

Distribution of organic carbon in physical fractions of soils as affected by agricultural management

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Abstract Soil organic carbon (SOC) is distributed heterogeneously among different-sized primary particles and aggregates. Further, the SOC associated with different physical fractions respond differently to managements. Therefore, this study was conducted with the objective to quantify the SOC associated with all the three structural levels of SOC (particulate organic matter, soil separates and aggregate-size fractions) as influenced by long-term change in management. The study also aims at reevaluating the concept that the SOC sink capacity of individual size-fractions is limited. Long-term tillage and crop rotation effects on distribution of SOC among fractions were compared with soil from adjacent undisturbed area under native vegetation for the mixed, mesic, Typic Fragiudalf of Wooster, OH. Forty five years of no-till (NT) management resulted in more SOC accumulation in soil surface (0–7.5 cm) than in chisel tillage and plow tillage (PT) treatments. However, PT at this site resulted in a redistribution of SOC from surface to deeper soil layers. The soils under continuous corn accumulated significantly more SOC than those under corn–soybean rotation at 7.5–45 cm depth. Although soil texture was dominated by the silt-sized particles, most of the SOC pool was associated with the clay fraction. Compared to PT, the NT treatment resulted in

(i) significantly higher proportion of large macroaggregates (>2,000 μm) and (ii) 1.5–2.8 times higher SOC concentrations in all aggregate-size classes. A comparative evaluation using radar graphs indicated that among the physical fractions, the SOC associated with sand and silt fractions quickly changed with a land use conversion from native vegetation to agricultural crops. A key finding of this study is the assessment of SOC sink capacity of individual fractions, which revealed that the clay fraction of agricultural soils continues to accumulate more SOC, albeit at a slower rate, with progressive increase in total SOC concentration. However, the clay fraction of soil under native woodlot showed an indication for SOC saturation. The data presented in this study from all the three structural levels of SOC would be helpful for refining the conceptual pool definitions of the current soil organic matter prediction models.

Keywords Soil organic carbon · Physical fractionation · Aggregates · Particulate organic matter · Tillage · Crop rotation

Introduction

Carbon sequestration in soils can offset the increasing atmospheric CO_2 concentration. The C sink capacity of world soils, estimated at about 1 Pg C year^{-1} , can annually offset 0.47 ppm of CO_2 increase in the atmosphere (IPCC 1999; Lal 2005). In addition, several ancillary benefits associated with soil organic carbon (SOC) sequestration are essential to numerous ecosystem services including food production, and soil, water and air quality. If the potential benefits of SOC sequestration are to be realized, then the importance of quantifying the underlying processes that

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lead to long-term biological stability of SOC cannot be overemphasized (Kong et al. 2005). Several mechanistic processes of SOC stabilization have been proposed including its encapsulation within and between aggregates, interaction with primary soil particles to form organo-mineral complexes and selective preservation of organic compounds because of their inherent recalcitrance (Sollins et al. 1996; Christensen 2001; Six et al. 2002). The interplay between these processes is very complex and, therefore, making it difficult to experimentally identify and quantify SOC pools associated with diverse mechanisms of its stabilization.

Physical fractionation methods are used to distinguish three levels of structural and functional complexity in soil on the basis of size and/or density of soil constituents: (i) uncomplexed organic matter/particulate organic matter (POM), (ii) primary organo-mineral complexes and (iii) secondary organo-mineral complexes/aggregates (Christensen 2001; Chenu et al. 2006). The POM, a primary energy source for heterotrophic microorganisms and a reservoir of labile C (Gregorich et al. 2006), is more influenced by land use and soil management practices than the total soil organic matter (SOM) pool. In soils under a permanent vegetation cover, POM can account for 15–40% of the total SOM in surface horizons, whereas this fraction usually makes up only <10% of the SOM in the surface layer of the long-cultivated cropland soils (Christensen 2001). Its turnover is slower than that of recently added litter but faster than that of SOC associated with mineral particles. Separation of SOC pool into primary organo-mineral complexes of different sizes is based on the understanding that the fraction associated with mineral particles of different sizes varies in structure and function. The formation of micro- and macro-aggregates is another major mechanism by which the SOC is physically protected from microbial decomposition (Hassink and Whitmore 1997).

Land use change significantly influences both quality and quantity of crop residue input to soil and the attendant changes in SOC pool (Bolin and Sukumar 2000). Reeves (1997), on the basis of data from several long-term studies, concluded that cropping results in a general loss of the SOC pool. Additionally, based on a meta-analysis using data from 74 publications from around the world, Guo and Gifford (2002) reported that 42% and 59% of the antecedent SOC pool was depleted upon conversion from forest to cropland and from grassland to cropland, respectively. Lal et al. (1998) estimated that US croplands lost 5 Pg C upon conversion to the agricultural land use. Within agroecosystems, the SOC pool is mostly influenced by crop and soil management practices (e.g. change in tillage, crop rotations). West and Post (2002) analyzed the results from 67 long-term agricultural experiments and concluded that, on average, a shift from intensive tillage to no tillage can

sequester nearly 600 kg C ha⁻¹ year⁻¹. Different crops and cropping sequences affect SOC accretion differently. The magnitude of this effect depends on the amount, chemical composition and decomposition rate of crop residues (Martens 2000).

The available literature on land use and management effects on SOC dynamics mostly focus on changes in total SOC pool. However, total SOC is heterogeneously distributed among three structural levels (POM, primary particles and aggregates), and these SOC fractions respond differently to soil disturbances and management. Handling bulk SOC as one homogeneous pool ignores the variation in relative abundances of differently stabilized SOC fractions in response to change in environmental and climatic factors (von Lützw and Kögel-Knabner 2009). Nonetheless, comprehensive evaluation of the effect of agricultural managements on all the three structural levels of SOC pools on similar soils has rarely been conducted. Few data currently available are mostly derived from experiments of relatively shorter duration. Therefore, the present study was conducted to evaluate how 45 years of agricultural management practices affect the total SOC pool as well as the OC associated with POM, primary particles and aggregates, in comparison with that of an adjacent undisturbed soil. A key aspect of this research is the evaluation of C sink capacity of individual particle-size fractions as influenced by the change in land use. We hypothesized that (i) the quantification of SOC stabilized in individual particle-size fractions, rather than the total SOC pool, may better reflect the management-induced changes in SOC dynamics and (ii) the SOC sink capacity of different-sized soil particles respond differently to land use change.

Materials and methods

Site description

The study was conducted at a long-term agricultural experimental site in the Corn Belt region of the Midwestern USA, located at the Ohio Agricultural Research and Development Center, Wooster, OH. This site lies at 40° 48' N latitude and 82° 00' W longitude. The soils of this site belong to Wooster series (mixed, mesic, Typic Fragiudalf), which are well drained and silt-loam in texture (USDA-SCS 1984). The parent material is low-lime glacial till with a sporadic loess mantle of up to 50 cm in thickness. The native vegetation was a continuous cover of mixed, deciduous hardwood forest. Relic forest remnants occur along creeks and as small woodlots (USDA-SCS 1984). The mean annual temperature is 9.1°C with 173 frost-free days occurring each year and the mean annual precipitation is 905 mm (Dick et al. 1998).

Experimental design and treatments

The experiment was initiated in 1962 in a randomized complete block design (RBD) with factorial arrangement of different tillage and crop rotation treatments. The treatments selected in the present study are three tillage types [plow tillage (PT), chisel tillage (CT) and no tillage (NT)] and two crop rotations [continuous corn (CC) and corn–soybean rotation (CS)]. The PT consisted of one moldboard plowing to a depth of 20–25 cm in spring of each season followed by two 10-cm deep secondary operations (disking, field cultivator) before planting. The PT operation resulted in a complete soil surface inversion and nearly 100% incorporation of crop residues. The CT involved plowing with a chisel, without soil inversion, to a depth of 20–25 cm in spring followed by a single pass of field cultivator to 10 cm depth just before planting. The CT operation retains ~30% of crop residues on the soil surface. The NT involved a complete elimination of pre-plant tillage. Crops in the NT plots were sown using a planter with single coulter to cut through the crop residues and loosen the soil ahead of standard planter units. The treatments were replicated three times, and each plot was 22.3×4.3 m in size. The fertilizer was applied for corn (every year for CC and alternate year when corn is in the field for CS) based on soil test recommendations, with the same rates of N, P and K applied to all the tillage treatments. Detailed record of management practices at this site is available in Dick et al. (1986). Pre- and post-emergence herbicides were applied to the soil for weed control. However, the specific herbicides and rates varied among years.

Soil sampling

The archived soil samples collected during 2003 from the experiment were used in this study. Random samples were obtained from several locations in each plot for 0–45 cm depth using a stainless steel probe of 2.54 cm diameter, composited and divided into four depth increments. Specific depth increments considered for the present study were: 0–7.5, 7.5–15, 15–30 and 30–45 cm. Another set of undisturbed samples from each plot was also obtained for bulk density (ρ_b) measurements. Soil samples were also collected from the nearby woodlot remnant to represent the native hardwood forest. Since the archived samples were ground and passed through 2-mm sieve immediately after collection, these samples were not useful for aggregate (5–8 mm) separation. Therefore, aggregate samples were obtained from horizon-wise profile samples collected during 2005 from the pits dug down to the glacial till. Profile aggregate samples were available for both the woodlot and agricultural soils (PT and NT soils of CC treatment alone, not for the CS treatment) (Table 1).

Table 1 Horizon and depth details of profile aggregate samples

Land use	Horizon	Depth (cm)
Woodlot	A1	0–4
	A2	4–13
	E	13–28
	Bt1	41–62
No-till	Ap1	0–3
	Ap2	3–10
Continuous corn	Ap3	10–22
	Bt1	33–48
	Btx1	77–88
	Ap1	0–3
Plow-till	Ap2	3–17
	Ap3	17–31
Continuous corn	Bt1	31–44
	Btx1	70–92

Soil and aggregate fractionation

Sand-size (>53 μm), silt-size (2–53 μm) and clay-size (<2 μm) separates were obtained through particle-size fractionation. The sand fraction including the POM was separated from the silt and clay fraction by ultrasonication and wet sieving (Schöning and Kögel-Knabner 2006). Air-dried soil samples of 30 g were suspended with deionised water at a soil/water ratio of 1:5 (w/w) and dispersed ultrasonically with energy input of 60 Jml^{-1} using a probe-type sonicator. At this low sonication energy, the fragmentation of POM and its redistribution to silt and clay fractions are minimal (Balesdent et al. 1998). Subsequently, the coarse sand particles (>200 μm), including POM, were removed by wet sieving. The remaining soil water suspension was further sonicated with an energy input of 440 Jml^{-1} at a soil/water ratio of 1:10. This energy level is sufficient for the complete dispersion of a wide range of North American soils (Amelung et al. 1998). Moreover, the particle-size distribution after sonication at this energy is comparable to that obtained after standard textural analysis (Diekow 2003). Afterwards, the fine sand particles with POM occluded within microaggregates (53–200 μm) were separated by wet sieving using a 53- μm sieve. The silt and clay suspensions, which passed through the 53- μm sieve, were transferred to an automatic fractionator that separated clay from silt by sequential decantation and siphoning (Rutledge et al. 1967). The sand and silt suspensions were oven dried at 40°C; however, the clay suspension was flocculated with 0.5 M MgCl_2 and repeatedly centrifuged to reduce the volume of water before drying. The oven-dried coarse and fine sand fractions including the POM were combined and subjected to density fractionation in sodium polytungstate solution of 1.6 Mg m^{-3} density to isolate the

lighter POM from the heavier sand fraction (Diekow et al. 2005).

Aggregate fractionation was performed by the wet sieving method (Nimmo and Perkins 2002). Fifty grams of air-dried aggregates of 5 to 8 mm size were placed on top of a nest of sieves of 2000, 250 and 53 μm size; wetted by capillarity for 30 minutes; and oscillated through a vertical distance of about 3 cm at 30 oscillations per minute in a water column for 30 minutes. The fractions retained in each sieve were washed into different beakers. The soil fraction of $<0.53 \mu\text{m}$ was obtained by collecting the sediment after decanting the water and centrifuging. Thus, four aggregate-size fractions were recovered: large macroaggregates ($>2000 \mu\text{m}$), small macroaggregates (250–2000 μm), microaggregates (53–250 μm) and silt- and clay-sized aggregates ($<53 \mu\text{m}$) (Tisdall and Oades 1982). The collected fractions were oven-dried at 40°C and weighed to compute the mean weight diameter (MWD), an important index of soil structural stability. Subsamples from whole aggregates and from each aggregate fraction were ground using a mortar and pestle, sieved through a 0.125-mm sieve and stored at room temperature for C analysis.

Soil analysis

Soil ρ_b was determined by the core method (Grossman and Reinsch 2002). The total C and N concentrations in bulk soil, aggregates and physically isolated fractions were determined by the dry combustion method (Nelson and Sommers 1996) using a NC 2100 soil analyzer (ThermoQuest CE Instruments, Milan, Italy). The SOC was assumed to be equal to the total C since inorganic C concentrations were negligible and soil pH was <7 . SOC pools (Mg ha^{-1}) were calculated using ρ_b for the corresponding depth (Lal et al. 1998).

Statistical analysis

The analysis of variance for testing the treatment effects at both sites was computed using PROC GLM (fixed effects

model) of SAS (SAS Institute Inc 2002). Mean and interactive effects of treatments were separated using the *F*-protected least significant difference test. The probability level (*P*) chosen to designate the significance was ≤ 0.05 . The area of native vegetation was outside of the experimental design and, therefore, not included in the statistical analysis.

Results and discussion

Bulk soil

Soil organic carbon pool

Depth distribution of SOC pool varied significantly among tillage treatments (Table 2). The SOC pool was significantly higher for NT (18.8 Mg ha^{-1}) than CT (13.6 Mg ha^{-1}) and PT (10.2 Mg ha^{-1}) treatments for 0–7.5 cm depth. There was no significant difference in SOC pool among tillage types at 7.5–15 cm depth. At 15–30 cm depth, however, the SOC pool was significantly more in PT (19.2 Mg ha^{-1}) than CT (15 Mg ha^{-1}) and NT (14.3 Mg ha^{-1}) treatments. The SOC pool at 30–45 cm depth was also more in PT, although the difference was not statistically significant. Overall, the SOC pool under NT, CT and PT managements was 84%, 78% and 75% of that under the woodlot for 0–45 cm depth. However, calculation of the total pool below 7.5 cm depth revealed that both tilled treatments (PT and CT) accumulated more SOC than NT treatment. This observation is in support of the finding that soils under long-term NT management accumulate more SOC near the surface, while SOC pool is distributed deeper in the profile in tilled soil. Therefore, the apparent SOC gains from NT that are based only on near-surface sampling offset when deeper soil depths are also considered (Carter 2005; Dolan et al. 2006; Baker et al. 2007; Blanco-Canqui and Lal 2008). VandenBygaart et al. (2003) analyzed data from 100 published studies on the impact

Table 2 Effect of tillage and crop rotation on organic carbon pool and C/N ratio of bulk soils

Treatments	Soil depth (cm)				Soil C/N ratio			
	0–7.5	7.5–15	15–30	30–45	0–7.5	7.5–15	15–30	30–45
NV ^a	22.7	15.7	13.8	9.0	11.3	11.3	10.6	9.3
Tillage								
NT	18.8 ab	14.1 a	14.3 b	8.7 a	9.8 a	9.7 a	8.7 a	6.1 a
CT	13.6 b	14.9 a	15.0 b	8.3 a	9.1 b	9.1 b	7.9 b	5.9 a
PT	10.2 b	11.0 a	19.2 a	9.2 a	9.1 b	8.9 b	8.3 ab	6.7 a
Crops								
CC	15.9 a	15.3 a	17.6 a	10.2 a	9.9 a	9.8 a	8.4 a	6.4 a
CS	14.0 a	11.4 b	14.8 b	7.2 b	9.1 b	9.1 b	8.2 a	5.8 a

^a NV = native vegetation (woodlot)

^b Mean values in a column within a treatment type followed by the different letters are significantly different at $P \leq 0.05$ using Fisher's protected least significant difference

of tillage on SOC sequestration in Canada and concluded that when sampling depth was ≤ 30 cm, most of the experiments reported more SOC in NT than in the tilled plots, whereas when the profile was sampled to ≥ 30 cm, NT treatment registered less SOC relative to conventional tillage.

The effect of crop rotation on SOC pool was significant in all depth increments except for the 0–7.5 cm depth, with higher SOC pool in CC than in CS: 15.3 vs 11.4 Mg ha⁻¹ for 7.5–15 cm, 17.6 vs 14.8 Mg ha⁻¹ for 15–30 cm and 10.2 vs 7.2 Mg ha⁻¹ for 30–45 cm. The reduced amount of residues returned to soil coupled with higher decomposition rate may be the reasons for lower SOC accumulation under soybean (Huggins et al. 2007).

Soil C/N ratio

The soil C/N ratio was significantly higher in NT than CT and PT treatments for 0–15 cm soil depth: 9.8 (NT) > 9.1 (CT and PT) for 0–7.5 cm and 9.7 (NT) > 9.1 (CT) = 8.9 (PT) for 7.5–15 cm (Table 2). The lower C/N ratio of tilled soils can be attributed to the positive relationship between the extent of SOM decomposition induced by tillage operations and the N enrichment in SOM (Kramer et al. 2003). The crop rotation also had a significant influence on soil C/N ratio for 0–15 cm depth, with significantly higher values under CC (9.9 for 0–7.5 cm and 9.8 for 7.5–15 cm) than CS (9.1 at both 0–7.5 cm and 7.5–15 cm depths). The lower C/N ratio of CS system may be due to the fixation of atmospheric N by soybeans and its enrichment in SOM through long-term residue addition to soil. The C/N ratio of agricultural soils, regardless of the tillage and crop rotation types, tended to be narrower than that of woodlot soils. Such a trend in C/N ratio can be explained by the application of inorganic N in agricultural plots and consequent increase in the retention of N in SOM (Guzman et al. 2006).

Particle-size fractions

Soil organic carbon in particle-size fractions

Irrespective of the treatment differences, the SOC concentration was significantly the highest in POM fraction (Table 3), although POM constitutes only 0.1–1.9% of total soil mass. Within 0–30 cm depth, the SOC concentration in the POM fraction ranged from 101 to 241 g kg⁻¹ for the agricultural soil, as compared to 308–369 g kg⁻¹ for the respective soil under native vegetation. This showed that substantial portion of SOC associated with POM was depleted upon conversion from native to agricultural ecosystems. Mueller and Kögel-Knabner (2009) also reported highest POM contents in soils of the forested

sites. Among the primary soil particles, a significantly higher amount of SOC was associated with the clay-sized particles followed by silt- and sand-sized (without POM) separates. The SOC concentration was the highest in clay fraction because clay minerals are the most active constituents in the formation of organo-mineral complexes because of high charge and specific area (Chenu et al. 2006). The higher SOC accumulation in clay minerals favors more vigorous biological activity leading to the formation of more microbial metabolites and residues, which are relatively less susceptible to decomposition (Kaiser et al. 1998). The highest proportion of SOC in clay fraction was also reported for this site after 30 years of NT by Haile-Mariam et al. (2008). On the other hand, the capacity of sand particles to hold SOC as organic coatings remains insignificant (Christensen 2001). Nonetheless, the presence of POM can contribute significantly to the SOC concentration of sand-sized separates.

Particle-associated SOC pool among agricultural treatments

The effect of management on concentration and pool of particle-associated SOC were similar. Therefore, only the data on SOC pool are presented herein. The POM-derived SOC pool was significantly higher for CT and NT than PT soil in both the 0–7.5 cm surface layer [1.4 Mg ha⁻¹ (NT) = 1.6 Mg ha⁻¹ (CT) > 0.75 Mg ha⁻¹ (PT)] and in the 0–30 cm profile [3.2 Mg ha⁻¹ (NT) = 3.7 Mg ha⁻¹ (CT) > 2.4 Mg ha⁻¹ (PT)] (Fig. 1a). The POM fraction mainly consists of partially decomposed plant residues. The increase in POM-derived SOC with decrease in tillage intensity may be because of slow decomposition of crop residues due to lower or no soil disturbance and protection of POM in aggregates (Tan et al. 2007; Álvaro-Fuentes et al. 2008). The POM-associated SOC pool for 30–45 cm depth was not accounted for because the recovery of POM fraction for this depth was insufficient for a reliable measurement of C using the dry combustion method. Between the two cropping systems, POM-derived SOC pool was significantly higher for CC than CS system in both 0–7.5 cm soil depth [1.8 Mg ha⁻¹ (CC) > 0.74 Mg ha⁻¹ (CS)] and 0–30 cm profile [4.4 Mg ha⁻¹ (CC) > 2.1 Mg ha⁻¹ (CS)] (Fig. 1b).

Forty-five years of tillage and cropping system treatments significantly influenced the silt- and clay-associated, but not the sand-associated (without POM), SOC pool (Fig. 2). Although the soil was dominated by the silt fraction (44–61%) (Table 3), the highest proportion of SOC was associated with the clay fraction. Compared to NT and CT treatments, intensive PT management had the lowest clay-associated SOC pool in the surface layers [5 Mg ha⁻¹ (PT) < 6.5 Mg ha⁻¹ (CT) < 7.4 Mg ha⁻¹ (NT) for 0–7.5 cm,

Table 3 Soil organic carbon concentration of primary particle fractions

Treatments		Particle fraction	0–7.5cm SOC (gkg ⁻¹)	7.5–15cm SOC (gkg ⁻¹)	15–30cm SOC (gkg ⁻¹)	30–45cm SOC (gkg ⁻¹)
Native woodlot		Sand	10.5 c ^a	5.1 c	3.8c*	3.3 c*
		Silt	22.5 c	13.4 c	5.7 c	3.2 c
		Clay	55.4 b	43.9 b	2.08 b	1.03 b
		POM	307.8 a	353.8 a	369.3 a	406.4 a
PT	CC	Sand	1.6 d	1.7 d	1.3 c	0.67 b*
		Silt	6.5 c	5.9 c	5.1 c	1.9 b
		Clay	31.8 b	31.4 b	24.0 b	7.9 a
		POM	124.9 a	129.3 a	188.4 a	nd
PT	CS	Sand	1.3 c	1.2 c	1.1 c	0.8 b
		Silt	4.8 c	4.4 c	3.1 c	1.5 b
		Clay	25.6 b	25.4 b	18.0 b	6.3 a
		POM	100.8 a	120.1 a	160.0 a	nd
CT	CC	Sand	1.9 d	2.8 c	1.4 c*	0.76 b*
		Silt	7.5 c	6.1 c	2.7 c	1.6 b
		Clay	39.7 b	36.1 b	15.2 b	7.9 a
		POM	140.0 a	190.1 a	240.9 a	nd
CT	CS	Sand	1.8 c	2.7 c	1.9 c*	1.0 b*
		Silt	5.6 c	5.4 c	2.8 c	1.6 b
		Clay	35.1 b	32.5 b	17.9 b	8.0 a
		POM	141.0 a	120.4	190.0 a	nd
NT	CC	Sand	2.1 d	2.7 c	2.3 c*	1.3 b*
		Silt	10.0 c	5.5 c	3.2 c	2.0 b
		Clay	53.0 b	36.9 b	23.7 b	10.7a
		POM	180.1 a	226.0 a	211.0 a	nd
zNT	CS	Sand	4.5 c	2.3 c	1.5 c	0.7 b*
		Silt	8.9 c	5.4 c	2.3 c	1.4 b
		Clay	49.5 b	36.4 b	17.4 b	8.2 a
		POM	185.0 a	189.2 a	220.4 a	nd

Mean values in a column within a land use type followed by the different letters are significantly different at $P \leq 0.05$ using Fisher's protected least significant difference.

P is < 0.0001 except where * = 0.0001–0.001

nd not determined

and 5.7 Mg ha⁻¹ (PT) < 6.5 Mg ha⁻¹ (CT and NT) for 7.5–15 cm] and the highest SOC pool in the 15–30 cm depth [9.7 Mg ha⁻¹ (PT) ≥ 8.8 Mg ha⁻¹ (NT) ≥ 7.3 Mg ha⁻¹ (CT)] (Fig. 2a). Tillage treatments had no effect on clay-associated SOC pool in 30–45 cm depth. However, effect of tillage on SOC associated with the silt fraction also followed a pattern similar to that of the clay fraction, with SOC pool being more in the subsoil layers of tilled than NT management. Overall, for the 0–45 cm soil profile, the clay-associated SOC pool decreased with increase in tillage intensity [28.3 Mg ha⁻¹ (NT) ≥ 25.2 Mg ha⁻¹ (CT) ≥ 24.9 Mg ha⁻¹ (PT)]. Due to the more humified and recalcitrant nature of mineral-associated than POM-derived SOC, greater clay-bound SOC in the 45-cm deep profile of NT than tilled soils implies more stabilization of SOC. Cambardella and Elliot (1992) also reported more mineral-bound SOC under NT than in a bare fallow treatment tilled with moldboard plow to 20 cm depth.

Soils under CC system contained significantly more silt- and clay-associated SOC than CS system with mean values

in the order of: 4.5 Mg ha⁻¹ (CC) > 3.7 Mg ha⁻¹ (CS) for 0–7.5 cm, 3.8 Mg ha⁻¹ (CC) > 3.1 Mg ha⁻¹ (CS) for 7.5–15 cm, 4.9 Mg ha⁻¹ (CC) > 3.5 Mg ha⁻¹ (CS) for 15–30 cm and 15.3 Mg ha⁻¹ (CC) > 12.0 Mg ha⁻¹ (CS) for 0–45 cm for silt-associated pool compared with 6.5 Mg ha⁻¹ (CC) > 6.1 Mg ha⁻¹ (CS) for 0–7.5 cm, 6.7 Mg ha⁻¹ (CC) > 5.8 Mg ha⁻¹ (CS) for 7.5–15 cm, 9.3 Mg ha⁻¹ (CC) > 8.0 Mg ha⁻¹ (CS) for 15–30 cm and 27.8 Mg ha⁻¹ (CC) > 24.6 Mg ha⁻¹ (CS) for 0–45 cm for the clay-associated pool (Fig. 2b).

Carbon sink capacity of mineral particle-size fractions

It is well established that fine-textured fractions contain more SOC than heavy-textured fractions (Theng 1979; Jenkinson 1988). However, relatively little is known about the sink capacity of different soil fractions. Hassink et al. (1997) suggested that the capacity of soil fractions to protect SOC is rather limited. In soils where the protective capacity is saturated, the newly added SOC remains in a

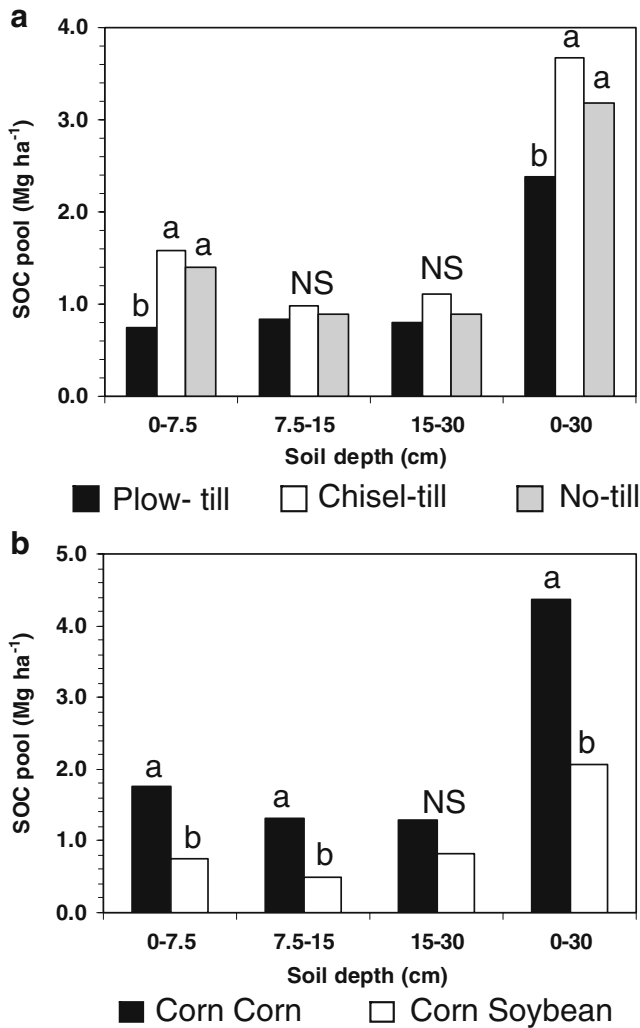


Fig. 1 Particulate organic matter-associated carbon pools as affected by tillage and cropping systems. **a** Effect of tillage. **b** Effect of cropping systems. Different letters on top of the histogram within a depth class indicates statistically different values at $P \leq 0.05$ using Fisher's protected least significant difference. *NS* nonsignificant

biologically available form and is mineralized (Krull et al. 2003).

The capacity of soil mineral fractions to protect SOC was assessed by plotting the SOC concentration associated with the sand, silt and clay fractions against the corresponding SOC concentration of the whole soil for the undisturbed as well as agricultural soils (Fig. 3). An exponential rise to maximum model fitted best for the clay-size fraction, and linear models fitted appropriately for the sand- and silt-sized fractions. For soils under woodlot, there was no increase in clay-associated SOC when total SOC concentration was higher than $\sim 30 \text{ g kg}^{-1}$. Rather, it led to a linear increase in the SOC- associated with sand and silt fractions (Fig. 3a). Such a trend indicated that the clay fraction of soil under native woodlot may have already attained its C sink capacity, and further accrual is possible

only in silt and sand fractions (Diekow et al. 2005). It is also likely that the SOC initially associated with the clay surface may impede further binding of recently formed organic compounds (Christensen 2001). Since the sand

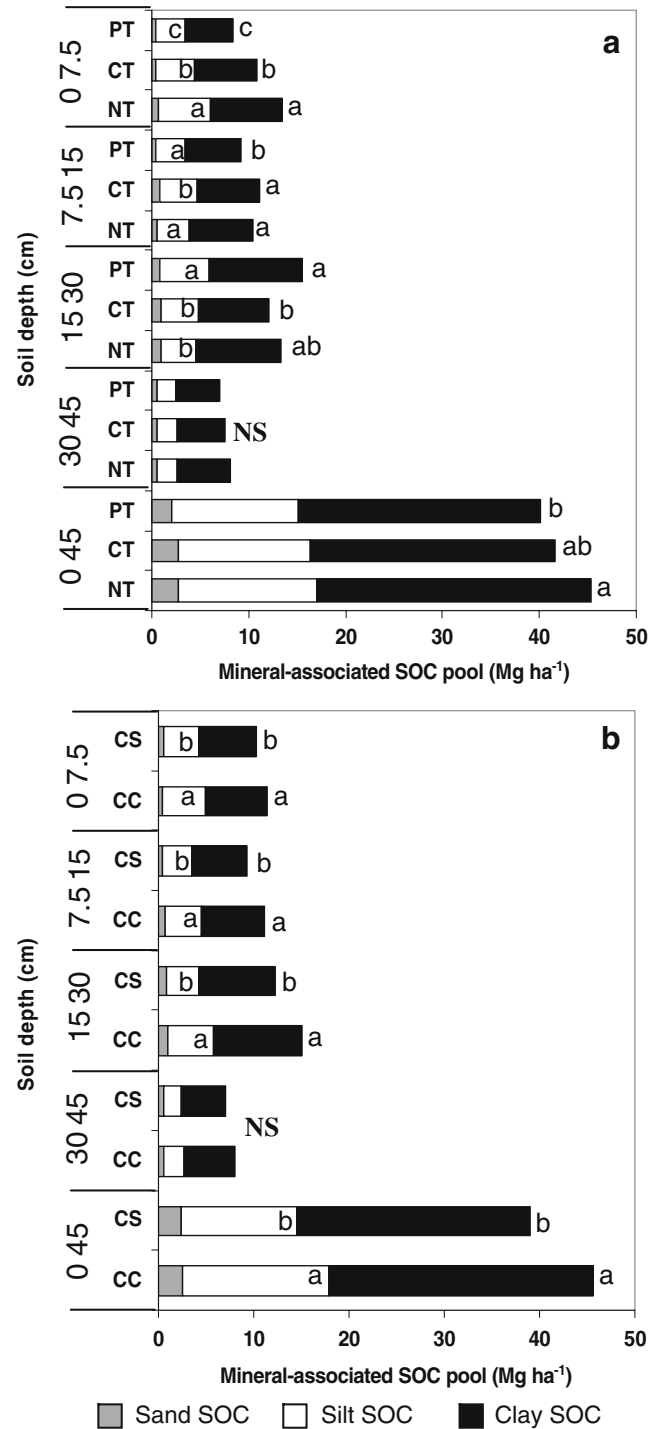
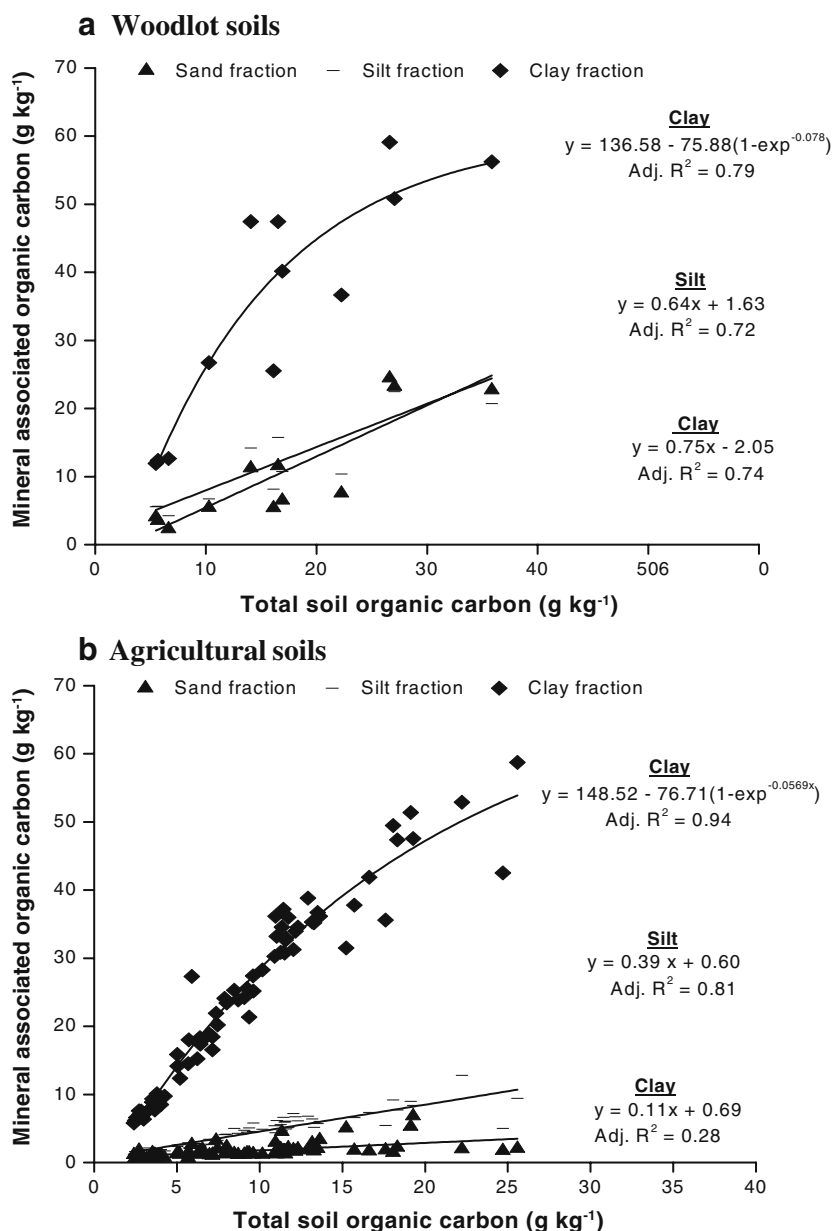


Fig. 2 Mineral-associated carbon pools as affected by tillage and cropping systems. **a** Effect of tillage. **b** Effect of cropping systems. Different letters on the histogram within a depth class indicates statistically different values among tillage/cropping systems treatments at $P \leq 0.05$ using Fisher's protected least significant difference. *NS* nonsignificant

Fig. 3 Relationship between mineral-associated soil organic carbon concentration and total soil organic carbon concentration of whole soil



particles are inherently weaker in binding organic compounds, the observed SOC enrichment in sand fraction, especially at higher bulk soil SOC level, might be due to possible retention of relatively heavier POM fraction in sand separates during the density separation process. Mineral and organic components of soil have a range of densities, and no one density can completely separate the POM from mineral-bound SOC (Oades 1989). The density of sodium polytungstate used in this study (1.6 Mg m^{-3}) was probably insufficient for the complete recovery of POM from the sand fraction. The indication of SOC saturation, as observed in the clay fraction of soils under woodlot, did not hold true in the clay fraction of agricultural soils (Fig. 3b). This observation is consistent

with the fact that the total SOC concentration of agricultural soils is well below the upper limit of saturation for the clay fraction under woodlot ($\sim 30 \text{ g kg}^{-1}$). Nonetheless, the rate of increase of SOC associated with the clay fraction in agricultural soils decreased with increase in total SOC concentration of the bulk soil. Therefore, with any further increase in SOC concentration of agricultural soils, the clay fraction may attain C saturation with attendant accumulation of SOC in the silt fraction.

Aggregate fractions

As discussed in “Soil sampling” section, the aggregates were not archived from the depth-wise samples collected in

2003. Therefore, we used another set of aggregate samples that were collected horizon-wise from the same treatments in 2005. The aggregate samples were available only for soils under the CC system (PT and NT soils). Therefore, discussion of aggregate fractions is limited to soils under the CC system.

Aggregate size distribution and stability

Both aggregate size distribution and MWD were significantly influenced by tillage treatments in all horizons except Bt1 (Table 4). The soils under NT management had significantly higher large macroaggregates (>2000 μm) compared to PT management: 83% (NT) vs 19% (PT) in Ap1, 71% (NT) vs 15% (PT) in Ap2 and 36% (NT) vs 9% (PT) in Ap3 horizon. Relative increase in the formation of large macroaggregates in NT soils can be attributed to the increased amounts of residue retention (De Gryze et al. 2005; Govaerts et al. 2006; Lichter et al. 2008). Relative abundance of other smaller aggregate-size fractions (small macroaggregates, 250–2000 μm; microaggregates, 53–250 μm; and silt and clay-sized aggregates, <53 μm) was significantly higher in PT than NT treatments. Consequently, MWD of aggregates was also significantly higher for NT than PT soils: 4.3 mm (NT) vs 1.4 mm (PT) in Ap1, 3.8 mm (NT) vs 1.2 mm (PT) in Ap2 and 2.2 mm (NT) vs 1.0 mm (PT) in Ap3 horizons. These results support the hypothesis that the transient binding agents of macroaggregates are disrupted by tillage-induced disturbance (Elliott 1986; Six et al. 2000b; Olchin et al. 2008). In addition, both size distribution pattern and MWD of

aggregates under NT soils were mostly similar to that of the soils under woodlots, indicating that aggregate dynamics may have reached a steady state due to >40 years of continuous NT management at this site.

Aggregate-associated SOC

Tillage treatment significantly affected C associated with aggregates in Ap1 and Ap2 horizons of soils (Fig. 4). In these horizons, the SOC concentrations were significantly higher (1.5–2.8 times) in all aggregate classes under NT compared to PT. No significant differences in SOC concentration of these aggregate classes were observed among NT and CT in subsoil horizons (Ap3 and Bt1 horizons). In general, the SOC concentration of aggregates of both tillage types increased with increase in size of aggregates, with more pronounced difference under NT management. These results agree with the concept of aggregate hierarchy model: microaggregates are bound together into macroaggregates by transient (i.e., microbial- and plant-derived polysaccharides) and temporary binding agents (roots and fungal hyphae) (Tisdall and Oades 1982; Six et al. 2000a). Consequently, macroaggregates are more enriched in SOC than microaggregates because they are essentially composed of smaller aggregates and the intra-aggregate organic binding agents (Elliott 1986). Progressive depletion of SOC with increasing tillage intensity is partly attributed to decrease in the proportion of SOC-enriched macroaggregates (Elliott 1986; Six et al. 2000a). The SOC concentration in the aggregates of surface layers of NT soils was almost similar to those of soils under woodlot. In

Table 4 Distribution of aggregate-size fractions and mean weight diameter of aggregates

Horizon	Treatments	Proportion of aggregate-size fractions (%)				MWD (mm)
		>2000μm ^a	250–2000μm	53–250μm	<53μm	
A1/Ap1	NV ^b	84	11	3	2	4.3
	Tillage					
	NT	83 ^a ^c	12 ^b	2 ^b	3 ^b	4.3 ^a
A2/Ap2	PT	19 ^b	38 ^a	21 ^a	22 ^a	1.4 ^b
	NV	81	13	2	4	4.2
	Tillage					
E/Ap3	NT	71 ^a	19 ^b	6 ^b	4 ^b	3.8 ^a
	PT	15 ^b	40 ^a	20 ^a	25 ^a	1.2 ^b
	NV	31	43	11	15	2.0
Bt1	Tillage					
	NT	36 ^a	31 ^b	17 ^b	16 ^b	2.2 ^a
	PT	9 ^b	41 ^a	24 ^a	26 ^a	1.0 ^b
Bt1	NV	3	52	21	24	0.8
	Tillage					
	NT	6 ^a	27 ^a	25 ^a	42 ^a	0.61 ^a
	PT	4 ^a	33 ^a	25 ^a	38 ^a	0.58 ^a

^a >2000 μm—large macroaggregates, 250–2000 μm – Small macroaggregates, 53–250 μm—microaggregates, <53 μm—silt- and clay-sized aggregates

^b NV = woodlot, representing native vegetation

^c Mean values in a column within tillage treatment followed by the different letters are significantly different at $P \leq 0.05$ using Fisher's protected least significant difference

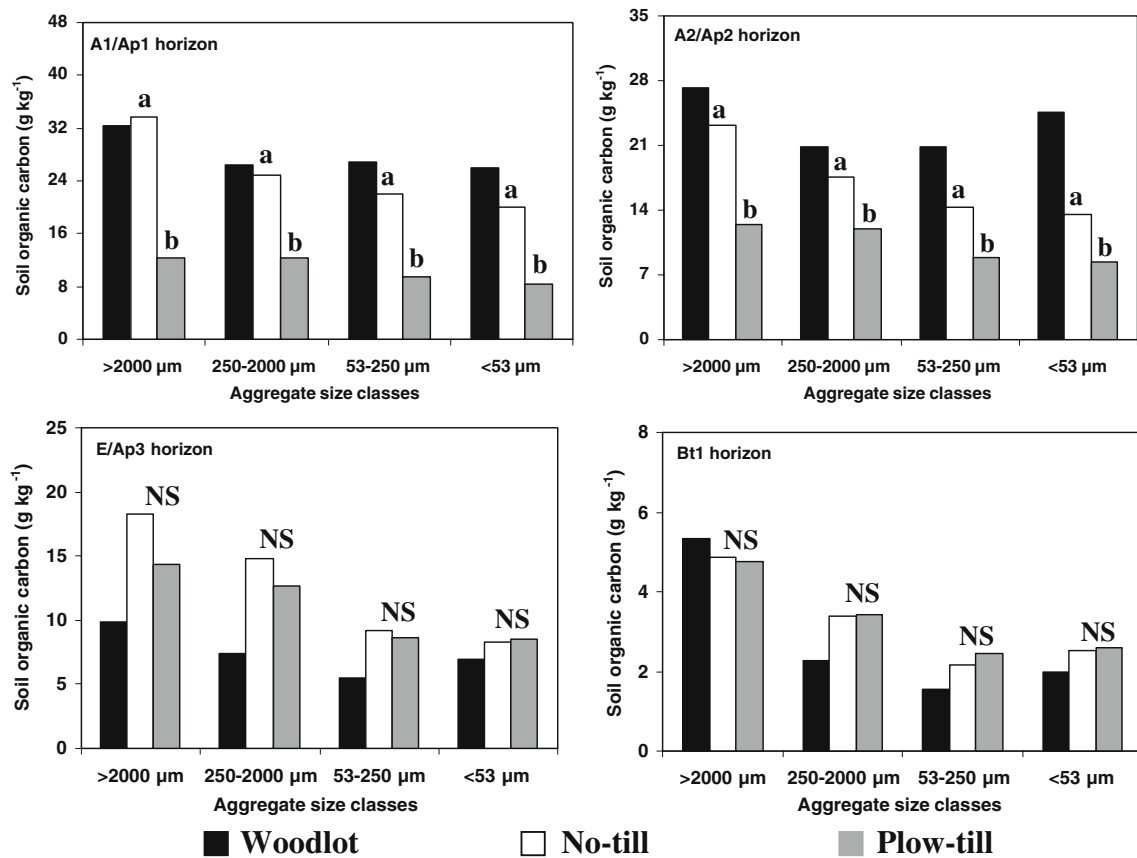


Fig. 4 Horizonwise distribution of SOC concentration in aggregate-size classes. Different letters on top of the histogram of each figure (within an aggregate-size class) indicates statistically different values

at $P \leq 0.05$ using Fisher’s protected least significant difference. Woodlot site was outside of statistical design, hence not included in statistical analysis. *NS* nonsignificant

addition, the aggregate-associated SOC in E horizon of the soils under woodlot was lower than the corresponding horizon (Ap3) of agricultural soils as a consequence of the process of elluviation in E horizon. Both large and small

macroaggregates of PT soils contained more SOC in the Ap3 horizon than those in the surface layers. This trend of redistribution of SOC from surface to deeper layers of PT soils was also reflected in the total SOC pool at this depth interval (Table 2).

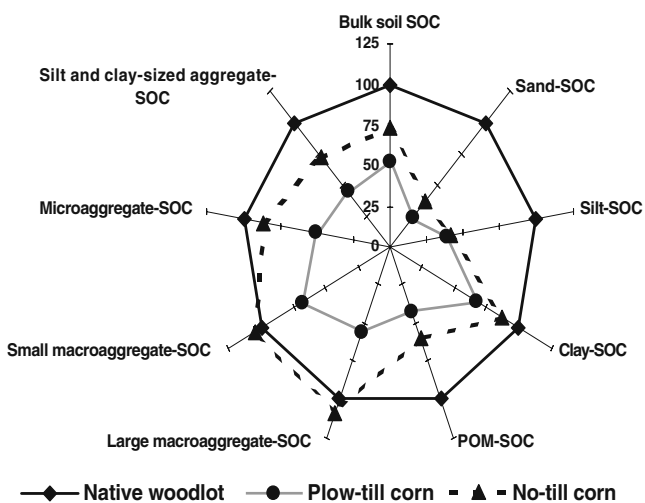


Fig. 5 Radar plot comparing the SOC associated with physical fractions for soils under continuous corn (plow-till and no-till) in relation to soils under native vegetation

Comparative evaluation of SOC associated with all physical fractions

Radar graph was plotted to visualize changes in SOC associated with physical fractions of soils and aggregates under different tillage managements vis-à-vis in soils under native vegetation (Fig. 5) for 0–30 cm depth. This analysis was done for soils under the CC system only because such data for aggregates were not available for soils under CS system. The SOC concentration for physical fractions in soil under native vegetation (woodlot) was set at 100% and values for the tillage treatments (PT and NT) were calculated in percentage thereof. The PT management resulted in a substantial loss of SOC associated with all fractions of soil and aggregates. The NT management for 45 years, however, resulted in better aggregate formation and therefore more SOC encapsulation in aggregates than

the PT management. Furthermore, concentrations of SOC associated with large and small macroaggregates of soils under NT management were more than those under woodlots. This trend shows that, among the physical fractions, the SOC associated with sand (without POM) and silt fractions quickly changed with land use conversion, which can be attributed to the weaker bonding relationship of these soil separates with organic compounds. This result supports our hypothesis that analysis of SOC stabilized in particle-size fractions is more useful than bulk SOC for capturing detailed and sudden changes in SOC dynamics induced by land use and managements.

Conclusions

This study focused on a detailed evaluation of SOC associated with POM, mineral particles and aggregates. The study also attempted to revisit Hassink's concept of finite capacity of soils to protect SOC. The results revealed that patterns of mineral-associated SOC accumulation with increase in total SOC concentration were different for different-sized primary particles. The clay fraction of the native soils showed an indication of SOC saturation with increase in bulk soil SOC, but that of agricultural soils continues to accumulate more SOC, albeit at a slower rate. Both sand- (without POM) and silt-associated SOC increased linearly with increase in total SOC concentration. Further, the SOC protection within macroaggregates after several decades of NT management was even higher than that in the nearby undisturbed soils. Thus, rather than quantifying the total SOC pool, a thorough understanding of the management-induced changes in SOC sequestration warrants consideration of SOC accumulation at all levels of structural and functional complexity (POM, primary organo-mineral complexes and secondary organo-mineral complexes) distinguished by physical fractionation techniques.

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