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QUANTIFYING LABORATORY AND FIELD VARIABILITY TO ASSESS POTENTIAL FOR CARBON SEQUESTRATION

R. A. Bowman,[1,*] J. D. Reeder,[2] and B. J. Wienhold[3]

[1]Soil Science, USDA-ARS, Akron, CO, 80720
[2]Soil Science, USDA-ARS, Crops Research Laboratory, 1701 Centre Ave., Fort Collins, CO, 80526
[3]Soil Science, USDA-ARS, 122 Keim Hall, University of Nebraska, Lincoln, NE, 68583

ABSTRACT

Accurate measurements of soil organic carbon (SOC) levels are essential to assess changes in C sequestration rates. To this end we conducted studies to evaluate laboratory variability in SOC concentration measured at USDA-ARS laboratories in Akron, CO, Cheyenne, WY, and Lincoln, NE. At the Akron laboratory we also evaluated field spatial variability within common cropping treatments in order to assess the potential to quantify significant changes in SOC content associated with rotations of varying cropping intensities. Our data showed very low coefficients of variation for SOC values from each of the three laboratories, and the same average SOC values for soils from each treatment. For mitigating spatial variability, the data showed that a 10-ha field required 10 cores, the 0.2-ha field, 2 cores, and the 0.02-ha field, one core, in order to achieve a difference of ≤ 10% from the mean

*Corresponding author. E-mail: rbowman@lamar.colostate.edu

95% of the time. With respect to cropping intensity, all rotations with fallow contained statistically the same SOC levels, with the continuous cropping treatment [winter wheat-(*Triticum aestivum* L.)–corn-(*Zea mays* L.)–proso millet (*Panicum miliaceum* L.) showing higher levels of SOC than the conventional-till winter wheat summer fallow and the wheat–sunflower (*Helianthus annuus* L.)–fallow. Data indicate that net changes in SOC content over time as a result of management will be very difficult to assess, and will require a sufficient minimum elapsed time, as well as great attention to sampling protocol.

INTRODUCTION

Reliable quantification of SOC is necessary if we are to reward good cropland stewardship and conservation through carbon (C) credits, and assess the potential to meet the Kyoto Protocol (7% reduction from 1990 levels of greenhouse gases between 2008 and 2012). Besides the benefits of reducing erosion and conserving water through adequate crop residue production and management (e.g., no-till practices, increasing the cropping intensity, adequate fertilization), SOC has been estimated to have a realistic value of $10 to $20 per ton (1,2). To implement a C credit program, we need an agreed upon methodology for determining verifiable changes in SOC stock (levels). Essentially, we need to be able to accurately measure existing C levels, and to determine how small a change can be measured over time. In order to do this, we need to quantify laboratory and field spatial variability for SOC and bulk density. Changes in these parameters need to be accurately assessed over time for the same treatment (rotation), or for a given time where different management treatments are compared.

Soil spatial variability is an insignificant factor in the development of large scale regional or global C budgets which evaluate differences in C sequestration among diverse cropping systems or different soil types (2,3–5). Spatial variability also is less important in evaluations of large losses in SOC content after decades of tillage of the same sites (6–8). However, for rewarding C credits, field spatial variability in SOC and variability in laboratory analysis of SOC are critical factors in accurately determining small but significant changes in SOC content as a result of management practices.

To quantify variability in SOC laboratory analyses, we evaluated differences in SOC concentration at three different laboratories (Akron, CO, Cheyenne, WY, and Lincoln, NE) measured on the same soil samples. Additionally, at the Akron laboratory we measured field spatial variability of SOC in common cropping rotations (replicate plots of the same treatment), and changes in SOC due to five different rotations (treatment variability differences) compared to the traditional winter wheat–summer fallow rotation, and an existing adjacent native sod.

The objectives of this study, therefore, were two-fold: first, to determine inter- and intra-laboratory variation in SOC measurements, and second, to assess SOC content across a range of cropping treatments by using three different SOC methods at the Akron location.

MATERIALS AND METHODS

Laboratory Variability

Soil samples (0–15 cm depth) for inter- and intra-laboratory comparisons of SOC variability were taken in the spring of 2000 from an alternative cropping rotation study on a Weld silt loam (fine, smectitic, mesic Aridic Paleustolls) at the Central Great Plains Research Station, Akron CO (9–11). Twenty-one different soil samples were sent to the three laboratories, representing all phases of six different cropping rotations and an adjacent native sod, with each field treatment replicated three times. The six cropping rotations were winter wheat-fallow (W–F), winter wheat–corn-fallow (W–C–F), winter wheat–corn–proso millet–fallow (W–C–M–F), winter wheat–sunflower-fallow (W–Sun–F), W–C–Sun–F, and W–C–M. All rotations were no-tilled except for the W–F which was conventionally tilled. The sunflower rotations received one tillage for herbicide incorporation (12), but this practice was discontinued in 1998 when the rotation became fully no-tilled. The adjacent native sod, established in the 1940s on go-back land, is dominated by blue grama (*Bouteloua gracilis*), buffalo grass (*Bucloe dactyloides*), and *Stipa* species.

Twelve soil cores were taken systematically from 30 m × 10 m plots and composited for each rotation (e.g., W–C–F) with each phase of the rotation in a block or replicate contributing equally [(e.g., W–C–F, 4 cores; C–F–W, 4 cores; and F–W–C, 4 cores) (see Fig. 1 in Bowman and Halvorson (9))]. The 21 composited soil samples were air-dried, screened through a 2-mm sieve, and thoroughly mixed representative samples sent to all three laboratories.

All three laboratories determined SOC concentration of finely-ground soil samples (approximately 20 mg) with similar model Carlo-Erba[1] C–N gas analyzers (13). Each laboratory had its own protocol and standards. Samples were not ground nor root materials removed before sending to cooperating

[1]Trade names are included for the benefit of the reader and do not imply endorsement or preferential treatment by USDA-ARS.

laboratories. Each laboratory was asked to assess the variability of SOC content with five replicate analyses of each soil sample determined on at least two separate dates. All three laboratories reported mean SOC concentrations and coefficients of variation for the 21 soil samples. The Akron laboratory additionally conducted SOC analysis on the 21 samples by using a chromic acid oxidation (modified Walkley–Black, W–B) (14) procedure, and a loss of organic matter on ignition (LOI) procedure (15). For the W–B procedure 0.20 g soil sample size was used, and for the LOI, 10 g. For the latter procedure, samples were dried at 105°C for 24 hr, then at 400°C for 2 hr. SOM was calculated by difference (105–400°C), and a factor of 0.6 was used to calculate the SOC. For the W–B procedure, after oxidation of SOM with 2.5 mL of 1.00 N $K_2Cr_2O_7$ and 5 mL of concentrated H_2SO_4 for 0.5 hr digestion at 135°C, samples were cooled, made to a volume of 25 mL, centrifuged at 10,000 rpm (radius = 10 cm), and the clear extract measured at 625 nm against glucose–carbon standards.

Spatial Variability

For an assessment of soil spatial variability, two field sites were used: one from the above-mentioned alternative crops study, and a second (same soil characteristics) with much larger experimental plots (Fig. 1). The second site

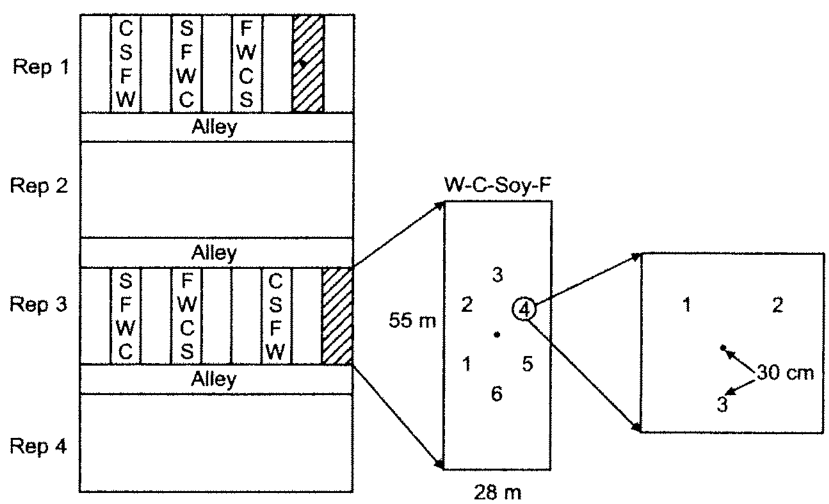


Figure 1. Representation of sampling scheme for assessing field spatial variability of soil organic carbon within a cropping rotation including wheat (W), corn (C), soybeans (S), and fallow (F).

contained three different rotations (W–F, W–C–F, and W–C–Soy (*Glycine max.* L. *merr*)–F replicated four times with all its rotation phases present every year (nine plots per replicate). Each plot was 28 m × 55 m. Two of the four replicates of the W–C–Soy–F rotation phase were assessed for spatial variability by sampling seven sites within each plot. Six sites in an oval pattern surrounding one central location were sampled, and each sample was comprised of three composite cores taken 30 cm apart (Fig. 1). Additionally, the same rotation with all its phases (16 plots) was assessed for spatial variability by using only the center soil core site.

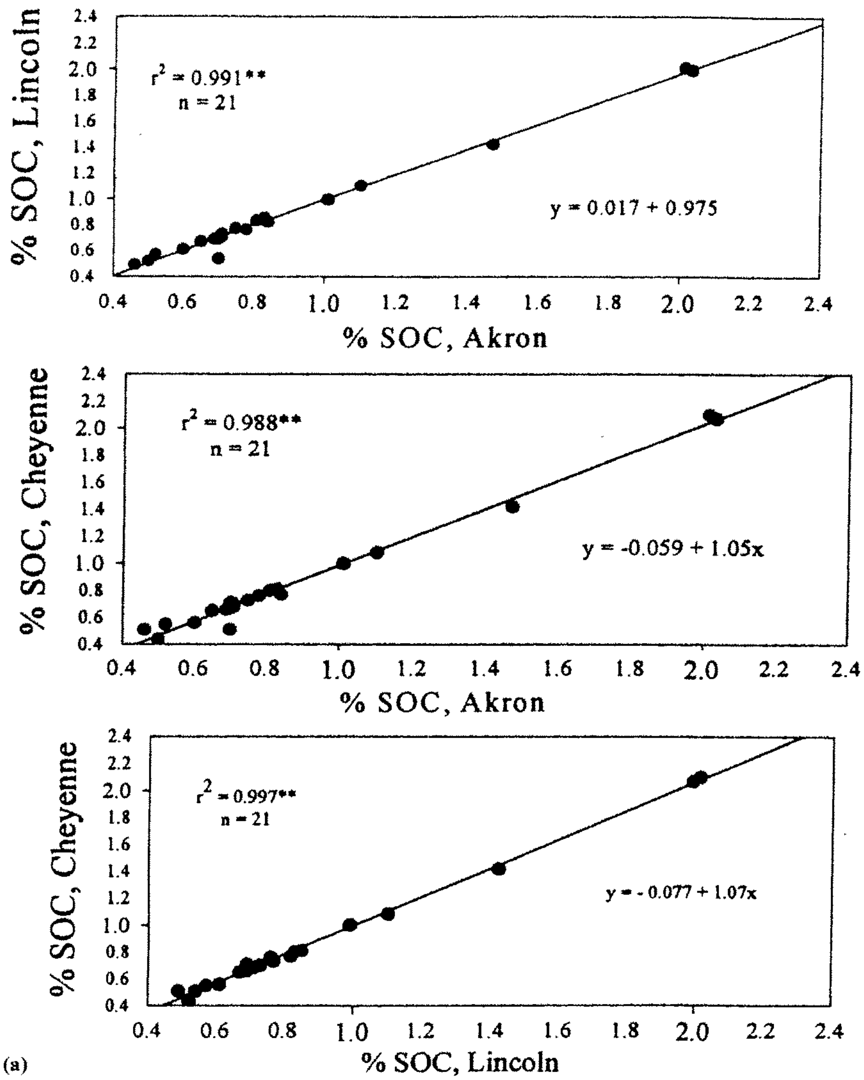
Statistics

Coefficients of variation and of linear determination were calculated for results from each of the three laboratories. The number of sample replicates needed to achieve a 10% error from the mean 95% of the time was also determined according to the following formula: $n = (cv^2)(t^2)/e^2)$ where n is the number of replicates necessary for a 10% error (e) from the mean, and t is Student's t_{05}. For cropping intensity rotations, analysis of variance was conducted at the 10% level (Tukey's mean difference).

RESULTS AND DISCUSSION

Laboratory Variability

If C credits are to be assessed regionally, the importance of consistency and good reproducibility among laboratories is critical. All three laboratories showed excellent agreement and reproducibility for SOC concentration for the 21 soil samples obtained from the various cropping rotations and native sod (Fig. 2a). With the exception of one sample from the Akron lab, correlation of determination exceeded 95% with nearly a 1:1 relationship (intercept of zero and slope of 1.0) for the three comparisons with the C–N gas analyzers. The outlier sample could have been reanalyzed by the Akron laboratory to determine whether an error was made in sample selection or preparation, but it was determined that without the data from the other two laboratories, this discrepancy would not have been known, and, therefore, the data is accepted as valid as would have been the case with most commercial laboratories. Coefficients of variation (Table 1) were generally less than 3% for the 10 subsamples from the six different rotations and the native sod, except for the "Day 1" run at the Akron station which was performed by a summer student helper who had minimal experience with the instrument.



(a)

Figure 2. (a) Comparison of soil organic C concentration measured by three laboratories using similar C–N gas combustion analyzers. (b) Comparisons of soil organic C concentration measured by C–N gas combustion analyzer (CN), Walkley–Black chromic acid oxidation (W–B), and loss-on-ignition (LOI).

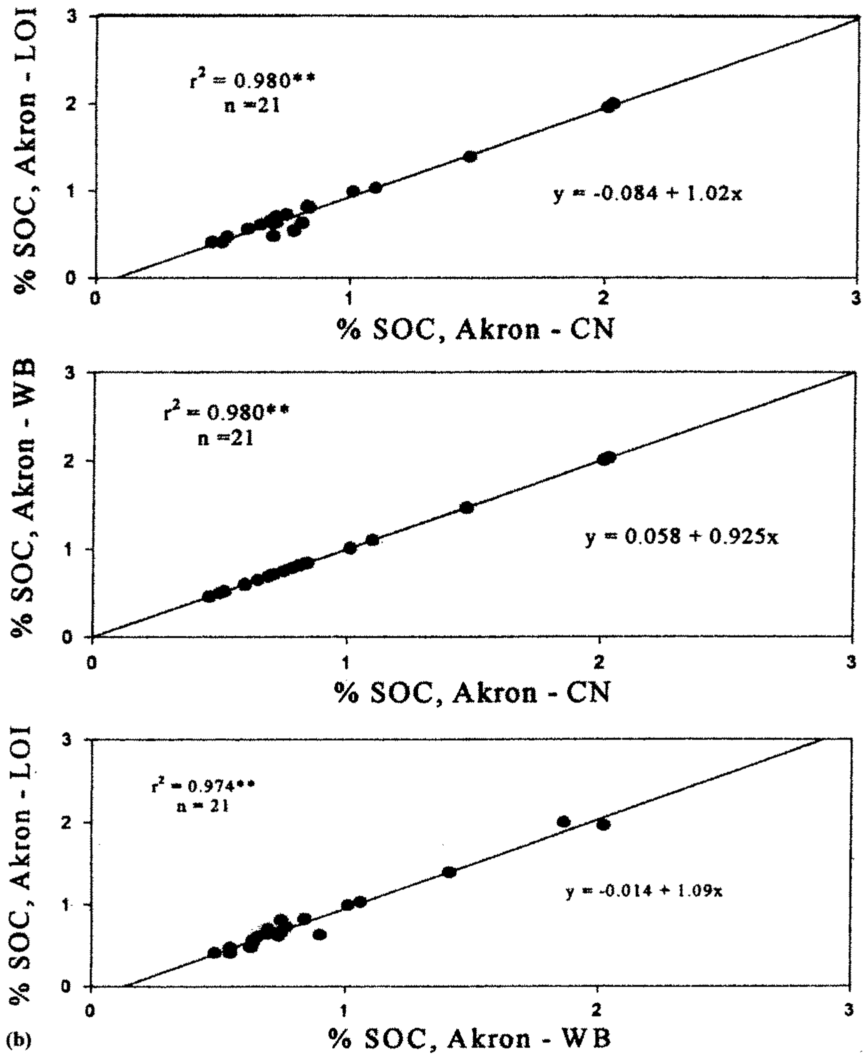


Figure 2. Continued.

Results from the W–B and the LOI methods compared reasonably well with those from the C–N gas analyzer values determined at the Akron station (Fig. 2b). These other procedures were included since many laboratories outside the industrialized countries still use one or the other of these methods. The W–B procedure also is especially useful where only a few soil samples need to be

Table 1. Reproducibility of Soil Organic Carbon Concentration as Assessed by Coefficients of Variation (CV) of 6 Different Soil Rotation Samples and a Native Sod Run at Least 5 Times on Two Different Days

	Laboratories (%CV)					
	Akron		Cheyenne		Lincoln	
Rotations[a]	Day 1[b]	Day 2	Day 1	Day 2	Day 1	Day 2
W-F	7.1	0.9	3.2	1.0	3.9	2.1
W–C-F	3.9	2.6	2.6	1.4	1.1	1.5
W–Sun-F	8.1	1.8	2.8	0.6	0.8	1.2
W–C–M-F	1.9	0.7	1.0	1.3	1.2	1.3
W–C–Sun-F	3.2	1.1	2.0	1.4	0.7	1.6
W–C–M	3.7	3.2	1.2	2.0	0.5	1.4
Rotation means	4.6	1.7	2.1	1.3	1.4	1.5
Sod	3.0	1.1	1.9	4.1	1.6	1.8

[a] W is wheat; C is corn; M is proso millet; Sun is sunflower; F is fallow.
[b] Analyses for rotations done by student summer help.

analyzed, or where the soils may contain $CaCO_3$ (14), as was the case in one of the 21 soils. This sample was overestimated by all three laboratories using the C–N gas analyzer. The overestimation became evident from an analysis of the C/N ratio which exceeded 14, and the fact that the soil sample effervesced (pH > 7.5) upon treatment with acid. The W–B procedure gave a very good estimate of SOC concentration for this soil sample because CO_2 gas from carbonates is not measured in this procedure. However, the procedure suffers from interferences from high chloride concentration, and its potential to pollute the environment from the strong chromic acid waste generated (14). Our modified procedure minimizes the amount of chromic acid used (2. 5 mL only), and upon completion of the analysis, the unreacted Cr^{+6} is reduced to Cr^{+3} with organic material (14). The LOI procedure measures soil organic matter (SOM), and its conversion to SOC is very empirical, with most laboratories using a constant value of 60% C in the SOM. Schulte et al. (15) gave a very through evaluation of this procedure which requires careful attention to accurate weights obtained of the initial soil sample, at 105°C (removal of free water and water associated with certain minerals), and at 400°C (complete oxidation of SOM without affecting carbonates).

The data in Fig. 3 showed that, although the SOC values for the W–B and LOI tended to be lower, there was no statistical difference among the means ($n = 21$) for the three laboratories using the C–N gas analyzer procedure, the W–B, or the LOI procedures.

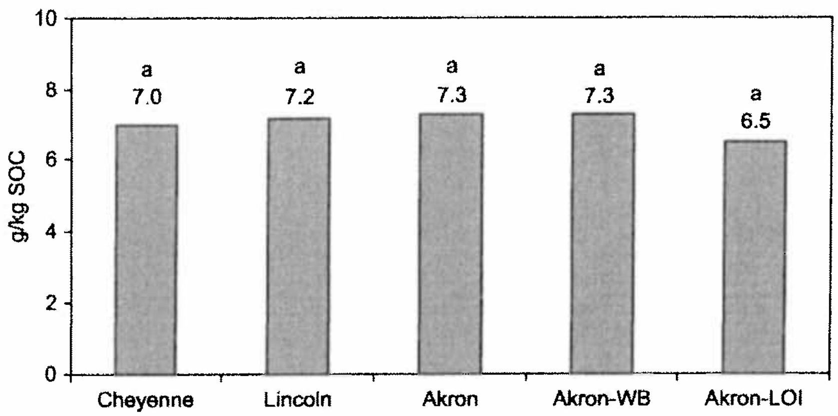


Figure 3. Comparisons among laboratories and methods of mean soil organic C concentration (n = 21 different soil samples) (Tukey, 0.1).

Soil Spatial Variability

Soil spatial variability can be large even across short distances and seemingly homogeneous areas (16). We therefore, decided to assess this variability within small research plots, and also within larger plots similar in size to a farmer's field. For the spatial variability experiments, samples were assessed on the basis of SOC concentration as well as on a volumetric basis (bulk density and soil depth correction to 15 cm).

The small plots (10 m × 30 m) are part of a major long-term study to understand soil water, nutrient, chemical and physical changes, and their effects on yield and crop residue production under different tillage and cropping systems (9–11). These plots were established in 1990 and 1991. For these relatively small plots, only one composited sample was necessary to arrive at a 10% error from the mean. Our one sample, though, was much more intensively obtained at the replicate level (12 composited cores from all phases of a rotation) than is customary for research plots. Nonetheless, the data did show that replicate (block) 1 was significantly greater with respect to SOC content from replicates 2 and 3 (Fig. 4).

Within the larger plots, variability for the W–C–Soy–F phase (two plots) and the 16 plots with all rotation phases were assessed on SOC concentration (%) and content (Mg/ha). Average concentrations for the two same-phase plots were 0.63% (high = 0.67% and low = 0.58%) and 0.67% SOC (high = 0.71% and low = 0.63%). Coefficients of variation were 4.5 and 4.6, respectively. Under these conditions at least two core samples across the plot were necessary to achieve 10% error from the mean.

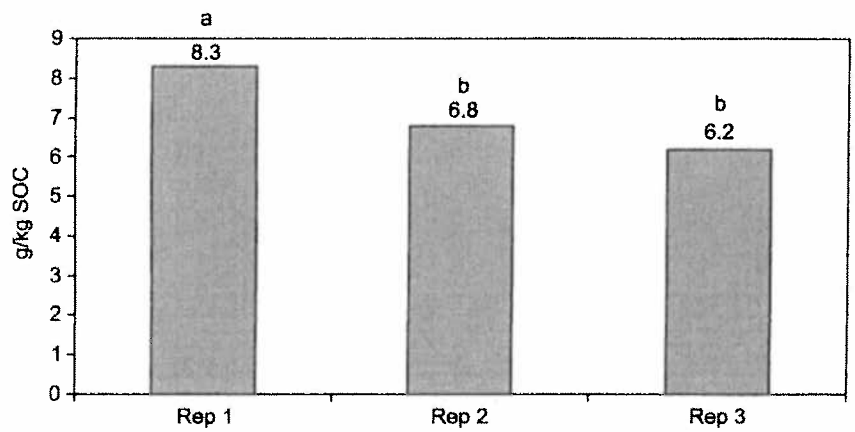


Figure 4. Comparison of mean soil organic C concentration replicate plots in an alternative crops rotation.

With the whole field (16 plots), average C concentration was 0.69% (high = 0.80%, low = 0.52%). The coefficient of variation (13.6%) was higher than that of the small plots. Thus, 10 composited soil cores were required for the whole field assessment to achieve the 10% error from the mean. The average C concentration for the whole field was only 6% higher than that for the average of the two subplot phases, but this field required more core samples because of the higher coefficient of variation. The analysis of variance showed no significant difference among rotation phases, but as with the small plots, the replicates were significantly different.

With respect to SOC content, results for the W–C–Soy–F phases were 14.9 and 14.6 Mg ha^{-1}, respectively. For the whole field, this value was 15.0 Mg ha^{-1}, a 2% greater value than the average for the subplots. One of the probable reasons for such a close correspondence was the fact that composite soil core samples were used for all analyses.

Rotation Comparisons

A previous paper based on cropping intensity (10) had shown increases in SOC content as cropping intensity increased from W–F (0.5 cropping intensity) to continuous cropping (1.0 cropping intensity). However, this previous study did not differentiate specific rotations with the same cropping intensity (for instance, W–C–F and W–Sun–F would have had the same (0.67) cropping intensity). We therefore evaluated the six different cropping rotations for differences in SOC, both among and within cropping intensities ranging from 0.5 to 1.0 (Fig. 5). The W–F (0.5 cropping intensity) and the W–Sun–F (0.67 cropping intensity) rotations produced the least amount of crop residue, and were probably subjected to greater loss of SOC to decomposition and wind erosion than the other cropping systems (12). These two rotations were, therefore, expected to result in lower SOC than other rotations with higher cropping intensities, or in which sunflowers were not included in the rotation. This study found that for mean comparisons of rotations, the general trends were increased SOC with increased cropping intensity, and within a given cropping intensity, lower SOC in the rotation including sunflowers (Fig. 5). The W–C–F rotation was 15% higher in SOC than the W–Sun-F rotation, and the W–C–M–F rotation was 11% higher in SOC than the W–C–Sun–F rotation. However, a difference of 3.6 Mg ha^{-1} C was required for statistical significance between cropping rotations, so no significant differences in SOC were observed among all rotations that included fallow. The W–C–M rotation was 11–34% higher in SOC content than the five rotations which included fallow. However, this continuous cropping treatment was significantly higher in SOC than the W–F and the W–Sun–F rotations only. As a percentage of the original sod, the W–F rotation lost approximately 60% of its SOC content, and the W–C–M, 56%.

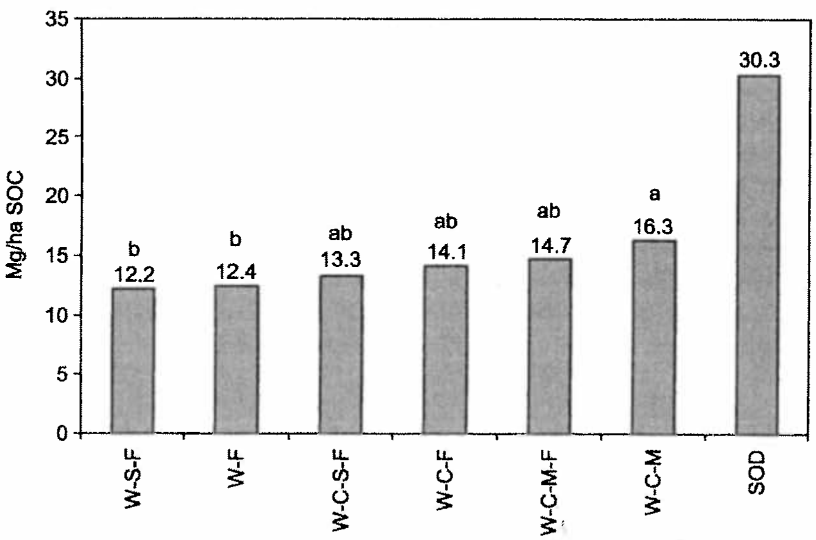


Figure 5. Comparison of mean soil organic C concentration among for six rotations (Mg SOC/ha) (Tukey, 0.1). Native sod shown for comparison.

Other studies have demonstrated that small increases in SOC can produce large agricultural benefits. Bauer and Black (1) showed that an increase of 0.6 Mg SOC in the top 30-cm layer produced about 16 kg ha^{-1} extra wheat grain yield in North Dakota. Rasmussen et al. (17) showed that 5 metric tons of mature crop residue/ha/yr maintained soil organic matter levels in a W–F system in the Pacific Northwest of the U.S. We observed a 4 Mg ha^{-1} SOC content difference between the conventional-till W–F and the no-till W–C–M rotations; this rate of increase in SOC is about twice the estimated rate reported by Lal (18) in a review of SOC increases due to conservation tillage. These data, however, also showed that because of spatial variability, a difference greater than 3.6 Mg ha^{-1} of SOC was required for statistical significance to occur.

Under conditions of low crop residue inputs, as is generally experienced by cropping conditions in the central Great Plains (11), positive increases in SOC content over a relatively short time may be difficult to quantify for a specific rotation. This may not be the case for comparisons of widely divergent rotations such as the conventional-till W–F and a no-till continuous cropping rotation. Thus, even if both rotations are still losing SOC to decomposition and erosion, the slope (rate of loss) for the continuous cropping may be less, therefore, indicating a significant reversal of SOC loss.

CONCLUSIONS

Our results indicate that different laboratories can arrive at the same numerical value for SOC content. Spatial variability and short-term rotation treatment differences in SOC content, however, will not be as easy to determine. Because of the intrinsic difficulties in assessing SOC changes, these changes probably will be modeled based on climate, soil type, topography, tillage, rotation, chemical inputs (best management practices), and biomass yields (crop residue and grain) with selected field verification or ground truthing. This ground truthing, however, will require great attention to sampling regime, and a sufficiently minimum elapsed time for changes to occur.

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