

CARBON AND NITROGEN POOLS IN SOIL AGGREGATES SEPARATED BY DRY AND WET SIEVING METHODS

Upendra M. Sainju

Soil aggregation influences conservation and mineralization of carbon (C) and nitrogen (N), but aggregate separation method may influence levels of aggregate size distribution and quantification of C and N pools. Aggregate size distribution and soil organic C, soil total N, particulate organic C and N, microbial biomass C and N, potential C and N mineralization, $\text{NH}_4\text{-N}$, and $\text{NO}_3\text{-N}$ concentrations in aggregates separated by dry and wet sieving methods were compared. The potential C and N mineralization, and microbial biomass C and N are considered as active pools, soil organic C and soil total N as slow pools, particulate organic C and N as intermediate pools, and $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ as available pools. Aggregate separation was made in soil samples from 0- to 5- and 5- to 20-cm depths with various properties and cropping systems in the northern Great Plains. Aggregate amount was higher in dry than in wet sieving in the 4.75- to 2.00-mm size class, but the amount was higher in wet than in dry sieving in the 2.00- to 0.25-mm class in all sites, except in Sidney. In cultivated soil, no definite trends in C pools in aggregates were observed between sieving methods. In no-till grassland soil, C pools were higher in dry than in wet sieving in the less than 0.25-mm fractions, but the trend reversed in the greater than 2.00-mm fractions. In all fractions, active and available N pools were 2- to 30-fold higher in dry than in wet sieving probably because of loss of water-soluble N during wet sieving. Both C and N pools, except active C pools, in aggregates were usually higher in the less than 0.25-mm than in the other fractions, regardless of sieving methods. Aggregate size distribution and C and N pools determined by dry and wet sieving were well correlated, except for active N pools. Dry sieving of moist soil (water content around 25% field capacity) can be used as a rapid and reliable method of separating soil aggregates for determining C and N pools compared with wet sieving, which reduces microbial activities and N mineralization because of the destruction of physical habitat of microbial communities in aggregates and excludes water-soluble C and N pools. (Soil Science 2006;171:937-949)

Key words: Aggregate separation method, soil aggregates, carbon pools, nitrogen pools.

SOIL aggregation has potential benefits on soil moisture status, nutrient dynamics, tillage maintenance, and erosion reduction (Oades, 1984; Kemper and Rosenau, 1986). The levels

of aggregate size distribution and associated carbon (C) and nitrogen (N) pools, however, are influenced by the aggregate separation method (Kemper and Rosenau, 1986; Seech and Beauchamp, 1988; Whalen and Chang, 2002). The wet sieving method is widely used to determine size distribution and stability of aggregates caused by raindrop impact on dry soil causing slaking and surface crusting (Elliott, 1986; Kemper and Rosenau, 1986; Cambardella and Elliott, 1993). The disadvantages of this method

U. S. Department of Agriculture—Agricultural Research Service-NPARRL, 1500 North Central Avenue, Sidney, MT. Dr. Sainju is corresponding author. E-mail: usainju@sidney.ars.usda.gov

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are (i) rupture of weak aggregates in water during sieving caused by ion hydration, sudden release of internal air pressure, osmotic swelling forces, or solubility of binding agents in water (Kemper and Rosenau, 1986; Perfect et al., 1990; Gale et al., 2000); (ii) increased disruption of physical habitat of microbial communities (Mendes et al., 1999; Schutter and Dick, 2002); and (iii) underestimation of C and N pools in aggregates because of exclusion of water-soluble C and N (Seech and Beauchamp, 1988). As a result, dry sieving of moist soil for determining microbial biomass and activities that include water-soluble C and N in aggregates is receiving increased attention because of less destruction to the physical habitat of microbial communities compared with wet sieving (Mendes et al., 1999; Schutter and Dick, 2002). Dry sieving also ruptures aggregates caused by the abrasive action of sieves so that average aggregate size is reduced to somewhat below that found in undisturbed samples (Kemper and Rosenau, 1986); however, it may not be as destructive as wet sieving in maintaining the physical habitat of microbial communities in aggregates (Mendes et al., 1999; Schutter and Dick, 2002).

Soil organic C (SOC) and total N (STN) concentrations are regarded as slow pools of soil C and N because they change slowly over time because of their large contents (Franzluebbers and Arshad, 1997; Franzluebbers et al., 1999). In contrast, potential C and N mineralization (PCM and PNM) and microbial biomass C and N (MBC and MBN) are considered as active pools that change seasonally (Franzluebbers and Arshad, 1997; Franzluebbers et al., 1999) and influence aggregation (Jastrow et al., 1998; Martens, 2000). Similarly, particulate organic C and N (POC and PON) are considered as intermediate pools for changes of soil C and N with time that provide substrates for microbes (Beare et al., 1994; Franzluebbers et al., 1999; Six et al., 1999) and also influence aggregation (Beare et al., 1994; Six et al., 1999; Gale et al., 2000). Available forms of N that influence plant growth or loss of N caused by leaching or volatilization are $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$. Because the level of aggregation and aggregate turnover influence soil organic matter (Six et al., 1998), determination of C and N pools in aggregates provides important information on soil C and N sequestration and mineralization in aggregate size fractions that could be protected by using appropriate soil and crop management practices (Whalen and Chang, 2002).

The distribution of C and N pools among soil aggregates is heterogeneous (Gupta and Germida, 1988; Seech and Beauchamp, 1988; Miller and Dick, 1995). Whereas some researchers have reported greater PCM, PNM, MBC, and PNM in macroaggregates (>0.25 mm) than in microaggregates determined by wet sieving (Gupta and Germida, 1988; Franzluebbers and Arshad, 1997), others have found higher values in microaggregates than in macroaggregates (Seech and Beauchamp, 1988; Jastrow et al., 1998). Still others have reported greater microbial biomass and activities in the intermediate size (0.25–1.00 mm) aggregates than in the greater than 1.00-mm or less than 0.25-mm size class (Beare et al., 1994; Six et al., 1998). Similar variations in C and N pools exist among aggregate size fractions determined by dry sieving (Mendes et al., 1999; Schutter and Dick, 2002; Sainju et al., 2003a,b). The differences in C and N pools among aggregate size fractions result from soil properties, such as clay and organic matter contents and clay mineralogy (Franzluebbers et al., 2000; Six et al., 2000).

Although information on C and N pools in soil aggregates determined by dry and wet sieving methods is available (Kemper and Rosenau, 1986; Cambardella and Elliott, 1993; Mendes et al., 1999; Schutter and Dick, 2002), little is known about variations in C and N pools in aggregate size fractions separated by dry and wet sieving methods. It is hypothesized that dry sieving would be as reliable as wet sieving in determining aggregate size fractions and soil C and N pools, and that these pools will be greater in dry than in wet sieving. The objectives of the study were to (i) compare dry and wet sieving methods of determining aggregate size fractions and associated C and N pools in soils with varying properties and cropping systems and (ii) determine if the dry-sieving method can be used to replace the tedious wet sieving method for soil aggregation and C and N pools.

MATERIALS AND METHODS

Site Description and Soil Sampling

Soil samples were collected from four locations with various properties and cropping systems (Table 1) after crop harvest in September to October 2004 in the semiarid regions of eastern Montana and western North Dakota. The locations contained both dryland and irrigated cropping systems with tilled crops relevant to the region and no-tilled conservation

reserve program (CRP) planting dominated by alfalfa (*Medicago sativa* L.), crested wheatgrass [*Agropyron cristatum* (L.) Gaertn], and western wheatgrass [*Pascopyrum smithii* (Rydb.) A. Love] for more than 20 years. Although recommended doses of N, phosphorus (P), and potassium (K) fertilizers were applied to crops before planting in April 2004 in Rasmussen and Froid, soil samples collected in Sidney had fertilizers applied immediately after crop harvest in October. As a result, soil NO₃-N concentration was higher in Sidney than in other locations (Table 1). In Nesson Valley, no fertilizers, herbicides, and pesticides were applied to CRP planting. Soils were collected randomly from 25 different places within a 0.25-ha area in each location, divided into 0- to 5- and 5- to 20-cm depths, composited within a depth, and passed through a 4.75-mm sieve by gently pressing the large aggregates by hand. Particles that did not pass through the 4.75-mm sieve contained mostly stone and plant fragments and were discarded. Half of the soil was stored moist at 4 °C to separate aggregates by dry-sieving method and the other half air dried at room temperature to separate aggregates by wet sieving method. A portion (10 g) of field-moist

soil was oven dried at 110 °C to determine moisture content.

Aggregate Separation

The dry-sieving method of aggregate separation was performed as described by Mendes et al. (1999) and Schutter and Dick (2002). Field-moist soils were air dried at 4 °C for 7 to 10 days until they reached a gravimetric water content of 80 to 140 g kg⁻¹, depending on clay content. This water content represented the moisture level at which soils can be sieved in finer sieves for aggregate size separation according to our preliminary observations (data not shown). Drying the soil at 4 °C reduces its impact on microbial communities and activities in aggregates (Mendes et al., 1999; Schutter and Dick, 2002). Aggregates were separated by placing 500 g of cold-dried soil fragments (<4.75 mm) in a nest of sieves (20-cm diameter) containing 2.00- and 0.25-mm sieves attached to a Tyler Ro-Tap sieve shaker (Combustion Engineering Inc., Mentor, OH). These sieve sizes were chosen to separate soil aggregates as large macroaggregates (4.75–2.00 mm), small macroaggregates (2.00–0.25 mm), and microaggregates

TABLE 1

Soil properties and cropping system of various locations used for determining aggregate size distribution and C and N pools

Soil properties	Locations							
	Froid		Nesson Valley		Rasmussen		Sidney	
	0–5*	5–20*	0–5	5–20	0–5	5–20	0–5	5–20
SOC (g kg ⁻¹)	9.5	8.0	14.7	10.9	13.3	10.6	10.5	9.9
POC (g kg ⁻¹)	1.53	0.84	2.10	1.66	1.73	1.75	2.07	2.55
PCM (mg kg ⁻¹)	139	147	177	121	219	103	171	136
MBC (mg kg ⁻¹)	571	531	893	512	747	541	531	446
STN (g kg ⁻¹)	0.75	0.67	1.53	0.96	1.14	0.96	1.13	0.86
PON (g kg ⁻¹)	0.10	0.06	0.11	0.05	0.15	0.12	0.25	0.13
PNM (mg kg ⁻¹)	8.5	6.8	10.9	8.4	17.0	11.7	9.9	7.1
MBN (mg kg ⁻¹)	27.5	10.7	86.4	14.1	61.2	23.1	56.7	14.7
NH ₄ -N (mg kg ⁻¹)	7.0	4.3	4.0	2.4	10.3	5.6	2.7	0.5
NO ₃ -N (mg kg ⁻¹)	0.9	1.6	1.7	0.6	6.7	5.9	46.0	13.4
Sand (g kg ⁻¹)	660	630	720	720	350	350	230	190
Silt (g kg ⁻¹)	180	190	100	130	310	340	450	470
Clay (g kg ⁻¹)	160	180	180	150	340	310	320	340
pH	7.1	7.2	7.7	7.8	7.0	7.3	7.1	8.4
Irrigation	Dryland		Dryland		Dryland		Irrigated	
Cropping system	Wheat (<i>Triticum aestivum</i> L.), barley (<i>Hordeum vulgare</i> L.), pea (<i>Pisum sativum</i> L.)		Alfalfa (<i>M. sativa</i> L.), grasses [†]		Wheat, barley, pea		Sugarbeet (<i>Beta vulgaris</i> L.), barley	
Tillage	Yes	Yes	No	No	Yes	Yes	Yes	Yes

*0- to 5- and 5- to 20-cm soil depths.

[†]Dominant grasses are crested wheatgrass [*Agropyron cristatum* (L.) Gaertn], western wheatgrass [*Pascopyrum smithii* (Rydb.) A. Love], and green needlegrass [*Nassella viridula* (Trin.) Backworth].

and silt and clay fractions (<0.25 mm) (Six et al., 1999, 2000). Sieves were shaken at 200 oscillations min^{-1} for 3 min and aggregates (4.75–2.00, 2.00–0.25, and <0.25 mm) retained and passed through the sieves were weighed (Mendes et al., 1999; Schutter and Dick, 2002). These were air dried at room temperature and stored in plastic bags until C and N pools were determined. The process was repeated two more times to obtain three replications of the experiment for statistical analysis of data.

The wet sieving method of aggregate separation was performed as suggested by Elliott (1986) and Six et al. (1999, 2000). Air-dried soil (500 g) was submerged for 5 min in a 2-mm sieve. Aggregates were separated by moving the sieve up and down for 3 cm with 50 repetitions for 2 min. Aggregates retained in the sieve (4.75–2.00 mm) were air dried at room temperature, weighed, and stored in the plastic bag. Sieving was repeated as above using the next sieve (0.25 mm) by pouring the soil and water that passed through the 2-mm sieve in the 0.25-mm sieve and shaking. Aggregates retained in the sieve (2.00–0.25 mm) and that passed through it (<0.25 mm) were collected, air dried, weighed, and stored in plastic bag. As with the dry-sieving method, the process was repeated two more times to obtain three replications of the experiment. Mean weight diameter (MWD) of aggregates separated by dry and wet sieving methods was calculated by summing the product of mean diameter of aggregates and proportion of soil in each aggregate-size class (Kemper and Rosenau, 1986).

Carbon and Nitrogen Analysis

The SOC concentration (g kg^{-1}) in soil aggregates was determined by using the high induction furnace C and N analyzer (LECO, St. Joseph, MI) after grinding the aggregates to 0.1 mm and pretreating the soil with 5% H_2SO_3 to remove inorganic C (Nelson and Sommers, 1996). The STN was determined by using the analyzer as above without pretreating the soil with acid. The PCM and PNM in aggregates were determined by the method modified by Haney et al. (2004). Two 10-g aggregates were moistened with water at 50% field capacity and placed in a 1-L jar containing beakers with 4 mL of 0.5 mol/L NaOH to trap evolved carbon dioxide (CO_2) and 20 mL of water to maintain high humidity. Aggregates were incubated in the jar at 21 °C for 7 days. At 7 days, the beaker containing NaOH was removed from the jar

and PCM was determined by measuring CO_2 absorbed in NaOH, which was back-titrated with 1.5 mol/L BaCl_2 and 0.1 mol/L HCl. One beaker containing aggregates was removed from the jar and extracted with 100 mL of 2 mol/L KCl for 1 h. The $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ concentrations in the extract were determined by using an autoanalyzer (Lachat Instrument, Loveland, CO). The PNM was calculated as the difference between the sum of $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ concentrations in the aggregates before and after incubation.

The other beaker containing moist aggregates and incubated for 7 days (used for PCM determination above) was used for determining MBC and MBN by the modified fumigation-incubation method for air-dried soils (Franzluebbers et al., 1996). The moist aggregates were fumigated with ethanol-free chloroform for 24 h and placed in a 1-L jar containing beakers with 2 mL of 0.5 mol/L NaOH and 20 mL water. As with PCM, the fumigated moist aggregates were incubated for 10 days and CO_2 absorbed in NaOH was back-titrated with BaCl_2 and HCl. The MBC was calculated by dividing the amount of $\text{CO}_2\text{-C}$ absorbed in NaOH by a factor of 0.41 (Voroney and Paul, 1984) without subtracting the values from the nonfumigated control (Franzluebbers et al., 1996). For MBN, the fumigated-incubated sample at 10 days was extracted with 100 mL of 2 mol/L KCl for 1 h, and $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ concentrations were determined by autoanalyzer as above. The MBN was calculated as the difference between the sum of $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ concentrations in the sample before and after fumigation-incubation and divided by a factor of 0.41 (Voroney and Paul, 1984; Brookes et al., 1985). The $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ concentrations determined in the nonfumigated-nonincubated aggregates were used as available pools of N.

Data Analysis

Data for soil aggregates and C and N pools in each location were analyzed using the MIXED procedure of SAS in a split-split plot analysis (Littell et al., 1996). Aggregate separation method was considered as the main plot factor, aggregate size fraction as the split plot factor, and soil depth as the split-split plot factor for analysis. Similarly, the fixed effects in the analysis were aggregate separation method, aggregate size fraction, and soil depth, whereas random effects were replication and aggregate separation method \times size fraction \times replication.

Analysis was repeated as above by replacing soil depth by location to compare soil aggregation and C and N pools by sieving methods among locations. Means were separated by using the least square means test when treatments and interactions were significant. Statistical significance was evaluated at $P \leq 0.05$, unless otherwise stated. Correlation analysis was done to determine the relationships between dry and wet sieving methods in soil aggregation and C and N pools.

RESULTS AND DISCUSSION

Aggregate Size Distribution

The amount of large macroaggregates (4.75–2.00 mm) was lower and small macroaggregates (2.00–0.25 mm) was higher than the amount of microaggregates and silt and clay fractions (<0.25 mm) in all samples, except for the 0- to 5- and 5- to 10-cm aggregates separated by dry sieving in Rasmussen and for the 0- to 5-cm aggregates separated by dry sieving in Sidney (Table 2). The amount of large macroaggregates was, however, higher in dry than in wet sieving for all soils, except for the samples at 0 to 5 cm in Nesson Valley and at 5 to 20 cm in Sidney. In contrast, the amount of small macroaggregates was higher in wet than in dry sieving for all sites, except in Sidney. The amount of microaggregates and silt and clay fractions between sieving methods varied between soil depths and sites. The amount of large macroaggregates using dry sieving was higher at 5 to 20 than at 0 to 5 cm but was similar between depths for aggregates using wet sieving at all locations, except in Sidney. In the less than 0.25-mm fractions, the amount of soil was higher at 0 to 5 than at 5 to 20 cm for both dry and wet sieving methods, except in Sidney. The amount of large macroaggregates using dry sieving within a depth was higher in Rasmussen than in other locations but was similar between locations using wet sieving. A highly significant relationship was observed between wet- and dry-sieving methods for aggregate size fractions (Table 3). The MWD of aggregates was higher in dry than in wet sieving at 0 to 5 cm in Rasmussen and Sidney and at 5 to 20 cm in Froid and Rasmussen but was higher in wet than in dry sieving at 0 to 5 cm in Nesson Valley.

The lower amount of large macroaggregates in wet than in dry sieving was caused by rupture of weak aggregates in water because of ion hydration, sudden release of internal air pressure,

osmotic swelling forces, or to solubility of binding agents in water (Kemper and Rosenau, 1986; Perfect et al., 1990; Gale et al., 2000). Large macroaggregates were especially vulnerable to aggregate rupture by water compared with small macroaggregates (Table 2). This resulted in the increase in the amount of other aggregates by wet sieving. The lower amount of large macroaggregates at 0- to 5- than at 5- to 20-cm depth by dry sieving was probably caused by their rupture at the soil surface from the use of heavy machinery equipments used for tillage, for applications of fertilizer, herbicides, and pesticides, and for crop harvest. Whalen and Chang (2002) also made similar observations in the northern Great Plains. The higher amount of large macroaggregates by dry sieving under dryland cropping system in Rasmussen than in Froid and Nesson Valley could be caused by their higher clay content (Table 1). Increased clay content increases the proportion of macroaggregates in the soil (Franzluebbers et al., 2000). Although clay content was higher in Sidney (Table 1), the lower amount of large macroaggregates separated by both dry and wet sieving could be a result of aggregate erosion caused by the action of water during irrigation. The significant relationship between dry and wet sieving methods for aggregate size distribution (Table 3) suggests that dry sieving, being a simple and rapid method, could replace the more complex and tedious wet sieving method for aggregate separation.

The higher MWD of aggregates, which measures aggregate stability (Kemper and Rosenau, 1986), by dry than by wet sieving was likely a result of a higher amount of large macroaggregates by dry sieving (Table 2). In Nesson Valley, the land had been under CRP planting for more than 20 years. As a result, higher MWD in wet than in dry sieving at 0 to 5 cm was probably caused by increased formation of water-stable aggregates because grassland favors the formation of more water-stable aggregates than croplands (Elliott, 1986; Six et al., 1998, 2000). Similarly, higher MWD at 5 to 20 than at 0 to 5 cm was probably caused by the destruction of macroaggregates at the soil surface as a result of heavy equipment use. Increased clay content also increased MWD in Rasmussen and Sidney than in other locations.

Aggregate Carbon Pools

The SOC at the 0- to 5-cm depth was higher in wet than in dry sieving in 4.75- to

TABLE 2

Effect of aggregate separation method on aggregate size distribution and MWD of aggregates from different locations

Aggregate size	Amount of soil in aggregate size fractions at soil depth (cm)			
	0-5		5-20	
	Dry*	Wet*	Dry	Wet
mm	g aggregate kg ⁻¹ soil			
Froid				
4.75-2.00	86c [†] A [‡]	22cB	271bA	23cB
2.00-0.25	620aB	729aA	640aB	823aA
<0.25	294bA	249bA	89cB	154bA
MWD	1.39A	1.37A	1.89A	1.54B
Nesson Valley				
4.75-2.00	28cA	34cA	77cA	34cB
2.00-0.25	809aB	870aA	789aB	884aA
<0.25	163bA	96bB	134bA	82bB
MWD	1.53B	1.63A	1.62A	1.65A
Rasmussen				
4.75-2.00	198bA	21cB	309bA	9cB
2.00-0.25	631aB	795aA	641aB	847aA
<0.25	171bA	184bA	50cB	144bA
MWD	1.69A	1.48B	1.98A	1.53B
Sidney				
4.75-2.00	53bA	5cB	30cA	9cA
2.00-0.25	811aA	711aB	800aA	770aA
<0.25	136bB	284bA	170bB	221bA
MWD	1.67A	1.31B	1.59A	11.40A

*Dry and wet aggregate separation methods.

[†]Numbers followed by different lowercase letter within a column at a soil depth in a location are significantly different at $P \leq 0.05$ by the least square means test.[‡]Numbers followed by different uppercase letter within a row at a soil depth in a location are significantly different at $P \leq 0.05$ by the least square means test.

2.00-mm fractions but was higher in dry than in wet sieving in less than 0.25-mm fractions in Nesson Valley (Table 4). In Rasmussen, the trend reversed. In Sidney, SOC at 5 to 20 cm was higher in dry than in wet sieving in less than 2.00-mm fractions. The SOC at 0 to 5 and 5 to 20 cm was higher in less than 0.25-mm fractions than in other fractions separated by dry sieving in Nesson Valley. In Rasmussen, SOC was similarly higher in less than 0.25-mm than in 4.75- to 2.00-mm fractions separated by dry and wet sieving, except for fractions at 0 to 5 cm by dry sieving where the trend reversed. Similar to SOC, POC was higher in less than 0.25-mm than in 2.00- to 0.25-mm fractions separated by dry sieving at 0 to 5 and 5 to 20 cm in Nesson Valley but was higher in 4.75- to 2.00-mm than in other fractions separated by dry sieving at 0 to 5 cm in Rasmussen. The POC was higher in dry than in wet sieving in less than 0.25-mm fractions at 0 to 5 and 5 to 20 cm in Nesson Valley and in 2.00- to 0.25-mm fractions at 0- to 5-cm in Rasmussen. Both SOC and POC

in aggregates were higher at 0 to 5 than at 5 to 20 cm, regardless of sieving methods and locations. Similarly, SOC and POC in aggregates were higher in Rasmussen and Sidney than in Froid and Nesson Valley, regardless of sieving methods. The lack of enough aggregates in 4.75- to 2.00-mm fractions separated by dry and wet sieving prevented the determination of SOC and POC in some of these fractions.

Differences in cropping systems and soil properties between locations influenced distribution of SOC and POC among aggregate size fractions determined by dry and wet sieving methods. In Nesson Valley, land had been kept under CRP planting with grasses and alfalfa under no-tilled condition for more than 20 years compared with tilled crops in Rasmussen and Sidney (Table 1). Inasmuch as grassland soils had higher SOC concentration than cropland soils because of continuous plant C inputs from above ground and below ground biomass and reduced rate of mineralization from decreased soil disturbance (Elliott, 1986; Cambardella and

TABLE 3

Correlation between dry and wet sieving methods for soil aggregate size fractions and C and N pools in individual and all aggregate sizes (n = 8–24)

Parameter	Correlation coefficient			
	All aggregates	4.75–2.00 mm	2.00–0.25 mm	<0.25 mm
Aggregate size fractions	0.94*	— [†]	—	—
MWD of aggregates	0.22	—	—	—
SOC	0.87*	0.88 [‡]	0.98*	0.82 [‡]
POC	0.72 [§]	—	0.80 [‡]	0.81 [‡]
MBC	0.74*	0.97 [§]	0.83 [‡]	0.83 [‡]
PCM	0.85*	0.92 [§]	0.90 [§]	0.87 [‡]
STN	0.91*	0.94 [§]	0.99*	0.91 [§]
PON	0.82*	—	0.83 [‡]	0.79 [‡]
MBN	0.51 [‡]	—	0.74 [‡]	0.22
PNM	0.46	—	0.75 [‡]	0.07
NH ₄ -N	0.47 [‡]	—	0.68	0.43
NO ₃ -N	0.80*	—	0.80 [‡]	0.92 [§]

*Significant at $P = 0.001$.

[†]Missing samples or values that cannot be determined.

[‡]Significant at $P = 0.05$.

[§]Significant at $P = 0.01$.

Elliott, 1993; Six et al., 1998, 2000), it could be possible that some of these C inputs were preferentially sequestered in less than 0.25-mm fractions in grassland soils. As a result, SOC and

POC were higher in less than 0.25-mm fractions than in other fractions separated by dry compared with wet sieving in Nesson Valley. Results of dry sieving include both water-soluble and -insoluble

TABLE 4

Effect of aggregate separation method on SOC and POC in sand-free aggregates from various locations

Aggregate size	SOC at soil depth (cm)				POC at soil depth (cm)			
	0–5		5–20		0–5		5–20	
	Dry*	Wet*	Dry	Wet	Dry	Wet	Dry	Wet
mm	g C kg ⁻¹ sand-free aggregates							
Froid								
4.75–2.00	16.7a [†]	— [‡]	11.6a	—	—	—	5.6a	—
2.00–0.25	12.7aA [§]	13.8aA	11.7aA	10.8aA	6.3aA	7.1aA	5.9aA	3.9aA
<0.25	14.0aA	12.9aA	13.6aA	11.5aA	7.4aA	4.2aA	5.20aA	3.9aA
Nesson Valley								
4.75–2.00	18.7bB	23.9aA	11.3bA	14.7aA	—	—	—	—
2.00–0.25	11.1cA	13.8bA	8.8bA	13.2aA	7.5bA	8.6aA	5.0bA	7.0aA
<0.25	36.0aA	26.5aB	32.0aA	13.0aB	18.2aA	8.0aB	15.8aA	6.8aB
Rasmussen								
4.75–2.00	36.2aA	26.6bB	27.0bB	30.5bA	15.9a	—	2.5a	—
2.00–0.25	40.1aA	34.6aA	28.6abA	29.9bA	12.8bA	6.7aB	4.5aA	4.6aA
<0.25	29.4bB	37.3aA	31.9aA	33.6aA	5.0cA	5.7aA	4.0aA	4.9aA
Sidney								
4.75–2.00	49.3a	—	43.1aA	45.8aA	19.0a	—	13.9a	—
2.00–0.25	49.4aA	36.9bB	48.9aA	38.2bB	20.7aA	12.6aA	15.0aA	18.2aA
<0.25	48.0aA	48.1aA	48.5aA	39.8bB	19.1aA	13.5aA	14.4aA	11.1bA

*Dry and wet aggregate separation methods.

[†]Numbers followed by different lowercase letter within a column at a soil depth in a location are significantly different at $P \leq 0.05$ by the least square means test.

[‡]Missing samples.

[§]Numbers followed by different uppercase letter within a row at a soil depth in a location are significantly different at $P \leq 0.05$ by the least square means test.

TABLE 5
Effect of aggregate separation method on soil PCM and MBC in sand-free aggregates from various locations

Aggregate size	PCM at soil depth (cm)				MBC at soil depth (cm)			
	0-5		5-20		0-5		5-20	
	Dry*	Wet*	Dry	Wet	Dry	Wet	Dry	Wet
mm	mg C kg ⁻¹ sand-free aggregates							
Froid								
4.75-2.00	289a [†] A [‡]	168aB	285bA	300aA	939aB	1486aA	819bA	534aA
2.00-0.25	290aA	148aB	293bA	108bB	855aB	1115abA	906abA	570aB
<0.25	208bA	195aA	478aA	162bB	836aA	994bA	1122aA	663aB
Nesson Valley								
4.75-2.00	— [§]	230a	—	259a	—	1728a	—	860b
2.00-0.25	160bB	214aA	135bA	171bA	1178bA	1134bA	758bB	903abA
<0.25	285aA	180aB	210aA	221abA	2174aA	1098bB	1308aA	996aB
Rasmussen								
4.75-2.00	599aA	506aB	242a	—	2100aB	3554aA	1282a	—
2.00-0.25	443bA	301bB	234aA	279aA	1986aA	1978cA	1274aB	2078aA
<0.25	470bA	387bA	230aA	332aA	2053aB	2360bA	1200aB	1801aA
Sidney								
4.75-2.00	1433a	—	640b	—	3492a	—	2943a	—
2.00-0.25	1086bA	494aB	1114aA	470aB	3593aA	2314aB	3020aA	2326aB
<0.25	1200abA	580aB	1200aA	530aB	3500aA	2933aA	3000aA	2621aA

*Dry and wet aggregate separation methods.

[†]Numbers followed by different lowercase letter within a column at a soil depth in a location are significantly different at $P \leq 0.05$ by the least square means test.

[‡]Numbers followed by different uppercase letter within a row at a soil depth in a location are significantly different at $P \leq 0.05$ by the least square means test.

[§]Missing samples.

C in total C determinations in these fractions that were not possible by wet sieving because of loss of water-soluble C during sieving of aggregates in water (Seech and Beauchamp, 1988). In contrast, variations in SOC and POC among aggregate size fractions in croplands could be a result of differences in mineralization rates of soil organic matter caused by destruction of aggregates during tillage, sequestration of C in aggregates during plant growth, and differences in soil properties, such as clay content (Wan and El-Swaify, 1996; Maguire et al., 1998; Whalen and Chang, 2002). The higher SOC in 4.75- to 2.00-mm fractions at 0 to 5 cm by wet than by dry sieving in Nesson Valley could be a result of preferential conservation of C in large aggregates in no-tilled sandy soil under CRP planting, where stored C can be determined only when aggregates were separated by wet sieving. Similarly, higher SOC in less than 0.25-mm fractions at 0 to 5 cm by wet than by dry sieving in Rasmussen suggests that C could be stored preferentially in micro-aggregates and silt and clay fractions in tilled soil with higher clay content, where stored C can be determined when aggregates were separated by wet sieving.

It is not surprising to observe higher SOC and POC concentrations in aggregates at the 0- to 5-cm than at the 5- to 20-cm depth because of increased plant residue addition at the surface soil because SOC and POC for the whole soil also showed a similar pattern (Table 1). This was especially true in no-till grassland soil in Nesson Valley. Similarly, higher SOC and POC in aggregates in Rasmussen and Sidney than in other locations was probably related to their higher clay content because increased clay content is usually associated with high organic matter concentration (Franzluebbers et al., 2000; Six et al., 2000). The SOC and POC in aggregates separated by dry and wet sieving correlated ($r = 0.70-0.97$, $P \leq 0.05$, $n = 8$) with clay content.

Unlike SOC, PCM was similar to or higher in dry than in wet sieving for most aggregate size fractions in all locations, except for the 2.00- to 0.25-mm fractions at the 0- to 5-cm depth in Nesson Valley (Table 5). The PCM was higher in 4.75- to 2.00- mm than in less than 0.25-mm fractions separated by dry sieving at 0 to 5 cm in Froid and Rasmussen, by wet sieving at 0 to 5 cm in Rasmussen, and by wet sieving at 5 to 20 cm in Froid. In contrast, PCM

TABLE 6
Effect of aggregate separation method on STN and PON in sand-free aggregates from various locations

Aggregate size	STN at soil depth (cm)				PON at soil depth (cm)			
	0-5		5-20		0-5		5-20	
	Dry*	Wet*	Dry	Wet	Dry	Wet	Dry	Wet
mm	g N kg ⁻¹ sand-free aggregates							
Froid								
4.75-2.00	1.12a [†] A [‡]	1.36aA	0.97aB	1.39aA	— [§]	—	0.72a	—
2.00-0.25	1.11aA	1.00bA	1.00aA	0.97bA	0.54aA	0.57aA	0.70aA	0.66aA
<0.25	1.10aA	0.93bA	1.10aA	1.17abA	0.69aA	0.65aA	0.68aA	0.67aA
Nesson Valley								
4.75-2.00	2.20bA	0.78bB	0.90bA	1.17bA	—	—	—	—
2.00-0.25	1.20cA	1.21abA	0.83bA	0.77cA	0.70bA	0.68aA	0.48bA	0.29aA
<0.25	3.39aA	1.62aB	2.86aA	1.82aB	1.96aA	1.15aB	1.90aA	0.35aB
Rasmussen								
4.75-2.00	3.16aA	3.43aA	2.82aA	2.45aA	1.14a	—	1.50a	—
2.00-0.25	3.23aA	2.53bA	2.64aA	2.03aA	1.00aA	0.73aA	1.87aA	0.82aB
<0.25	2.69aA	2.38bA	2.39aA	2.74aA	1.38aA	1.06aA	1.75aA	1.47aA
Sidney								
4.75-2.00	5.84a	—	5.84aA	6.89aA	2.53b	—	3.62a	—
2.00-0.25	6.46aA	4.40aA	5.88aA	4.72bA	3.72aA	1.58aB	2.38aA	2.01aA
<0.25	5.90aA	5.99aA	5.95aA	4.43bA	4.00aA	1.92aB	3.00aA	1.64aB

*Dry and wet aggregate separation methods.

[†] Numbers followed by different lowercase letter within a column at a soil depth in a location are significantly different at $P \leq 0.05$ by the least square means test.

[‡] Numbers followed by different uppercase letter within a row at a soil depth in a location are significantly different at $P \leq 0.05$ by the least square means test.

[§] Missing samples.

was higher in less than 0.25-mm than in 4.75- to 2.00-mm fractions separated by dry sieving at 5 to 20 cm in Froid and Sidney. The MBC was higher in dry than in wet sieving for less than 0.25-mm fractions at 0 to 5 cm in Nesson Valley, for 2.00- to 0.25-mm fractions at 0 to 5 cm in Sidney, and for 2.00- to 0.25-mm fractions at 5 to 20 cm in Froid and Sidney. On the other hand, MBC was higher in wet than in dry sieving for 4.75- to 0.25-mm fractions at 0 to 5 cm in Froid, for 2.00- to 0.25-mm at 5 to 20 cm in Nesson Valley, and for 4.75- to 2.00-mm and less than 0.25-mm fractions at 0 to 5 cm and for less than 2.00-mm fractions at 5 to 20 cm in Rasmussen. Both PCM and MBC in aggregates were not influenced by soil depths. The PCM and MBC were higher in Rasmussen and Sidney than in Froid and Nesson Valley. Similar to aggregate size distribution, C pools in individual and all aggregate size fractions determined by dry and wet sieving methods were significantly correlated (Table 3).

The dry-sieving method of aggregate separation had a more distinct influence on PCM than on MBC. As with SOC and POC, higher

PCM in aggregates separated by dry than by wet sieving (Table 5) was probably caused by loss of water-soluble C during wet sieving. Carbon stored in microbial communities (MBC) seemed to be less influenced by loss of water-soluble C than in PCM, thereby resulting in a variable distribution of MBC among aggregates separated by dry and wet sieving. Similar to SOC and POC, variability in PCM and MBC among aggregate size fractions separated by dry and wet sieving methods in croplands and grasslands has been shown by others (Beare et al., 1994; Seech and Beauchamp, 1988; Mendes and Bottomley, 1998; Sainju et al., 2003a,b). Higher PCM and MBC in Rasmussen and Sidney than in other locations were probably related to their higher clay content ($r = 0.62$ to 0.99 , $P \leq 0.10$, $n = 8$). As in aggregate size distribution, significant correlation of C pools in aggregates between dry and wet sieving suggests that the dry-sieving method of aggregate separation is as good as the wet sieving method in the determination of C pools.

Aggregate Nitrogen Pools

Similar to SOC and POC, significant differences in STN and PON in aggregates between

TABLE 7
Effect of aggregate separation method on soil PNM and MBN in sand-free aggregates from various locations

Aggregate size	PNM at soil depth (cm)				MBN at soil depth (cm)			
	0-5		5-20		0-5		5-20	
	Dry*	Wet*	Dry	Wet	Dry	Wet	Dry	Wet
mm	mg N kg ⁻¹ sand-free aggregates							
Froid								
4.75-2.00	— [†]	—	5.5a	—	—	—	20.8c	—
2.00-0.25	8.6b [‡] A [§]	1.8aB	6.1aA	2.3aB	53.0aA	13.6aB	38.1bA	9.4aB
<0.25	10.6aA	0.89aB	6.3aA	3.2aB	52.8aA	13.8aB	60.0aA	10.2aB
Nesson Valley								
4.75-2.00	—	—	—	—	—	—	—	—
2.00-0.25	13.9bA	1.1aB	34.2aA	1.10bB	141.7bA	20.6aB	34.4bA	4.3aB
<0.25	28.9aA	3.1aB	19.2bA	4.5aB	236.1aA	42.9aB	108.2aA	4.8aB
Rasmussen								
4.75-2.00	69.6a	—	45.6a	—	407.0a	—	210.8a	—
2.00-0.25	63.5aA	9.5aB	44.3aA	2.3aB	506.3aA	45.8aB	154.7aA	6.9aB
<0.25	63.3aA	3.7aB	45.0aA	0.7aB	521.6aA	29.6aB	180.5aA	21.1aB
Sidney								
4.75-2.00	—	—	8.8a	—	—	—	300.0a	—
2.00-0.25	23.8aA	1.5aB	10.5aA	1.4aB	624.7aA	27.7aB	373.7aA	12.0aB
<0.25	18.5aA	3.1aB	11.3aA	3.9aB	600.1aA	11.4aB	390.5aA	16.4aB

*Dry and wet aggregate separation methods.

[†]Missing samples.

[‡]Numbers followed by different lowercase letter within a column at a soil depth in a location are significantly different at $P \leq 0.05$ by the least square means test.

[§]Numbers followed by different uppercase letter within a row at a soil depth in a location are significantly different at $P \leq 0.05$ by the least square means test.

dry and wet sieving occurred only in some samples (Table 6). The STN was higher in dry than in wet sieving in 4.75- to 2.00-mm and less than 0.25-mm fractions at 0 to 5 cm and in less than 0.25-mm fractions at 5 to 20 cm in Nesson Valley but was higher in wet than in dry sieving in 4.75- to 2.00-mm fractions at 5 to 20 cm in Froid. The PON was higher in dry than in wet sieving in less than 0.25-mm fractions at 0 to 5 and 5 to 20 cm in Nesson Valley, in less than 0.25-mm fractions at 0 to 5 and 5 to 20 cm in Sidney, and in 2.00- to 0.25-mm fractions at 5 to 20 cm in Rasmussen. The STN was higher in 4.75- to 2.00-mm than in less than 2.00-mm fractions separated by wet sieving at 0 to 5 cm in Froid and Rasmussen and at 5 to 20 cm in Sidney but was higher in less than 0.25-mm than in 4.75- to 2.00-mm fractions separated by dry and wet sieving at 0 to 5 and 5 to 20 cm in Nesson Valley. The PON was higher in less than 0.25-mm than in 2.00- to 0.25-mm fractions separated by dry sieving at 0 to 5 and 5 to 20 cm in Nesson Valley and higher in less than 2.00-mm than in 4.75- to 2.00-mm fractions separated by dry sieving at 0 to 5 cm in Sidney. Unlike SOC and

POC, STN and PON were not influenced by soil depth but were higher in Rasmussen and Sidney than in Froid and Nesson Valley.

The PNM, MBN, NH₄-N, and NO₃-N concentrations were higher in dry than in wet sieving for all aggregate size fractions in all locations, except for NH₄-N and NO₃-N concentrations in some aggregates where the concentrations were similar or higher in wet than in dry sieving (Tables 7 and 8). The PNM was higher in less than 0.25-mm than in 2.00- to 0.25-mm fractions separated by dry sieving at 0 to 5 cm in Froid and separated by dry sieving at 0 to 5 cm and by wet sieving at 5 to 20 cm in Nesson Valley (Table 6). In contrast, PNM was higher in 2.00- to 0.25-mm than in less than 0.25-mm fractions separated by dry sieving at 5 to 20 cm in Nesson Valley. The MBN was higher in less than 0.25 mm than in 2.00- to 0.25-mm fractions separated by dry sieving at 0 to 5 and 5 to 20 cm in Nesson Valley and by dry sieving at 5 to 20 cm in Froid. The NH₄-N concentration was higher in less than 0.25-mm than in 2.00- to 0.25-mm fractions separated by wet sieving at 0 to 5 and 5 to 20 cm in Froid, by dry and wet sieving at 0 to 5 cm and by dry

TABLE 8

Effect of aggregate separation method on soil $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ concentrations in sand-free aggregates from various locations

Aggregate size	$\text{NH}_4\text{-N}$ at soil depth (cm)				$\text{NO}_3\text{-N}$ at soil depth (cm)			
	0-5		5-20		0-5		5-20	
	Dry*	Wet*	Dry	Wet	Dry	Wet	Dry	Wet
mm	mg N kg ⁻¹ sand-free aggregates							
Froid								
4.75-2.00	— [†]	—	0.97a [‡]	—	—	—	1.93a	—
2.00-0.25	1.63aA [§]	0.40bB	0.90aA	0.40bA	1.40aA	3.00aA	1.80aA	2.20aA
<0.25	1.40bA	0.57aB	0.90aA	1.07aA	0.77aA	2.13aA	1.60aA	3.10aA
Nesson Valley								
4.75-2.00	—	—	0.62b	—	—	—	0.80b	—
2.00-0.25	0.77bA	0.30bA	0.66bA	0.37aA	2.07bA	3.07aA	0.67bB	1.47bA
<0.25	1.60aA	1.40aA	2.00aA	1.05aB	3.73aA	3.05aA	1.47aB	2.15aA
Rasmussen								
4.75-2.00	3.90ab	—	2.73c	—	16.50a	—	13.03a	—
2.00-0.25	4.03aA	0.87aB	3.03bA	1.13bB	15.90aA	4.17aB	15.16aA	4.47bB
<0.25	2.83bA	1.50aB	3.70aA	1.90aB	13.70aA	5.57aB	13.40aA	8.27aB
Sidney								
4.75-2.00	—	—	2.40a	—	—	—	56.90a	—
2.00-0.25	2.27aA	1.37bB	2.30bA	1.30bB	160.2bA	8.33bB	65.53aA	9.66aB
<0.25	1.63bB	2.43aA	2.50aA	1.80aB	156.5aA	18.40aB	70.21aA	15.63aB

*Dry and wet aggregate separation methods.

[†]Missing samples.[‡]Numbers followed by different lowercase letter within a column at a soil depth in a location are significantly different at $P \leq 0.05$ by the least square means test.[§]Numbers followed by different uppercase letter within a row at a soil depth in a location are significantly different at $P \leq 0.05$ by the least square means test.

sieving at 5 to 20 cm in Nesson Valley, by dry and wet sieving at 5 to 20 cm in Rasmussen, and by wet sieving at 0 to 5 cm and by dry and wet sieving at 5 to 20 cm in Sidney (Table 8). In contrast, $\text{NH}_4\text{-N}$ was higher in 2.00- to 0.25-mm than in less than 0.25-mm fractions separated by dry sieving at 0 to 5 cm in Froid, Rasmussen, and Sidney. The $\text{NO}_3\text{-N}$ concentration was higher in less than 0.25 mm than in 2.00- to 0.25-mm fractions separated by dry sieving at 0 to 5 cm and by dry and wet sieving at 5 to 20 cm in Nesson Valley, by wet sieving at 5 to 20 cm in Rasmussen, and by dry and wet sieving at 0 to 5 cm in Sidney. The MBN was higher at 0 to 5 than at 5 to 20 cm, but PNM, $\text{NH}_4\text{-N}$, and $\text{NO}_3\text{-N}$ were not influenced by soil depth. As with other C and N pools, MBN, PNM, $\text{NH}_4\text{-N}$, and $\text{NO}_3\text{-N}$ were higher in Rasmussen and Sidney than in Froid and Nesson Valley. Significant correlation was observed between dry and wet sieving methods in soil N pools in individual and all aggregate size fractions, except for MBN, PNM, and $\text{NH}_4\text{-N}$ (Table 3).

The higher N pools in aggregates in dry than in wet sieving, except for STN, suggests that soil N is even more soluble than soil C, which reduces the levels of N pools when aggregates are sieved in water. Water-soluble N contributed a significant proportion of intermediate, active, and available pools of N compared with STN, thereby influencing N pools in aggregates determined by dry and wet sieving methods. Similarly, higher N pools in less than 0.25-mm than in other fractions, except STN, suggests that a major proportion of active N pools is preferentially stored in microaggregates and silt and clay fractions where most of the N mineralization occurs. Inasmuch as the trends of active and available N pools among aggregate size fractions were similar in tilled cropland and no-tilled grassland, N mineralization and availability among aggregate size fractions were less influenced by cropping systems. Increased clay content not only increased soil aggregation and C pools, but also increased N pools, as evidenced by higher N pools in Rasmussen and Sidney than in Froid and Nesson

Valley. As with C pools, significant correlation between dry and wet sieving methods in STN and PON (Table 3) suggests that the dry sieving method of aggregate separation can be used to replace wet sieving for determining slow N pools in aggregates. However, dry sieving estimates higher concentrations of active and available N pools than wet sieving caused by loss of water-soluble N during wet sieving of aggregates, thereby resulting in nonsignificant relationships between dry and wet sieving methods for PNM, MBN, and $\text{NH}_4\text{-N}$ concentrations in aggregates. Significant correlation between N pools for all aggregates and poor correlation between active N pools in individual aggregates suggests that dry and wet sieving methods impose different biases on N parameters within aggregate size fractions.

CONCLUSIONS

The results suggest that soil aggregate size distribution and C and N pools in aggregates vary between dry and wet sieving methods depending on soil properties and cropping systems. Dry sieving of moist soil is as good as wet sieving in determining soil aggregation, active and slow C pools, and slow N pools, and even better for determining active and available N pools. Higher solubility of N than C in water probably reduced active and available N pools when aggregates were sieved in water. Comparison of C/N ratio (using SOC and STN) in aggregates separated by dry (C/N ratio 7.4–14.9) versus wet (C/N ratio 6.6–16.4) sieving demonstrated that C and N measurements in aggregates were affected differently by sieving methods. The dry-sieving method is simple and rapid and can determine active and available N pools in aggregates for estimating N mineralization and availability in soils which are underestimated by wet sieving method. Wet sieving is not a valid method for aggregate separation when examining biologically active N pools in aggregates. Regardless of sieving methods, soil properties, and cropping systems, most of the C and N pools were stored in microaggregates and silt and clay fractions compared with large and small macroaggregates. In the semiarid drylands, where rainfall is lower than in subhumid regions and soil erosion is mostly caused by the action of wind, dry sieving of moist soil may be used as a rapid and alternative method to the more tedious wet sieving method for determining soil aggregate size distribution to measure wind erosion and

C and N pools in aggregates that influence C and N sequestration and N mineralization in the soil.

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