

THE MICROSCOPIC ESTIMATION OF COLLOIDS IN SOIL SEPARATES¹

By WILLIAM H. FRY

Scientist in Soil Laboratory Investigations, Bureau of Soils, U. S. Department of Agriculture

In a recent work upon soil colloids,² in which a description of the soil materials classified as colloids was given, it became desirable to compare the values obtained by an adsorption method for determining the colloidal content of soils with values obtained by some method independent of adsorption phenomena. The most satisfactory method for this purpose would be a mechanical separation of colloidal from noncolloidal soil materials. But it has been shown in previous publications^{2,3} that a mechanical analysis probably does not effect a complete separation. However, it seemed possible that colloidal matter not separated from the soil by mechanical analysis might be estimated by microscopical means. Then the sum of the colloid extracted and that estimated microscopically would give a value for the total colloid in the soil by methods quite independent of adsorption.

For the purpose of separating the colloids from the coarser particles of the soils the samples were repeatedly treated by agitation with distilled water containing a trace of ammonia, rubbed with a rubber pestle, and the supernatant liquid decanted and centrifuged. From 40 to 60 such treatments yielded practically all of the colloid extractable by this method. For convenience of manipulation the soils were divided, during the washing and rubbing process, into the colloidal fraction made up of particles less than 0.001 mm. in diameter, a fine fraction made up of particles ranging approximately from 0.001 to 0.050 mm. in diameter, and a coarse fraction consisting of particles larger than about 0.050 mm.²

The adsorptive capacities of these fine and coarse residues indicated that they contained colloidal material.² Observations of the residues with the ultramicroscope showed that the mineral particles had been fairly well cleaned of adhering colloidal matter by the washing and rubbing, and that the colloidal material remaining in these fractions was in the form of lumps or aggregates made up, at least superficially, of very large numbers of particles less than 0.001 mm. diameter. But since the light coming to the eye from the ultramicroscope is reflected from the surfaces of the particles under examination it was possible that the colloidal aggregates thus observed were simply mineral grains completely coated with colloids or that, at least, the colloidal aggregates

¹ Accepted for publication Apr. 7, 1923.

² ANDERSON, M. S., FRY, W. H., GILB, P. L., MIDDLETON, H. E., and ROBINSON, W. O. ABSORPTION BY COLLOIDAL AND NONCOLLOIDAL SOIL CONSTITUENTS. U. S. Dept. Agr. Bul. 1122, 20 p. 1922. Literature cited, p. 19-20.

³ DAVIS, R. O. E. THE INTERPRETATION OF MECHANICAL ANALYSIS OF SOILS AS AFFECTED BY SOIL COLLOIDS. *7th Jour. Amer. Soc. Agron.*, v. 14, p. 293-298. 1922. Literature cited, p. 298.

inclosed minute mineral particles. The clean appearance of the obviously mineral particles was, of course, a strong indication that such was not the case, but the element of doubt remained nevertheless.

Under the petrographic microscope in transmitted light these colloidal aggregates were found to be almost universally transparent and, in the rare cases where this was not so, to be highly translucent. Therefore any mineral grain included within the colloidal aggregate would be readily visible provided that either its color or index of refraction was different from the color or index of the colloid. Observation of birefringence between crossed nicol prisms would, of course, readily differentiate between minerals and colloids regardless of similarity of color or refractive index. Such observation, however, showed that practically all of the colloidal aggregates were free from mineral inclusions. Presumably, aggregates containing such had been broken up by the repeated rubbing and washing process.

Therefore, since the residues consist of easily determinable minerals and colloidal aggregates, one readily distinguishable from the other, a microscopic estimation of the relative quantity of each in a given sample was easily obtainable. Owing to the extreme variation of size of particles a straight count would have been subject to large errors. Therefore, the estimation was made by means of a checker-work eyepiece micrometer, relative space occupied by the minerals and colloids being the basis of the calculation. It is believed, on the basis of results obtained with synthetic samples, that errors due to the varying thickness of the particles, both minerals and aggregated colloids, are fairly well balanced and do not appreciably affect the results. Quartz is the predominant mineral in the fractions, and since the specific gravity of this mineral (2.66) and that of the colloids (2.53-2.68) extracted from the soils are very near together, it was believed that differences in specific gravity would not give rise to any serious error.

In order to test this method of microscopic estimation, four synthetic samples were made up, each containing a known amount of colloidal material. Air dried Marshall soil colloid, which had been extracted mechanically and graded by means of the supercentrifuge, was mixed with quartz in the proportion of 1.2 gm. colloid to 0.8 gm. quartz and 0.8 gm. colloid to 1.2 gm. quartz. The first of these mixtures was ground dry to pass a 200-mesh sieve. In order to facilitate any tendency of the colloid to form coatings on the mineral particles, the second sample was ground wet, dried and subsequently rubbed lightly with a pestle to pass a 200-mesh sieve.

Since quartz is practically without cleavage, the particles formed by grinding have a tendency to assume shapes in which the dimensions vary little in different directions within the same particle, although the dimensions of different particles may vary widely. This statement also holds good for the colloidal aggregates. Such a similarity of fracture facilitates a microscopic estimation of the relative quantities of the different constituents present, and therefore does not afford a very difficult test of the method. But, since quartz is the predominant mineral constituent of soils, the results obtained with quartz are probably very near those obtained on the soil separates themselves.

For the purpose of testing the method under more unfavorable conditions, Orangeburg subsoil colloid, extracted mechanically and graded by the supercentrifuge, was mixed with hornblende which has a tendency

to cleave into oblong particles with the greater dimensions several times the magnitude of the smaller, thus presenting a widely different shape from the colloidal aggregates with which it was to be compared. Two samples were prepared consisting of 0.3 gm. colloid to 1.7 gm. hornblende and 0.4 gm. colloid to 1.6 gm. hornblende. The first of these was mixed dry and the second wet, as with the Marshall colloid-quartz samples.

The results of the microscopic examination of these samples are given in Table I.

TABLE I.—*Microscopic estimation of colloids in synthetic samples*

Sample.	Colloid present.	Colloid observed.
	<i>Per cent.</i>	<i>Per cent.</i>
Marshall colloid and quartz, mixed dry.....	60	57
Marshall colloid and quartz, mixed wet.....	40	45
Orangeburg colloid and hornblende, mixed dry.....	15	12
Orangeburg colloid and hornblende, mixed wet.....	20	23

These results showed that the colloid in the fine and coarse residues of the soils could be estimated with fair accuracy by this microscopic method.

Microscopic examinations were therefore made of the fine and coarse residues of eight soils which had been subjected to the repeated washing and rubbing process. The results are given in Table II. The quantity of the unextracted colloid in the fine and coarse residues, as determined by this microscopic method, are given in column 3. The additional data in Table II are given for the purpose of showing the relative amounts of "extractable" and "unextractable" colloids in the soils.

From Table II it will be seen that from 9.4 to 42.4 per cent of the soil was made up of colloids extractable by the methods employed. The microscopic examination of the residues shows that from 25 to 97 per cent of the fine residues and 2 to 25 per cent of the coarse residues were colloidal aggregates not extractable by the methods employed.

These results throw considerable doubt upon the results of several investigators who have determined the colloidal contents of soils and clays by purely mechanical methods. Hissink⁴ and Sven Odén,⁵ for example, have dispersed colloids in soils by very elaborate washing and rubbing processes. It is of course possible that they may have effected complete dispersion by their methods, but apparently no investigations were undertaken to ascertain whether, or to what extent, aggregates of undispersed colloids remained in their soil suspensions. Williams,⁶ in the course of a very elaborate and painstaking separation of colloids from soils by washing, rubbing and boiling, did examine his residues microscopically. Details of his microscopic method are not given, but it is apparent that he used an ordinary chemical microscope and mounted his residues either in air or water. Owing to the wide differences in refractive indices between these mounting media and the ordinary soil minerals, scarcely more than surface phenomena could be

⁴ HISSINK, D. J. DIE METHODE DER MECHANISCHEN BODENANALYSE. *In* Internat. Mitt. Bodenkunde, Bd. 11, p. 1-11. 1921.

⁵ ODÉN, SVEN. ÜBER DIE VORBEHANDLUNG DER BODENPROBEN ZUR MECHANISCHEN ANALYSE. *In* Bul. Geological Inst., Univ. Upsala, v. 16, p. 125-134. 1918-19. Bibliographical footnotes.

⁶ WILLIAMS, W. R. UNTERSUCHUNG ÜBER DIE MECHANISCHE BODENANALYSE. *In* Forsch. Geb. Agr.-Phys. (Wollny), Bd. 18, p. 225-350. 1895. Bibliographical footnotes.

observed under these conditions; and consequently the distinction between lumps of aggregated colloids and soil minerals would reduce itself purely to a question of personal judgment based on appearance. Colloidal aggregates might bear very close resemblances to corroded mineral particles and ferruginous or rutilated quartz, for example. Even with a completely equipped petrographic microscope careful cognizance must be taken of the pseudo-similarities of the optics as well as the resemblances of some crystalline and amorphous materials, such as the similarity between colloids subjected to strain and the matted structure of such minerals as chlorite.

TABLE II.—*Microscopic estimation of unextracted colloids in fine and coarse residues of soils*

Sample.	Quantity of fractions separated mechanically, as part of whole soil.	Quantity of unextracted colloid in residues determined microscopically, as part of residue.	Unextracted colloid in combined fine and coarse residues, as part of whole soil.	Unextracted colloid, as part of total colloidal material in soil.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Cecil clay loam, soil:				
Colloid extracted.....	9.4			
Residue finer particles.....	7.6	74		
Residue coarser particles.....	83.8	4	9.0	48.9
Cecil clay loam, soil:				
Colloid extracted.....	9.4			
Residue finer particles.....	10.4	55		
Residue coarse particles.....	78.3	4	8.9	48.6
Huntington loam, soil:				
Colloid extracted.....	10.3			
Residue finer particles.....	21.9	48		
Residue coarse particles.....	64.0	8	15.6	60.2
Huntington loam, subsoil:				
Colloid extracted.....	13.3			
Residue finer particles.....	19.4	25		
Residue coarse particles.....	63.3	10	11.2	45.7
Sassafras silt loam, subsoil:				
Colloid extracted.....	14.4			
Residue finer particles.....	20.6	38		
Residue coarse particles.....	61.9	2	9.1	38.7
Sharkey clay, soil:				
Colloid extracted.....	42.4			
Residue fine and coarse particles combined.....	53.1	42	22.3	34.5
Sharkey clay, soil:				
Colloid extracted.....	31.5			
Residue finer particles.....	38.9	52		
Residue coarse particles.....	31.8	25	28.2	47.2
Vega Baja clay loam, soil:				
Colloid extracted.....	30.5			
Residue fine particles.....	38.6	97		
Residue coarse particles.....	31.8	14	41.9	57.9

The calculations given in column 4 of Table II show that from 8.9 to 41.9 per cent of the whole soil was composed of colloidal aggregates not extractable by the methods used. It should be mentioned, however, that the Vega Baja soil which showed a total colloidal content of 72.4

per cent is an exceptional soil. Usually the quantity of colloid present is much lower. Basing the calculations on the colloidal matter alone, it is seen from the table that 34.5 to 60.2 per cent of the total colloids in the soils were not extracted by the methods used. Although this method of repeated rubbing and washing for the extraction of colloids from soils was carried out as carefully and thoroughly as possible, and although we are confident that no more than traces of colloidal material could have been extracted by further washing and rubbing, it by no means follows that other more efficient methods might not be devised.

As practiced in this bureau, microscopic estimation of the amount of colloidal aggregates in the fine and coarse residue is, at best, a long and tedious process. In order to insure fair sampling of the sample under study several mounts must be examined, and several areal counts should be made on each mount. Experience showed that a total of 10 to 12 counts on about four slides gave averages very comparable with those obtained by triple that number of counts on as many more mounts. Nevertheless when only 12 counts are made, the method is too long for routine application.

Unfortunately, the preliminary washing and rubbing are essential in order to remove coatings of the adhering extractable colloid. These coatings are rarely thick enough to interfere with the determination of the mineral; but the microscopic estimation of the quantity of colloid in the coatings would be extremely difficult and inexact. This necessity for washing the soils adds tremendously to the time consumed. Nevertheless, the method is of value in special studies.

SUMMARY

(1) A microscopic method for estimating the quantity of colloidal soil aggregates in soil separates is given.

(2) This method was applied to the residues left after extracting, by repeated rubbing and washing, all colloid possible from eight samples of soil. The results showed that from 34.5 to 60.2 per cent of the total colloidal material in the soil was in the form of colloidal aggregates not extractable by washing and rubbing.

ADDITIONAL COPIES

OF THIS PUBLICATION MAY BE PROCURED FROM
THE SUPERINTENDENT OF DOCUMENTS
GOVERNMENT PRINTING OFFICE
WASHINGTON, D. C.

AT

10 CENTS PER COPY

SUBSCRIPTION PRICE, \$4.00 PER YEAR

PURCHASER AGREES NOT TO RESELL OR DISTRIBUTE THIS
COPY FOR PROFIT.—PUB. RES. 57, APPROVED MAY 11, 1922

△

