Aggregation and Aggregate Stability in Forest and Range Soils

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ABSTRACT

A simple method for measuring aggregation and aggregate stability across soils of widely varying structure and texture is presented. The method uses standard techniques of particle-size analysis to measure yield of fine particles ($<26-\mu m$ estimated spherical diam.) after three types of dispersion treatments: shaking in water or in sodium oxalate (Ox), and sonication in sodium pyrophosphate (PPi). PPi sonication, which provided maximum dispersion, served as our benchmark; it was substantially more effective than the commonly used stirring in hexametaphosphate. Aggregation was then calculated as yield of fines after PPi sonication minus yield of fines after the water shake, the minimum dispersion treatment. Aggregate stability was measured by comparing yield of fines after the Ox shake, the mild dispersion treatment, with that after the water shake; aggregate instability was then calculated. The five soils studied varied markedly in both aggregation and aggregate stability, suggesting that mechanisms of aggregate stabilization differ markedly across soil types. Despite these differences, our method of quantifying mild dispersion by shaking with Ox, which should have negligible effect on subsequent microbiological studies, relative to complete dispersion by PPi sonication should be consistent and applicable over a reasonably wide range of soil types.

Additional Index Words: Dispersion, Sodium oxalate, Ultrasound. Intersite comparison.

THE PHYSICAL STRUCTURE of a soil reflects the na-L ture of its component primary grains and the extent to which they have aggregated into larger particles. Soil physical structure determines pore-size distribution, which in turn affects water flow and erosion potential (White, 1985), microbial and faunal behavior (Edwards and Bremner, 1967; Elliott et al., 1980; Van Veen et al., 1984), and organic matter dynamics (Campbell and Souster, 1982; Tisdall and Oades, 1982; Schimel et al., 1985; Schimel, 1986).

Soil particles aggregate as a result of physical binding by roots and hyphae, "gluing" by bacterially produced polysaccharides, and physical-chemical interactions between silicate clay surfaces and functional groups of partially decomposed organic matter (Aspiras et al., 1971; Aringhieri and Sequi, 1978; Tisdall and Oades, 1979; Sorensen, 1981). Aggregation may affect nutrient turnover by controlling microbial predation (Van Veen et al., 1984) and by protecting organic matter from microbial degradation (Young and Spycher, 1979; McGill et al., 1981; Van Veen and Paul, 1981; Voroney et al., 1981). Although various methods have been used to compare aggregation and aggregate stability among small numbers of soil types sometimes varying in texture or management history (e.g., Christensen, 1985; Churchman and Tate, 1987),

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we know of none that have been applied across a wide range of soil types.

In the work reported here, we modified existing particle-size analysis methods to develop a simple method for determining aggregation and aggregate stability that could be used to compare soils of widely differing textures. We then applied the method to six North and Central American soils that varied markedly in physical structure. Our long-term objective was to develop a method of altering soil physical structure with a mild dispersive process that might actually operate in soil so that we could later experimentally study how physical structure might control nitrogen (N) mineralization.

MATERIALS AND METHODS

Soil samples were collected from the Al horizon (0-15 cm) of each site (Table 1) and stored field-moist at 4 °C in our laboratory in Oregon until needed. The soils spanned a range of mineralogies, textures, and carbon (C) contents (Table 2). All procedures were replicated three times for each soil sample. Statistical analyses were performed with SYSTAT (Wilkinson, 1986). Tukey's HSD test was used to determine significant differences between means at the 95% confidence interval. Throughout this paper, aggregation is defined as the proportion (g per kg) of primary grains bound into larger particles that are stable when shaken in water. Aggregate stability is defined as the resistance of aggregates to a given dispersive process.

Wet Sieving

All soils were first wet sieved into two fractions. Ten grams of each soil were placed in a 7.5-cm sieve (50-µm screen size) ground to fit snugly into the top of a 600-mL beaker. Each beaker was filled with 400-mL of deionized water (dIH₂O) so that the sieve screen was 1 cm below the surface. and shaken on a reciprocating shaker at 120 excursions per min for 30 min. The sieve and material remaining on it were reimmersed in fresh dIH₂O and shaken for an additional 15 min. For each sample and replicate, suspensions of material passing through the sieve were combined and centrifuged at 4080 g for 20 min. The < 50- μ m fractions were resuspended in 10 mL of dIH₂O and stored cold (4 °C) until needed. After plant debris was removed by hand, the $>50-\mu m$ fractions, which remained on the sieve surface, were transferred to a Buchner funnel and aspirated for 10 min to remove excess water.

Additional 10-g subsamples of each soil were wet sieved (50 µm), dried at 100 °C for 48 h, and weighed so that moisture content of both wet-sieve fractions, as well as the proportion of whole soil accounted for by each, could be determined.

Dispersion Treatments

The two wet-sieve fractions of each soil sample received three treatments: (i) shaking in water, to simulate a minimum dispersive force, (ii) shaking in a sodium oxalate (Ox) solution (0.15 M, pH 7.5), to simulate a mild dispersive force, and (iii) sonication [5 min at 100 W with a Braun-Sonic 2000 (Braun Scientific, West Germany)] in a sodium pyrophosphate (PPi) solution (0.075 M, pH 7.5), to simulate a maximum dispersive force (Gee and Bauder, 1986). As

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Site	Location	Soil type	Vegetation [†]
Waldo	Florida, USA	Ultic Haplaquod	Slash pine
La Selva	Costa Rica, Central America	Oxic Humitropept‡	Abandoned pasture
Konza unburned	Kansas, USA	Pachic Argiustoll	Tallgrass prairie
Konza burned	Kansas, USA	Pachic Argiustoll	Tallgrass prairie
Cascade Head	Oregon, USA	Typic Dystrandept	Douglas fir, red alder
H.J. Andrews	Oregon, USA	Andic Haplumbrept	Burned clearcut formerly old- growth Douglas fir/western hem lock

† Slash pine, Pinus elliotii Engelm.; Douglas fir, Pseudotsuga menziesii (Mirb.) Franco; red alder, Alnus rubra Bong.; western hemlock, Tsuga heterophylla (Raf.) Sarg.

‡ Upper terrace (old alluvium) listed as a Typic Hydrandept by Johnson et al. (1979) and Sollins et al. (1984).

with other methods that use sonication to disperse aggregates (e.g., Christensen, 1985), we assumed that complete dispersion was achieved.

Soil suspensions ($<50 \ \mu m$) or particles ($>50 \ \mu m$) were placed in a 125-mL erlenmeyer flask with 50 mL of dispersant (water or Ox) and shaken at 150 excursions per minute for 30 min. For the <50- μ m fractions, we used a well-mixed 1-mL aliquot of suspension containing 0.01 to 0.5 g of particles, depending on the soil type, for shaking and sonication. For the >50-µm fractions, we used the same quantities (0.01-0.5 g) of aspirated particles for both shakes but no more than 0.25 g for PPi sonication because it yielded more fines. Flasks were kept submerged in ice water during sonication to minimize temperature increases.

We also used two standard dispersion techniques, (i) sonication in water (5 min, 100 W) (Christensen, 1985) and (ii) stirring in a sodium hexametaphosphate (HMP) solution (e.g., Edwards and Bremner, 1967; Watson and Parsons, 1974; Turchenek and Oades, 1979), to compare results with those of previous studies. After stirring in HMP, particle sizes were measured by the hydrometer method. After all other dispersion treatments, particle sizes were measured with the modified pipet method described below.

Particle-size Distribution

The dispersed suspensions were transferred into 40- by 100-mm glass jars and allowed to settle at room temperature (25 °C). Two mL of suspension were collected 2 cm below the suspension surface at times when particles with equivalent spherical diameters (ESD) of >242, 242 to 140, 140 to 100, 100 to 32, 32 to 26, and $<26 \mu m$ would have settled from solution; collected solutions were then diluted with 2 mL of dIH₂O. The concentration of suspended particles was assumed to be proportional to light absorbence at 420 nm (Williams et al., 1966). For the coarser fractions (>26- μ m ESD), we calculated change in absorbence over each time interval, then divided absorbance change by initial sample dry mass to convert change in light absorbence to proportion of soil added. For the finer fractions ($<26-\mu m ESD$), light was absorbed by both suspended particles and dissolved organic matter, and we assumed the same specific absorbence for both. Some of the $>50-\mu m$ fractions had to be diluted further after sonication to bring suspensions within an acceptable absorbence range. Trials indicated that dilution had little effect on specific absorbence. Although the specific absorbence of particles in different fractions and dissolved organic matter may vary because of surface-area differences, the error involved is small enough that the method is useful for comparisons (e.g., Williams et al., 1966).

Table 2. Selected properties of soils tested.

	Soil					
Property	Waldo	La Selva	Konza†	Cascade Head	H.J. Andrews	
Silt, g kg ⁻¹ ‡	52	176	601	451	315	
Clay, g kg ⁻¹ ‡	33	734	202	137	222	
Clay minerals§	Qz	Kl	Qz	Qz	Qz	
	Kl	Gt	Kl	Vm	Kl	
		Gb	Ks	Sm	Cb	
					Ct	
					Gb	
1.5 MPa moisture, kg kg ⁻¹						
Air dry	0.06	0.32	0.18	0.28	0.18	
Field moist	0.06	0.56	0.32	0.46	0.29	
$pH (H_2O)$	3.6	4.2	6.4	4.3	6.1	
C, g kg ⁻¹ #	27	39	33	142	51	

† Unburned only

‡ Bouyoucous hydrometer method after stirring in sodium hexametaphosphate. § Clay minerals were determined by x-ray diffraction. Abbreviations: Qz

= quartz; Kl = kaolinite; Ks = K-spar; Vm = vermiculite; Sm = smec-tite; Cb = cristobalite; Ct = chlorite; Gb = gibbsite; and Gt = goethite.

H_0 O/soil = 1:1. # Carbon determined with LECO combustion analyzer (Laboratory Equipment Corp. St. Joseph, MI).

Except for the Konza soils, there were no significant differences in the proportion of aggregates remaining in any individual size class >26-µm ESD after shaking in Ox (data not reported) and in water. However, in many cases the combined yield of fines from dispersion of aggregates in all size classes >26- μ m ESD differed significantly with treatment; therefore, the increase in fines ($<26-\mu m$ ESD), rather than the decrease in coarse particles, was used as an index of dispersion. The yield of fines (g per kg, wet-sieve fraction) was determined for each soil type after each treatment. To calculate aggregation, we subtracted the proportion of fines after the water shake from that after PPi sonication. To calculate aggregate instability (proportion of aggregates dispersed by oxalate treatment), we subtracted the proportion of fines after the water shake from that after the Ox shake, then divided by the aggregation value and multiplied by 100.

RESULTS AND DISCUSSION

Although sodium pyrophosphate is used widely to disperse soil, the resulting phosphorus fertilization is unacceptable when microbiological incubations will follow. We therefore chose oxalate, which is commonly used to extract "mineral" Fe and Al (i.e., not in association with soil organic matter), as the mild dispersant. Oxalate, an energetically poor substrate, should have relatively little effect on the outcome of subsequent microbiological studies; its heat of combustion is only 19% that of acetate. Moreover, we found that oxalate and PPi (0.15 M and 0.075 M, respectively) were equally effective soil dispersants if used at the same concentration (0.3 N) and pH (7.5)(data not shown).

Table 3. Amount (±SE) of each soil sample in two wet-sieve fractions: n = 3.

Soil	< 50 - μ m fraction	$>$ 50- μ m fraction	
	g kg ⁻¹		
Waldo	99 (2)	900 (7)	
La Selva	166 (10)	832 (13)	
Konza unburned	538 (59)	468 (57)	
Konza burned	684 (14)	324 (7)	
Cascade Head	87 (2)	912 (1)	
H.J. Andrews	107 (2)	893 (2)	

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DRY

%

All soils except Konza yielded mainly water-stable particles > 50 μ m in diameter (Table 3). This was true even for the La Selva soil, which had a clay content of 734 g kg⁻¹, as determined by HMP dispersion (Table 2). However, the composition of these particles differed among soil types (Fig. 1). Only the Konza soils had substantial amounts of aggregates <100- μ m ESD (170 and 250 g kg⁻¹ vs. 10-40 g kg⁻¹ for the other soils); the aggregates of the other four soils were almost exclusively >240-µm ESD (Fig. 1). Moreover, the La Selva and Cascade Head soils consisted primarily $(>700 \text{ g kg}^{-1})$ of aggregates, whereas the Waldo soil was almost exclusively primary grains (~930 g kg⁻¹); the Konza and H.J. Andrews soils were an intermediate mixture of aggregates and primary grains (\sim 350-600 g kg⁻¹ aggregates; Fig. 1, Table 4).

Each soil contained a different proportion of aggregates resistant to dispersion by the Ox shake (Table 4). For example, although the La Selva and Cascade Head soils contained similar amounts of aggregates (Fig. 1, Table 4), the Cascade Head aggregates were more readily dispersed by oxalate. Interestingly, the two Konza soils differed in stability, as well as amount, of aggregates even though the sites differed only by fire history. This observation suggests that our method of characterizing aggregates may be useful in monitoring effects of perturbation on soil physical properties.

Aggregate stability differed within each wet-sieve fraction among most of the soils and between the two fractions for three of the six soils (Table 5). For example, the Ox shake dispersed more $<50-\mu m$ aggregates than did the water shake in the Waldo and Konza unburned soils and more $>50-\mu m$ aggregates than did the water shake in the La Selva and Cascade Head soils. Dispersion after both water and Ox shakes did not significantly differ in the Konza burned and H.J. Andrews soils in either wet-sieve fraction. For all soils and fractions, PPi sonication significantly increased the amount of material vielded as fines (range of increase 330-490 g kg⁻¹ of <50- μ m fraction, 820-990 g kg⁻¹ of >50- μ m fraction).

Aggregation and aggregation mechanisms probably differ among soil types and particle size classes from a given soil. Tisdall and Oades (1982) present evi-

Table 4. Comparison of aggregation and aggregate instability across samples of six soils.

	Agg	Aggregate		
Soil	After water shake	ter water shake After oxalate shake†		
	g	kg-1		
Waldo	63e§	37e	41.3	
La Selva	747c	715c	4.3	
Konza unburned	599b	536b	10.5	
Konza burned	464a	441a	5.0	
Cascade Head	707c	550b	22.2	
H.J. Andrews	355d	327d	7.9	

 $\dagger \ensuremath{\operatorname{Relative}}$ to sodium pyrophosphate sonication, the amount of fines not dispersed by shaking in oxalate.

‡ [(Aggregation after water shake - aggregation after oxalate shake). aggregation after water shake)] \times 100.

§ Within a column, means followed by the same lowercase letter are not significantly different (P = 0.05, Tukey's HSD test, n = 3). We assumed that disruption of aggregates in all size classes results in an increase of primary grains in the fine ($< 26 \mu m$ estimated spherical diameter) fraction.

. 4





Fig. 1. Percentage of each soil composed of primary grains and aggregates after sonication in sodium pyrophosphate (PPi) solution, which maximizes dispersion.

dence for a hierarchy of aggregation mechanisms operating at different size classes within a single soil type (Calcic Rhodoxeralf). They stress, however, that their model should not be expected to hold for other soil

Soil by	Yield of fines, by treatment				
wet-sieve fraction	Water shake	Ox shake	PPi sonication	Aggrega- tion‡	Aggregate instability§
	g	kg ⁻¹ , wet-	sieve fraction	n	
Waldo					
$< 50 \ \mu m$	601a	745b	906c	304.7	47.26
$> 50 \ \mu m$	8a	21a	44b	36.0	36.11
La Selva					
$< 50 \ \mu m$	514a	613a	941b	426.2	23.23
$> 50 \ \mu m$	11a	30b	823c	812.4	2.34
Konza unburned					
$< 50 \ \mu m$	473a	566b	870c	396.9	23.43
$> 50 \ \mu m$	34a	60a	858b	823.8	3.16
Konza burned					
$< 50 \ \mu m$	474a	498a	863b	391.2	6.13A
> 50 µm	57a	75a	667b	609.5	2.95A
Cascade Head					
$< 50 \ \mu m$	451a	561a	882b	430.9	25.53B
$> 50 \ \mu m$	44a	205b	773c	729.1	22.08B
H.J. Andrews					
$< 50 \ \mu m$	424a	476a	799b	367.3	14.16C
> 50 µm	23a	47a	368b	345.8	6.94C

Table 5. Yield of fines (<26- μ m estimated spherical diameter) after the three dispersion treatments [shaking in water and in oxalate (Ox); sonication in sodium pyrophosphate (PPi)] and the resulting aggregation and aggregate instability.[†]

† For any soil and wet-sieve fraction (row), means followed by the same lowercase letter are not significantly different (P = 0.05, Tukey's HSD test, n = 3). For wet-sieve fractions of any soil (column), means followed by the same uppercase letter are not significantly different (P = 0.05, Tukey's HSD test, n = 3). We assume that disruption of aggregates in all size classes results in an increase of primary grains in the fine (<26um estimated spherical diameter) fraction

Yield of fines after PPi sonication less yield of fines after shaking in water. § [(Yield of fines after Ox shake - yield of fines after water shake)/aggregation $\times 100$



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Fig. 2. Relationships between aggregation (water stable and oxalate stable) and yield of fines (<26-µm estimated spherical diameter) after (a) stirring in hexametaphosphate (HMP) solution and (b) sodium pyrophosphate (PPi) sonication.

types because the same mechanisms could not be expected to operate to an equal degree in all soils. Our data support their warning. The results in Tables 4 and 5 suggest that aggregates are stabilized by different mechanisms in at least some of the different soils that we studied, as well as in different size classes within any one soil type.

A reasonably complete dispersion is needed to measure aggregation across a wide range of soil types and to provide a benchmark against which to compare mild treatments when measuring aggregate stability. Strictly chemical dispersion treatments seem unacceptable, although water sonication is used routinely (Williams et al., 1966; Edwards and Bremner, 1967; Watson and Parsons, 1974; Turchenek and Oades, 1979). Our results show that neither oxalate shake without sonication (Table 5) nor PPi shake without sonication (data not shown) produced complete dispersion. The overall pattern is still unclear, however; Christensen (1985), for example, found no difference in clay content after water sonication and stirring in HMP, whereas we found PPi sonication to be significantly more effective than water sonication in dispersing water-stable aggregates in half of our soils (Table 6). Once again, results for one soil type may not apply to others. Perhaps the clearest evidence in favor of sonication in PPi is that it gave a far superior correlation with percent of fines across all our soils than did stirring in HMP (Fig. 2)—a correlation that is to be expected if aggregation in a soil depends on the amount of fine particles available for binding.

We hoped to develop a way to experimentally alter soil structure to examine effects of that structure on microbiological processes such as N mineralization. Shaking in water is too drastic-although dispersion is minimal, the resulting sorting and reordering of soil particles grossly alter soil architecture. Inorganic chemicals such as PPi and HMP are unacceptable beTable 6. Amount $(\pm SE)$ of soil in fine fraction (<26- μ m estimated spherical diameter) after sonication in water and sodium pyrophosphate (PPi).

Soil	Water sonication	PPi sonication		
	g kg-1			
Waldo	122a (2)†	129a (2)		
La Selva	693a (3)	841b (12)		
Konza unburned	823a (12)	869b (3)		
Konza burned	781a (17)	804a (7)		
Cascade Head	545a (9)	783b (5)		
H.J. Andrews	384a (11)	414a (15)		

[†] For any soil sample (row), means followed by the same lowercase letter are not significantly different (P = 0.05, Tukey's HSD test, n = 3).

cause of P fertilization. We propose that our method of quantifying mild dispersion in Ox relative to complete dispersion by PPi sonication will be consistent and applicable over a reasonably wide range of soil types.

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