

THE APPROXIMATE SIZE OF SOIL PARTICLES AT
WHICH THE HEAT OF WETTING IS MANIFESTED
AND ITS USE IN DIFFERENTIATING
COLLOIDAL FROM NON-COLLOIDAL
SOIL MATERIAL.

by

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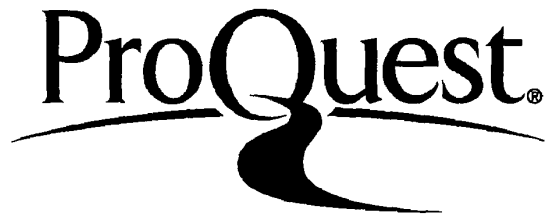
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INTRODUCTION

Recently there has been a great deal of controversy concerning the basis of distinguishing and defining soil colloids from non-colloids. Even in the text books on colloids there is a difference in size attributed to colloidal systems. It is true that size is one of the most dependable criteria for colloids but there are wide variations in the diameter of the particles of different elements and compounds at the point where the exhibiting of the generally accepted properties of colloids cease. The Brownian movement is given as one of the properties of colloidal systems, although it is now definitely known that an absolute size limit cannot be set where this movement ceases for all colloidal systems. Therefore, why should we consider this Brownian movement as a true colloidal property when we realize that the larger size particles give us other colloidal properties and not this one? This has been considered by most workers as only one property of a colloid smaller in size than the maximum limit which we attribute to soil colloidal material.

Anderson et al (2) selected one micron as the upper limit for colloidal particles in the study of soil colloidal material absorption. Davis (8) in the study of the plasticity and coherence of different soils used one-tenth of a micron

as the upper limit. Most workers state that one micron is taken as the diameter of the largest size soil colloidal particle as this is the upper limit of Brownian movement. Anderson (1) in the work on the heat of wetting of different minerals takes as the upper limit of non-colloidal material that which passes through a 130 mesh sieve but does not set a definite lower limit. Due to the difficulties and labor involved in a complete mechanical separation of a soil and finely ground minerals, the lower limit of size that most workers were dealing with is not known.

It must be admitted that at present the classification of soil particles into colloidal and non-colloidal material is not based on any fundamental grounds, but is merely an arbitrary classification. Sjøllema (15) considers that practically all the constituents of soils, except quartz grains and undecomposed mineral fragments, are colloids, because they are colored by dyes. Gile et al (9) states that the data on the heat of wetting of minerals closely parallel the results obtained in a more extensive study of soil minerals with respect to absorption, indicating that the heat of wetting is almost a function of the colloidal material. Bouyoucos (6) states that recent studies on heat of wetting indicates that the finer silt separates manifest the heat of wetting. In the studies of soils by the hydrometer method he includes the fine silt in the colloidal content of a soil. He believes that the material which exhibits the property of heat of wetting should be spoken of as colloidal material, and that which does not possess this

property should be spoken of as non-colloidal material, regardless of the size particles. Since this property is an energy manifestation, rather than arbitrary size limit, it appears most logical to adopt it for distinguishing colloidal from non-colloidal material.

Since the absorptive power and heat of wetting become more pronounced as the particle size decreases, it was considered advisable to separate different soils into different size separates as accurately as possible, and to ascertain the size at which the heat of wetting ceases to be manifested. To secure soil separates suitable for heat of wetting determinations, it was necessary to free the soils from calcium and magnesium carbonates, organic matter, and coating of gels around the soil particles.

The calcium and magnesium carbonates, if present, would lower the heat of wetting of the separates by decreasing the effective weight of the soil, as carbonates according to Bouyoucos do not possess the heat of wetting property. This effect would be more pronounced in the fine separates, such as colloid and clay, as they are in most cases evaporated to dryness and the carbonates from the entire soil are thus concentrated in these separates.

The organic matter and coating of gels must be removed, as organic material and gels give a high heat of wetting. The coatings exist in natural field conditions as sols around the soil particles, and no doubt are hard to remove completely.

It was thought that pure minerals and rocks being free from organic matter and coating of gels should be ground for

comparison with the soil separates. In this manner a few interesting facts may be secured on the important question of the approximate size soil particles at which the heat of wetting property cease to be manifested.

EXPERIMENTAL PROCEDURE

The heat of wetting was measured according to the method described by Bouyoucos (3). It consisted of placing twenty-five to fifty grams of air-dried material in a wide glass tube and drying in an electrically heated oven at a temperature of 110° Centigrade for twenty-four hours. The tube was removed, closed with a rubber stopper, and allowed to attain the room temperature. After exact readings of weight and temperature were observed, the soil was quickly and carefully poured into a calorimeter containing 100 grams of water. Extreme care was exercised before mixing to have both the soil and water at exactly room temperature. In order to be able to convert, if necessary, the rise of temperature into heat calories, the water equivalent of the calorimeter was determined. This was found to be twenty-five grams of water. For the specific heat of the material used in this work, the value of 0.2 was employed throughout. This value was used for the mineral separates as well as for the soil separates as Regnault, Joly, Batholi and Pionchon (13) found this value for different mineral powders several years ago.

The temperature of a gray soil was used as a standard for obtaining the temperature of the materials used in each determination. This gray soil had been subjected to the same conditions of heating and cooling as the other materials. Suppose that the temperature of the gray soil was 0.05° C. lower than that of the other materials used. As the specific heat is 0.2 calorie per gram, the water would be increased 0.01 calorie for each gram added to the calorimeter. Therefore,

by using 20 grams of material the increase heat transferred to the water would be only 0.2 calories. It is impossible to read this increase in 100 grams of water. Therefore, the small difference in temperature between the standard and the materials used, would not effect the results to a measurable extent.

The thermometers used were checked against each other. They could be read within 0.025° C. and slightly less with the aid of a reading glass. Therefore, if the heat of wetting was only 0.2 calorie per gram and fifteen grams were used, the increase in temperature would be only 0.024° C. on the determination thermometer. This increase in temperature could be read quite easily.

If extreme care is used, the temperature of the water in the calorimeter would never be higher than that of the separates. In every case it would be lower, due to the cooling effect by evaporation from the water surface and wet parts of the calorimeter.

After considering the above sources of error, it appears logical to believe that 0.2 calorie per gram of material is sufficient to consider as an experimental error for each determination.

The soils were treated with dilute hydrochloric acid, washed with suction until free from chlorides, and then treated with dilute hydrogen peroxide. The hydrogen peroxide used was Mercks Superoxyl, a commercial product containing thirty per cent hydrogen peroxide. The dilute hydrogen

peroxide treated soils were heated on a steam bath until the effervescence ceased and more concentrated solution could be used. Finally the thirty per cent solution was used. The soils were then transferred to a Buchner funnel and washed free from the formed organic compounds. They were then dispersed every day for two weeks in a special dispersion machine described by Bouyoucos (5). After each dispersion, the soil was placed in a liter beaker which was filled with distilled water to a depth of 13 cm. The temperature of the water was taken and after a certain interval of time, read from Table 1, the particles in suspension were siphoned off. In order to obtain the particles, this suspension was placed on a steam bath to evaporate the water present or the supernatant liquid siphoned off.

The soils were treated with dilute hydrochloric acid to decompose the carbonates present and remove the bases in the form of chlorides. The carbonates are known to have a decided effect upon the heat of wetting produced by soil as their presence reduces the weight of soil and thus gives a low heat of wetting determination per gram of soil material. Pate (12) has shown that monovalent bases in a soil decreased the heat of wetting as much as 46% in some cases. The divalent bases did not exert such an influence.

As organic matter gives a high heat of wetting, as much as possible was removed by the hydrogen peroxide to secure samples suitable for the determinations. Many workers have estimated the amount of organic matter that can be removed by the use of this compound. Other chemicals or heat could not

be used because this would influence or destroy the heat of wetting property of the soils. Robinson (14) in his recent work on the hydrogen peroxide method of oxidizing organic matter in soils, states that the variety of soils and colloids containing 0.42 to 95.85% of organic matter show that practically all the organic matter can be decomposed in some soils and that in other soils considerable organic matter is unattacked. If the organic matter is not destroyed, one may expect it to have an effect on the heat of wetting determination.

The intensive dispersion treatment should rid the particles of most of their gel coatings and give particles above 0.005 mm. in diameter of pure crystalline minerals. However, it is doubtful if all the colloidal gel coatings can be removed, especially from the small silt particles, since the gels, adhere very strongly to the particles.

The minerals used were ground to a fine state with an agate mortar and pestle. It was realized that as quartz has the same hardness as agate which is a quartz given this name because of its color, the powder would not be contaminated with any foreign matter as it might be if another apparatus were used for grinding. The other materials used will not grind away the agate mortar and pestle as their hardness is below that of agate.

After grinding, the different sized mineral particles and treated soils were separated by the application of Stokes' law. Although there may be many objections to the

employment of this law, still it has been adopted by the National Soil Science Congress (17) as an aid in the mechanical analysis of soils. The formula used in calculating the size of the particles at various rates of falling is as follows:

$$v = \frac{2 \text{ g d}^2 (s-s_1) .6}{9 \text{ nt} .4}$$

v - distance in cm. of a particle d size to fall in one minute

d - diameter of particle in mm.

s - specific gravity of material used.

s₁ - specific gravity of water.

nt - coefficient of viscosity of the water at given temperature.

g - force of gravity or 980 dynes.

2/9 - factor expressed in Stokes original law.

6/40 - factor used to express v in terms of centimeters per minute when diameter is in millimeters.

As $v = \frac{\text{cm}}{t \text{ in minutes}}$ as well as the formula above, and as we are interested in finding the time, the equation becomes (A) $t \text{ in minutes} = \frac{\text{cm depth of liquid} .9 . \text{nt} .40}{2 . \text{g} . \text{d} .^2 . (s-s_1) .6}$

The value of (s) changes for different minerals but for the soil separates 2.65 was always used.

Table 1 was calculated by the above general formula (A) for a depth of 13 cm. of water for both kinds of separates.

In the process of separation by the application of Stokes' law, the concentration of the material in water was never allowed to exceed 5% of weight. By keeping the concentration below 5%, the errors due to the friction of the solid particles in suspension would not be encountered and

TABLE 1 - TIME IN MINUTES FOR FOLLOWING SIZE (mm.) PARTICLES
TO SETTLE 13 CM.

Temp. in C.	Coefficient of viscosity of water	Size of Particles in Millimeters				0.035	0.05
		0.00125	0.005	0.01	0.02		
20	0.01005	1554.40	96.96	24.24	6.06	1.98	0.9696
21	0.0098	1512.60	94.54	23.63	5.91	1.93	0.9454
22	0.00958	1479.60	92.42	23.10	5.77	1.89	0.9242
23	0.00936	1444.80	90.30	22.57	5.64	1.84	0.9030
24	0.00915	1412.40	88.27	22.07	5.52	1.80	0.8827
25	0.00894	1380.00	86.25	21.56	5.39	1.76	0.8625
26	0.00875	1350.60	84.41	21.10	5.28	1.72	0.8441
27	0.00856	1321.20	82.58	20.64	5.16	1.70	0.8258

the results would be more accurate. In making the separations, the samples were thoroughly stirred with the water, the temperature was taken, and after the mixture had been allowed to stand for the length of time read from the table for the specific sample, the water and its suspended particles were siphoned off by using a glass tube having a side hole at the lower end. This was used to avoid drawing into the tube the particles which had already settled to the bottom.

EXPERIMENTAL RESULTS

Table 2 presents the data obtained on the heat of wetting of different soil separates expressed as calories per gram of material. In examining the table, it is noticed that the heat of wetting ceases entirely to be manifested for the separate 0.035 to 0.05 mm. in diameter for each soil except one. In soil separate 0.02 to 0.035 only four soils gave a heat of wetting above the considered experimental error. The Missouri soil shows a heat of wetting of 1.11 calories per gram in the 0.005 to 0.02 separates. The Florida soil separate gave a heat of wetting of 3.65 calories for the 0.005 to 0.02 mm. in diameter separate. In the 0.005 to 0.01 separate column, it is noticed at once that all the soil give a decided heat of wetting. In this separate the Florida and Missouri soils would be expected to give high heat of wettings as their clay separates give a high heat of wetting as well as their next finer separate size. One would conclude from the data presented that the heat of wetting ceases in the finer silt, but in some soils may be found in the coarser material also. The reason that these coarse materials of the soil tends to give the heat of wetting may be due to the presence of colloidal coatings of gels around the particles, the unremoved organic matter, or both.

This data tends to confirm Bouyoucos' (6) contentions, as stated previously, that the finer silt should be included in colloidal material as it tends to give the heat of wetting property similar to that of clay and the colloid separates,

TABLE 2 - HEAT OF WETTING OF SOIL SEPARATES EXPRESSED AS CALORIES
PER GRAM OF MATERIAL

Soils	Colloids	Clay	0.005-0.01	0.005-0.02	0.01-0.02	0.02-0.035	0.035-0.05	0.05-1a
Iron Mountain	6.56	2.44	0.65	0.65	0.45	0.25	0.00	0.00
Napanee	4.12	1.32	0.43	0.33	0.00	0.00	0.00	0.00
Ontonagon	6.50	2.57	0.43	0.22	0.20	0.00	0.00	0.00
Florida	15.42	8.43	----	3.85	----	----	0.00	0.00
Miami	5.61	3.16	0.42	0.34	----	0.15	0.00	0.00
Soil	----	----	0.91	----	0.62	0.36	0.00	0.00
Missouri	18.22	6.02	----	1.11	----	0.54	0.36	0.36
Gray Soil	5.45	----	----	0.32	----	0.00	0.00	0.00
Silt	-----	----	----	----	----	0.00	0.00	0.00
Brockston	5.32	1.40	0.74	----	0.17	0.00		
Isabella	2.13	1.40	0.44	----	0.22	0.00		
College Farm	5.63	2.83	0.32	----	0.26	0.00		
Hillsdale	6.48	4.05	0.85	----	0.40	0.00		
Chippewa		5.63	0.65	----	0.00	0.00		

The blanks indicate that the amount obtained was insufficient for a determination.

but in a different degree.

Table 3 presents the heat of wetting expressed as calories per gram of the mineral separates obtained. One will notice that all the ground mineral separates give a definite heat of wetting in the fine material or for particles smaller than 0.005 mm. in diameter. The pure zircon mineral gave a heat of wetting of over 0.3 calorie per gram in the 0.005 to 0.01 mm. separate. The biotite gave 0.35 calorie for the 0.02 to 0.035 mm. size but the heat of wetting of the remaining larger particles are within the experimental error and should not be considered significant. The hornblende shows a large heat of wetting even in the particles larger than 0.05 mm. in diameter. This heat of wetting, 1.26 calories per gram, cannot be disregarded as hornblende is a constituent of many soils and must play an important role in the heat of wetting of soils as a whole. Cushman (7) suggests that a colloidal film is formed on the surface of some minerals due to the formation of insoluble hydrolytic products of a colloidal nature. This may be the explanation for the hornblende and biotite minerals giving a higher heat of wetting than the other minerals shown in the table. From the results recorded it shows that different minerals exhibit the property in different degrees. This bears out the theory of Harkins and Ewing (10) that the quantity of the heat of wetting of a colloid is indicative of the nature of the material. The compression force is considered to be due to the attraction

force between the molecules of the liquid and the molecules of the absorbent. The heat evolved by wetting is due largely to the heat of the compression of absorbed liquid. Bouyoucos (5) has shown that the force with which the soils absorb and probably compress the water that produces the heat of wetting is very high.

In comparing tables 2 and 3, it is observed that the soil separates are larger in diameter than the mineral separates where the heat of wetting property ceased to be obtained. This would be expected as the soil particles are probably more porous and have an increased degree of activation, due to weathering processes. It is probable that the organic matter and gels had not been entirely removed or that some particles are coated with hydrolytic products of minerals similar to hornblende.

As was stated previously, all the organic matter is not removed from some soils by treatment with hydrogen peroxide. Since the organic matter particles have a lower density than the soil particles, we would expect to find most of the small size particles of organic matter in the fine separates. On the other hand, the large size organic matter particles would be reduced in size as the hydrogen peroxide would oxidize the easily oxidized part of the particle and leave the more resistant. In this manner, we would expect to find the most organic matter in the colloid and clay separates.

In order to ascertain if the separates after being

treated with hydrogen peroxide still contained organic matter, their nitrogen content was determined. Table 4 shows the per cent of nitrogen remaining in the soil separates as determined by the Gunning-Hibbard method for total nitrogen (16). All the nitrate nitrogen had previously been removed by repeated washings as determined by the colorimetric method. Table 4 shows that the fine materials contain the largest amount of total nitrogen. In every case, except the Missouri soil, the colloid separate contained more nitrogen than the clay separate. Mc Cool et al (11) showed that the fine soil material gives a high heat of wetting and has a higher nitrogen content than the coarse material. In the Missouri soil the clay had the highest nitrogen content. The 0.005 to 0.01 mm. in diameter separates also gives a fairly high percentage of nitrogen. It is very uncertain whether the organic matter present as determined by the total nitrogen determination causes the difference between the heat of wetting of a given size soil separate and the corresponding mineral separate, providing the number of each size particles were equal in both cases.

Since the soils are conglomerations of different minerals, it was thought that possibly hornblende, biotite and similar minerals in the soil would coat the other particles as well as themselves with a hydrolytic colloidal coating, and thus, increase their heat of wetting property. Several samples of different soil separates and mineral separates were treated for ten days with hornblende particles less than 0.005 mm.

TABLE 4 - PERCENTAGE OF NITROGEN IN THE SEPARATES AFTER TREATING WITH "SUPEROXYL"

Soils	Colloids	Clay	0.005-smaller	0.005-0.01	0.005-0.02	0.01-0.02	0.02-0.35
Missouri	0.13443	0.269	0.094	-----	0.015	0-----	0.011
Brookston	0.058	0.046	-----	0.016	-----	0.008	0.007
Isabella	0.313	0.063	-----	0.025	-----	0.007	0.012
College Farm	0.261	0.069	-----	0.019	-----	0.011	0.007
Hillisdale	0.077	0.090	-----	0.032	-----	0.022	0.014
Chippewa	-----	-----	0.069	0.027	-----	0.017	0.007

in diameter in suspension. After this time, the fine hornblende particles were separated by sedimentation and the heat of wetting determined. No increase in heat of wetting property could be detected after this treatment.

Since the fine material of the soils and minerals were present, it was decided to determine the effect of the heat of wetting property. Bouyoucos (4) has shown that at 800° C. the physical properties of soils, such as plasticity, heat of wetting, and unfree water, had completely disappeared. The separates at hand were heated at different temperatures up to 830° C. in an electric muffle furnace. Table 5 shows the gradual decrease in the heat of wetting property after exposure to higher temperature. All of the materials except limonite decreased gradually until at 830° C. only the kaolin, limonite and silica gave any heat of wetting. The limonite at 110° C. gave a heat of wetting of 1.12 calorie per gram, at 308° C. it gave 3.48 calories per gram and then gradually decreased until at 830° C. it gave 0.49 calorie per gram. This is interesting as there was an increase and then a decrease with the addition of heat. We know that there are many forms of the oxide of iron, the only difference in chemical composition being the water of hydration. In heating the limonite up to 305° C. some water may have been driven off or the material chemically changed. It is doubtful if by dehydrating it, we had decreased the size of the particles materially.

Therefore, the increase in heat of wetting per gram of material must have been due to a physical change of the surface caused by a chemical change through the ignition process. This rearrangement of molecules on the surface or change of surface due to the loss of water gave the material a greater attraction force for the water and thus increased the heat of wetting.

The moisture contained in each sample used here should be the same at the time the heat of wetting of the heated material was determined as it was originally. Each material was heated in the muffle furnace and then allowed to cool at room temperature before being placed in the heat of wetting tube and heated to 110° C. in the electric oven. It was thought that by this procedure the moisture content would be the same in each case as it was originally providing that no other changes had taken place during the heating to 830° C.

But why did the materials decrease in their heat of wetting property as is shown by the other minerals and soil separates in table 5?

Since one observes that the sharp edges of a jagged piece of glass will fuse long before the main piece of glass, it was thought that possibly the small particles had fused together. The substances were then placed in distilled water and separated by the application of Stokes' law to determine if the particles had increased in size. Table 6 shows the different size particles after heating to 830° C. In every case there was 89% or more of particles

TABLE 5 - CALORIES PER GRAM OF SOIL COLLOIDS AND THREE MINERALS
(0.005 to smaller) AT DIFFERENT TEMPERATURES
OF IGNITION

Material	110°C.	308° C.	405° C.	550° C.	850° C.
Napanea	3.88	2.75	2.13	1.41	0.00
Iron Mountain	6.91	6.41	5.51	4.58	0.00
Ontonagon	6.54	4.89	3.96	3.26	0.00
Kewanee	3.61	3.53	2.01	1.61	0.00
Florida	15.40	10.64	9.00	6.95	0.00
Kaolin	1.24	1.25	1.28	1.40	0.23
Limonite	1.12	3.48	3.06	2.65	0.49
Silica (ppt.)	5.90	5.25	4.91	4.10	0.90

TABLE 6 - PER CENT OF DIFFERENT SIZE PARTICLES AFTER HEATING
MATERIALS TO 830° CENTIGRADE

Soils	0.005 - smaller	0.005 - 0.02	0.02 - larger
Napanee	1.35	3.79	94.85
Iron Mountain	1.40	2.00	96.50
Ontonagon	3.31	3.68	92.74
Kewanee	6.60	1.19	92.62
Florida	0.60	3.40	96.00
Kaolin	1.24	2.60	97.24
Limonite	0.22	8.58	91.26
Silica	5.55	5.20	89.20

over 0.02 mm. in diameter left in each sample. This would indicate that the particles had fused together and as the particles become larger the heat of wetting decreased. The particles present after heating, as shown by separation, indicated that different minerals and soil particles have different fusion points. The silica has a high fusion temperature. Therefore, it lost its heat of wetting property gradually as the size increased, but at 830° C. the heat was not sufficient to form all particles that give no heat of wetting. In the case of soils, their composition is heterogeneous and contain some minerals that will fuse at a low temperature and will thus coalesce to the minerals that have not fused. By doing so, the size of the particles will increase proportional to the number of low or high fusion minerals present in the sample.

The Florida colloid and Kaolin mineral after heating and separating into their different size particles were reground by hand to try to restore their original heat of wetting. The Kaolin was increased to 0.82 calorie per gram while the Florida sample was increased from 0 calorie per gram to 1.53 calorie per gram. This is very small indeed when one remembers that the heat of wetting of the Florida material was 15.4 calories per gram before heating. Still one must consider that those particles were very small and a larger amount of energy must be expended to break down a particle 0.02 mm. in diameter to 0.005 mm. and smaller.

There is no doubt, of course, that the soil material

which gives rise to the phenomenon of heat of wetting is the colloidal matter and their degree of activation developed by the weathering processes. Therefore, one would expect to restore the original heat of wetting of the Florida soil on grinding by hand.

SUMMARY

In this paper it was proposed to show the size of particles from different soils at which the heat of wetting property ceased to be manifested. In order to secure suitable samples of different soil separates from soils for heat of wetting determinations, they were treated with dilute hydrochloric acid to remove the carbonates present and with hydrogen peroxide to remove the organic matter. The soils thus treated were separated by applying Stokes' law of sedimentation.

All the soil separates give a heat of wetting in the finer silt separate while a few possess this property in the coarse material, 0.02 to 0.35 mm. in diameter. It appears that the finer silt should be included in the colloidal content of the soil as they possess the property of heat of wetting.

Pure minerals finely ground and separated did not give such a high size limit as the soil at the place where the heat of wetting ceased. Hornblende and biotite gave a high heat of wetting even in the large silt particles.

The amount of nitrogen in the soil separates varies. Larger amounts of nitrogen and, therefore, organic matter were found in the colloidal material of the soil. This organic matter may account for part of the heat of wetting above that of the same size material separates.

Soil colloidal materials and finely ground minerals were ignited to 830° C. to determine why the heat of wetting

was destroyed. After heating to 830° C. and separating into the different size separates, it was found that in every case, the percentage of 0.02 mm. in diameter particles was over eighty-nine.

One soil colloid and one mineral after ignition were reground and the heat of wetting was partly restored.

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