloid ranges from 63 to 70.4. This is comparable to the montmorillonite type of clay; but by the benzidine hydrochloride test, as shown in table 5, it approaches the kaolinite. One may assume that this colloid contains a high per cent of both montmorillonite and kaolinite. According to figure 1, the dehydration curve shows that the Oswego colloid resembles the clay mineral, halloysite, more nearly than any of the other colloids examined by this method. However, it has a fair correlation with kaolinite.

The Randall colloid as shown in table 1 contains 65.42% silica with a high silica-sesquioxide ratio of 4.36 as shown in column 5. The high per cent of potassium in the colloid may be due to the illite mineral as shown in table 2. The Randall colloid fixed 2.73 mg. of phosphorus in two-gram samples of colloid. The base-exchange capacity was fairly low, ranging from 43.2 to 44 milliequivalents per 100 grams of colloid. The benzidine hydrochloride test (13 and 15) indicates that the montmorillonite mineral is present.

The Vernon colloid was from Vernon soil collected in Payne County, Oklahoma. Table 1 shows a silica content of 65.4% and a silicasesquioxide ratio of 2.75. This is slightly higher than the data shown

by Middleton, Stater, and Byers (23). These investigators found the silica-sesquioxide ratio to be 2.65. The potassium content as shown in table 2 was 1.04. The phosphorus fixed, as shown in table 3, is 2.26 mg. per two grams of colloid. The Vernon colloid shows a medium base-exchange capacity, the various pH values showing very little variation. The Vernon colloid gave a bluish red-purple color with the benzidine test. This indicates that this particular colloid probably contains in its mixture of minerals a mineral not present in the other colloids examined.

#### SUMMARY

An investigation was made of ten Oklahoma soil colloids: Gerald, Foard, Hanceville, Kiowa, Kirkland, Miller, Osage, Oswego, Randall, and Vernon. Three clay minerals: halloysite, kaolinite, and montmorillonite were also investigated in pure form in order that a correlation might be made with the soil colloids in an attempt to determine the clay minerals present.

Six tests for differentiating clay minerals were used on all the colloids with the exception of the dehydration test which was conducted on only six of the colloids.

The tests made were:

- 1. Sesquioxide ratio determination
- 2. The per cent of total potassium
- 3. The fixation of phosphorus
- 4. Base-exchange capacity at various pH values
- 5. Benzidine hydrochloride test for montmorillonite
- 6. Dehydration test

It is concluded that, generally, the higher the silica-sesquioxide ratio, the higher the percentage of montmorillonite contained in the soil colloids examined. All the colloids contained a high per cent of silica in comparison to colloids studied by other investigators.

Generally, there was no correlation with all six tests used on the soil colloids; however, there was definitely a correlation with some of the positive tests for certain minerals.

The base-exchange capacity of the colloids increases with the increase in pH values.

It is possible to determine the montmorillonite type of clay present in colloids by the blue color reaction with benzidine hydrochloride.

The dehydration curves appear to be somewhat characteristic of each type of colloid, the shape of the curve depending very largely on the

kinds and relative proportions of the different clay minerals present.

In no case was there an occurrence of just one specific clay mineral in any of the soil colloids examined. Despite the fact that many of the colloids have shown positive results for certain clay minerals, the lack of experimental data makes it impractical to rely completely on these tests.

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Maxine C. Farris, typist