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Review

Review of Soil Organic Carbon Measurement Protocols: A U.S. and Brazil Comparison and Recommendation

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Abstract: Soil organic carbon (SOC) change influences the life-cycle assessment (LCA) calculations for globally traded bio-based products. Broad agreement on the importance of SOC measurement stands in contrast with inconsistent measurement methods. This paper focuses on published SOC research on lands managed for maize (*Zea mays* L.) in the U.S. and sugarcane (*Saccharum officinarum* L.) in Brazil. A literature review found that reported SOC measurement protocols reflect different sampling strategies, measurement techniques, and laboratory analysis methods. Variability in sampling techniques (pits versus core samples), depths, increments for analysis, and analytical procedures (wet oxidation versus dry combustion) can influence reported SOC values. To improve consistency and comparability in future SOC studies, the authors recommend that: (a) the methods applied for each step in SOC studies be documented; (b) a defined protocol for soil pits or coring be applied; (c) samples be analyzed at 10 cm intervals for the full rooting depth and at 20 cm intervals below rooting until reaching 100 cm; (d) stratified sampling schemes be applied where possible to reflect variability across study sites; (e) standard laboratory techniques be used to differentiate among labile and stable SOC fractions; and (f) more long-term, diachronic approaches be used to assess SOC change. We conclude with suggestions for future research to further improve the comparability of SOC measurements across sites and nations.

Keywords: carbon sequestration; soil sampling; bioenergy; ethanol; sugarcane; maize

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1. Introduction

Global soils contain roughly twice as much carbon (C) as the atmosphere and three times the amount of C in above ground vegetation [1–3]. Soil organic carbon (SOC) is defined as carbon in soils derived from the decay of plant and animal residues, living and dead microorganisms, and soil biota [3]. Interest in managing SOC is driven by climate concerns and the need to maintain or improve soil qualities to meet increasing demands for food, feed, fiber, and other bio-based products (e.g., [1,4–8]). A perquisite to SOC management is consistent SOC measurements [9–11]. SOC content is important due to SOC's relationship with soil properties and functions ranging from the regulation of water runoff to reductions of erosion and greenhouse gas (GHG) emissions as targeted by Sustainable Development Goals [12].

Converging interests from climate and soil scientists have fueled diverse efforts to monitor SOC as a key indicator of sustainability, especially in agricultural areas (e.g., [8,11,13–15]). One use for the quantitative SOC data generated by monitoring is to improve computer simulations of SOC changes and support more accurate and consistent estimates of life-cycle GHG emissions for biofuels and other products. This is important because existing LCA calculations reflect large uncertainties for the carbon-cycle effects of bioenergy crop production [16,17]. Estimated values for SOC change are important in determining the carbon intensity of fuels under California's Low Carbon Fuel Standard (LCFS) in the U.S. [18] and the emerging RenovaBio Program for bioethanol in Brazil [19]. Thus, improving SOC measurements can support more consistent and reliable modeling with implications for the acceptability of biofuels as renewable alternatives to fossil fuels.

This review was initiated because a lack of consensus on measurement and verification methods undermines the ability to provide clear guidance about the effects of land management practices and land-use change on SOC [4,20]. Diverse SOC measurement methods restrict researchers' ability to compare studies and generate conclusive results. For example, a meta-analysis of 537 observations in 74 studies of SOC and land-use change found that the diverse measurement methodologies limited conclusions to "working hypotheses" [20]. Heterogeneity of soils, variable environmental conditions, and different land use histories at almost every soil sampling site create challenges for researchers [4]. Organizations such as the Food and Agriculture Organization of the United Nations (FAO) [21] and the United States Department of Agriculture Natural Resource Conservation Service (NRCS) [13] have attempted to provide some guidance for site and soil profile descriptions, but have not recommended specific standards or measurement protocols. Uncertainties about SOC content are compounded when making estimates of the global soil carbon pool [22].

An SOC assessment typically involves several steps: sampling strategy, timing, and design; soil collection tools, depths, and techniques; handling, storage, labeling, and aggregation of samples; laboratory analyses; and verification techniques. Methodological differences in each step can lead to different estimated SOC values for the same location [19,23]. Different sampling strategies and calculations are applied for different purposes, for example, depending on whether a study is focusing on short-term or long-term change, or whether measurements are intended to determine concentrations (mg/kg) or to estimate stocks (g/m^3). The United Nations' Global Soil Partnership reports that users of soil data prioritize data generation methodology as a key challenge influencing reported values [14], whereas field researchers identify efficient sampling design and estimation schemes as the primary challenge [15].

The purpose of this review is to compare SOC sampling protocols in the U.S. and Brazil and to initiate a discussion about how methodological inconsistencies might be resolved. We review a set of published research focused on SOC measurements in fields managed for maize (*Zea mays* L.), commonly referred to as corn in the U.S., and sugarcane (*Saccharum officinarum* L.) in Brazil. Maize and sugarcane are the two primary feedstock crops used for global fuel ethanol production. We chose this focus because policies promoting the use of fuel ethanol are justified by the assumption that net CO_2 emissions are reduced when ethanol replaces fossil fuels. Yet, the calculation of emission reductions significantly depends on any net changes in SOC that occur due to changes in land cover or management under biofuel production scenarios [20,24]. Brazil and the U.S. produce approximately 85% of total global fuel ethanol, which is the most important biofuel in use today [25]. In both countries, estimated life-cycle emissions are critical for market access, as well as public and political support [26].

Our hypothesis is that developing agreement among U.S. and Brazilian researchers on a set of recommended SOC measurement protocols [27–29] is a good first step toward developing broader consensus on SOC measurement protocols. Furthermore, recent research on SOC changes for biofuel crops and the ongoing debates among scientists concerning biofuel sustainability, offer recent data and high relevance to the focus of our review. Consistent SOC measurements are also relevant to other productive land uses and to national GHG inventories and accounting under the Paris Accords. The following sections discuss the selection of literature reviewed, key characteristics that differentiate SOC measurement approaches, and proposals for improving future collection and use of SOC data. We hope that the results of this review lead to more accurate and representative LCAs and ongoing recommendations for management practices that lead to increased SOC in managed lands.

2. Materials and Methods

2.1. Identification of Published Literature to Review

Literature searches were performed using electronic, online research platforms (ISI Web of Science [30], Google scholar [31], and ResearchGate [32]). Search criteria (Figure 1) were applied to identify SOC studies published between 1995 and 2015 (when the literature search was conducted). This time frame is intended to focus on current methods for SOC measurement in studies associated with feedstocks for ethanol production, one of the criteria for identifying the literature to review.

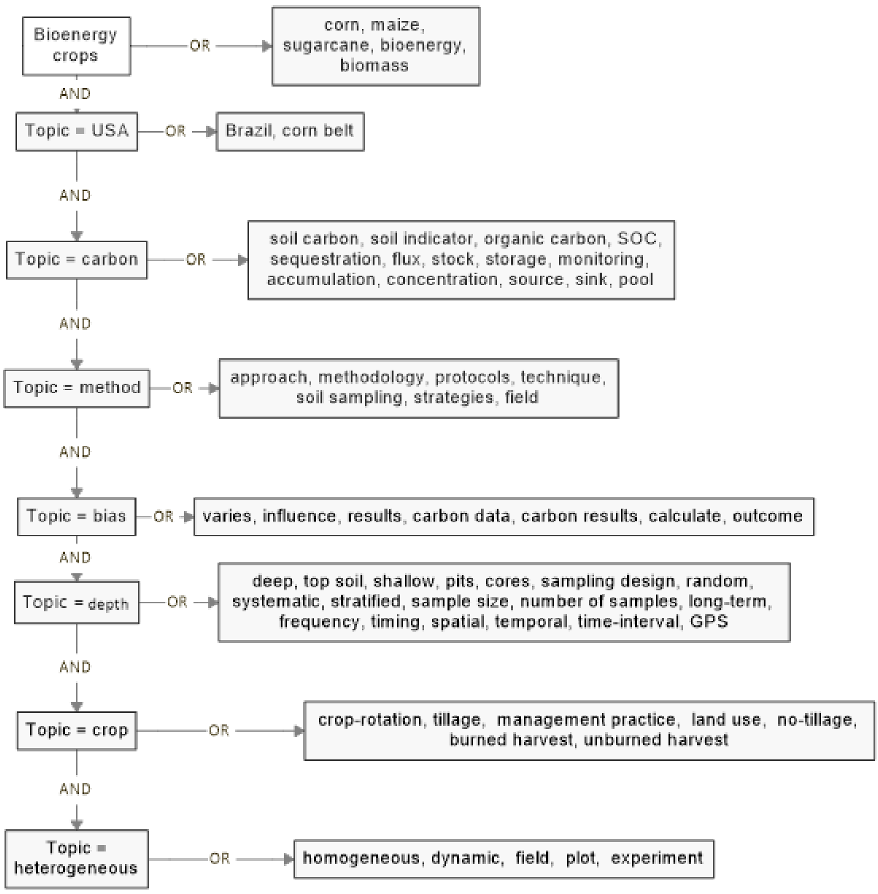
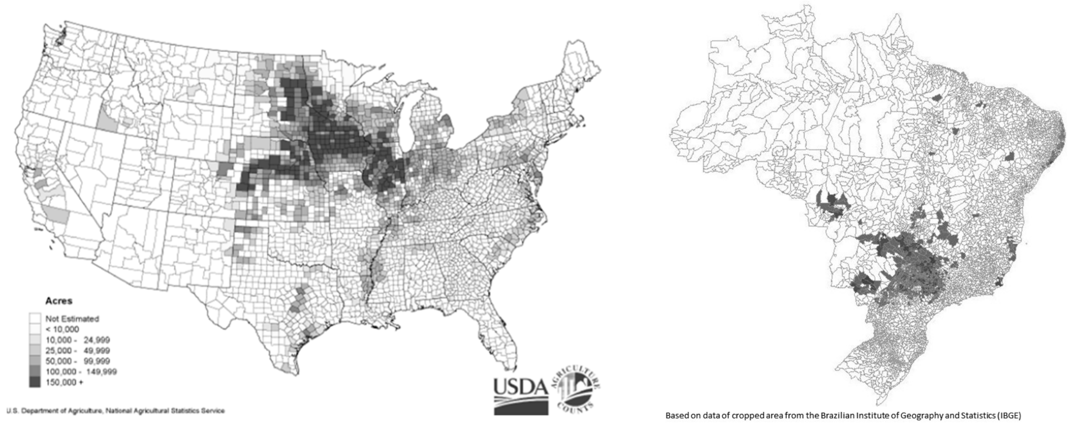


Figure 1. Search criteria applied to identify SOC literature published between 1995 and 2015 relevant to the goals of this review.

The papers identified using the search criteria were categorized according to factors including: country, crop(s), sample design (random or systematic), sample (point, plot or landscape), sampling protocol (if cited), soil collection method (pit or core), sampling depth (cm), and laboratory method for carbon quantification (dry combustion [33], Walkley-Black titration [34], or other). To the degree data were provided, the results of each paper were recorded in terms of the reported SOC values (e.g., SOC, total organic C), the location and its characteristics (region, biome, land use, soil type and if possible geographic coordinates), and the time of analysis or period considered for SOC change analyses.

2.2. Description and Background of Study Areas

Forty-one studies were selected for review, 26 from the U.S. (Table 1) and 15 from Brazil (Table 2). Most U.S. studies were from the "corn belt" in the Midwestern states (Figure 2) and considered maize grown in rotation with other annual crops such as soybean and wheat. The Midwestern region is characterized by relatively flat topography, fertile soils, and high organic soil concentrations. The temperate climate is humid and hot in summer, but temperatures typically drop below zero for extended periods in the winter. Most Brazilian studies involved sugarcane, a semi-perennial crop typically grown for five to 10 years before fields are tilled and renewed. Most of the Brazilian studies were conducted in the Centre-South region (Figure 2) characterized by weathered soils with limited SOC concentrations, and hilly topography. The tropical climate typically generates relatively warm dry winters and wet summers. Meta-analyses of land-use change suggest that clearing pasture or forest lands to cultivate annual crops or sugarcane will tend to cause SOC concentrations in soils to decline. However, managing annual crops or sugarcane on lands that are degraded or are subject to frequent burning, can increase SOC concentrations [35,36]. Additionally, a study in Brazil found that sugarcane succeeding native vegetation or pastures will lead to a decrease in soil organic matter (and soil carbon), while the opposite was observed if sugar cane followed annual crops [37].



(**A**) (**B**)

Figure 2. Concentration of (**A**) USDA corn production in USA, primarily covering the states of Iowa, Illinois, Nebraska, and Minnesota [38]; and (**B**) for sugarcane production in Brazil, primarily covering the states São Paulo, Minas Gerais, Mato Grosso do Sul and Goiás [39].

2.3. Predominant Sampling Methods in the U.S. and Brazil

Most U.S. studies considered in the review (Table 1) followed the Greenhouse gas Reduction through Agricultural Carbon Enhancement network (GRACENet) protocols for measuring SOC. Several U.S. studies were also designed to be consistent with Renewable Energy Assessment Project (REAP) protocols [40,41]. GRACEnet is a research program initiated by the U.S. Department of Agriculture's (USDA) Agricultural Research Station that recommends uniform soil sampling protocols (i.e., sampling to a depth of 100 cm and compositing multiple soil cores) for all sampling sites. In contrast, most Brazilian studies (Table 2) followed the Intergovernmental Panel on Climate Change (IPCC) guidelines for National Greenhouse Gas Inventories [42], as well as the International Organization for Standardization (ISO) 10381-1 [43], ISO 10381-2 [44], and ISO 10381-4 [45] standards for SOC measurements. These four guidelines are consistent with most recommendations by Cerri et al. [11].

2.4. Predominant Laboratory Methods in the U.S. and Brazil

Two basic methods were commonly used for laboratory analysis of carbon content in soils: the Walkley-Black titration method (WB) [34] and dry combustion (EA) methods [33,46]. Equipment for the WB methodology is generally available in most soil-testing laboratories since a single, relatively low cost, instrument is required [47]. The EA method is based on thermal oxidation of organic C and decomposition of inorganic C to CO_2 within a furnace where soil samples are burnt at temperatures exceeding 1000 °C to 1600 °C depending on the equipment set up [33] to ensure that all C species are quantitatively converted to CO_2 [47].

The primary limitation associated with measuring total C in soil samples using WB is that C recovery is not always complete [48,49]. The C quantified by this method is that oxidized by dichromate, which is very inefficient to recalcitrant C forms such as charcoal, graphite, and soot [33]. Moreover, there are evidences that trapped C in soil aggregates are protected to some extent from dichromate oxidation [50]. Interferences caused by Cl, ferrous Fe, and MnO_2, combined with the existence of refractory organic C in the soil samples, contribute to the weaknesses in the WB method. Several modifications of the WB method using salts and external heat to achieve a boiling reflux [51] have been used to reduce interferences and enhance organic C oxidation to 100%, but these modifications are not always efficient [52]. Furthermore, the interfering agents seem to have different weights depending on soil type, clay content, sampling depth, and land use [48]. These limitations make it challenging to propose a global correction factor for the WB method. One of the few limitations associated with the EA method when determining organic carbon (OC) is the presence of inorganic C (IC), a common problem in calcareous or recently limed soils. In this case, OC can be estimated by the difference between total carbon (TC) and IC or directly after removing IC [47].

3. Results

Selected attributes of the U.S. and Brazilian studies reviewed are listed in Tables 1 and 2. The two predominant sampling designs were either systematic [53] or random [54], with most Brazilian studies choosing a systematic approach using a grid pattern for samples, and most of the U.S. studies choosing a random design: subdividing a land unit to be studied and randomly selecting coordinates for each sample. Nearly all Brazilian studies used excavated pits, although several also used a coring device for a portion of the sampling. This was true across Brazilian studies including the few that focused on maize [55–57]. All reviewed studies from within the conterminous U.S. used a coring device as described within the GRACENet protocol (See Table 1, [58]). Samples collected in both countries represented a wide variation in soil depth ranging from 10 cm (e.g., [59]) to 100 cm (e.g., [48]). Most U.S. and Brazilian studies used depth increments of 5 or 10 cm for the upper 30 cm of the soil profile [60,61], but below 30 cm the depth increments varied substantially. In general, however, Brazilian researchers collected several samples in 10- to 20-cm increments [11,62], while in the U.S., most samples were taken in 20- to 30-cm depth increments [63,64]. Comparisons of management practice effects on soil C were generally made using a synchronic approach with paired plots or a chronosequence (i.e., diachronic approach), with the most common design being paired plots [65,66]. We also found that the majority of the studies reviewed were sampled only once during the course of the experiment [67,68].

It is difficult to quantify differences in data associated with the SOC estimates due to the complex interactions among the variables involved. Even if it was possible to identify some sources of difference in the reported data, establishing relative weights among potential sources of uncertainty or error is difficult [69] unless duplicate sampling could be performed simultaneously using different methodologies—an approach not identified among the literature reviewed. Re-performing SOC measurements at later times can be confounded by changing weather, tillage, crop management, and other conditions that affect SOC values over time. Thus, for this review, estimating the degree of uncertainty associated with particular differences in SOC measurement strategies was not possible.

Table 1. Source and characteristics of the U.S. SOC assessment studies that were reviewed.

Authors	Crop †	Sample Design	Sample Collection Method	Sampling Depth (cm)
Yang and Wander (1998) [70]	M/S	Random	Cores	30
Bolinder et al. (1999) [65]	M	Random	Cores	30
Duiker and Lal (1999) [71]	M/W	Systematic	Cores	30
Yang and Wander (1999) [72]	M/S	Random	Cores	90
Clapp et al. (2000) [73]	M	Random	Cores	30
Motta et al. (2000) [74]	M/S/So	Random	Cores	30
Rhoton et al. (2002) [75]	M	Random	Cores	15.2
Wilts et al. (2004) [76]	M/W	Random	Cores	45
Halvorson et al. (2003) [77]	M/W	Random	Cores	15.2
Hooker et al. (2005) [78]	M/W	Systematic	Cores	15
Olson et al. (2005) [67,79]	M/S	Systematic	Cores	75
Gál et al. (2007) [63]	M/S	Systematic	Cores	100
Ramirez et al. (2007) [80]	M/S	Random	Cores	100
Sainju et al. (2008) [81]	M	Systematic	Cores	20
Varvel and Wilhelm (2010) [82]	M/S	Random	Cores	30
Follett et al. (2012) [83]	M/SG	Systematic	Cores	150
Aziz et al. (2013) [84]	M/S/W	Random	Cores	30
Evers et al. (2013) [60]	M/S/ SG	Random	Cores	15
Follett et al. (2013) [64]	M	Not reported	Cores	120
Franzluebbers and Stuedemann (2013) [66]	M/S/W	Random	Cores	150
Karlen et al. (2013) [61]	M/S	Systematic	Cores	15
Ashworth et al. (2014) [85]	M/S	Random	Cores	15
Obade and Lal (2014) [86]	M/S	Random	Cores	60
Fan et al. (2014) [87]	M	Systematic	Cores	60
Kumar et al. (2014) [88]	M	Not reported	Not reported	60
Xiaong et al. (2014) [89]	Sc, other	Random	Cores	20

† Crop abbreviations: M—maize (*Zea mays* L.); S—soybean (*Glycine max* (L.) Merr.); SG—switchgrass (*Panicum virgatum* L.); So—sorghum (*Sorghum bicolor* L.); Sc—sugarcane (*Saccharum officinarum* L.); W—wheat (*Triticum aestivum* L.); When "other" is indicated, the study included areas not dedicated to agricultural production (e.g., native forests) and other non-bioenergy crops (e.g., coffee (*Caffea Arabica* L.)). "Not reported" indicates the information was not supplied in the article being reviewed.

Table 2. Source and characteristics of the Brazilian SOC assessment studies that were reviewed.

Author	Crop †	Sample Design	Sample Collection Method	Sampling Depth (cm)
Cerri et al. (2004) [68]	Sc	Systematic	Pits ‡	20
Souza et al. (2005) [90]	Sc	Random	Cores	30
Diekow et al. (2005) [55]	M	Systematic	Pits	107.5
Razafimbelo et al. (2006) [59]	Sc	Systematic	Not reported	10
Resende et al. (2006) [62]	Sc	Systematic	Pits/auger	60
Szakács (2007) [91]	Sc	Systematic	Pits	30
Calegari et al. (2008) [56]	M/S	Systematic	Pits	60
Galdos et al. (2009) [92]	Sc	Systematic	Pits	100
Pinheiro et al. (2010) [93]	Sc	Systematic	Pits/auger	100
Tivet et al. (2012) [29]	Sc	Random	Pits	100
Rossi et al. (2013) [94]	Sc	Systematic	Pits/auger	60
Zotarelli et al. (2012) [57]	M/S	Systematic	Pits/auger	80
Borges et al. (2014) [95]	Sc	Random	Not reported	20
Kuwano et al. (2014) [96]	Sc/Ca/other	Random	Auger	10
Seben Junior et al. (2014) [97]	M/S	Systematic	Trowel, cores	Not reported
De Vasconcelos et al. (2014) [98]	Sc	Random	Not reported	60

† Crop abbreviations: M—maize (*Zea mays* L.); S—soybean (*Glycine max* (L.) Merr.); SG—switchgrass (*Panicum virgatum* L.); So—sorghum (*Sorghum bicolor* L.); Sc—sugarcane (*Saccharum officinarum* L.); W—wheat (*Triticum aestivum* L.); When "other" is indicated, the study included areas not dedicated to agricultural production (e.g., native forests) and other non-bioenergy crops (e.g., coffee (*Caffea Arabica* L.)). "Not reported" indicates the information was not supplied in the article being reviewed. ‡ Pits were opened for quantification of soil bulk density and other assessments. Samples for C analysis represented composite samples taken with an auger; "Trowel" indicates sampling such as soil blocks or "grab samples" which are a form of sampling using a trowel or shovel to open a small or shallow pit.

4. Discussion

4.1. U.S. and Brazilian SOC Assessment Inconsistencies

Our review of the literature in the U.S. and Brazil did not identify a consistent use of a single SOC measurement approach. Issues of sampling design, methods, and research intent are applicable within both the U.S. and Brazil. Several distinctions can be made when comparing the predominant SOC methods used in each country.

4.1.1. Design and Number of Samples

Developing appropriate sampling designs is a basic, but crucial step for obtaining unbiased mean SOC estimates for a given area [54]. Two types of sampling designs were used to estimate C stocks [75] in the reviewed studies—systematic and random sampling, and in general, methodologies for implementing both designs are well documented [42]. However, these sampling designs may not accurately capture inherent variability in SOC distribution. In both the Midwestern U.S. and Brazil (e.g., São Paulo State), high variability in soil types and within-field conditions are common [99,100] and may warrant the use of stratified techniques where a field or plot is divided into sub-populations or strata for random sampling within each strata [101,102]. When selecting sampling designs, researchers must consider how data will be used and how many samples will be needed to detect a statistically significant and reliable change in SOC. For example, the sample design used for a regional study from which to calculate SOC change for simulation models will be distinct from the design used to consider the effects of different management practices involving a single farm operator. Also, since SOC distribution in soils is inherently heterogeneous, scaling from point samples to the landscape scale has been shown to increase the coefficient of variation by as much as 30% [69]. Although the number of samples collected will depend on many factors including available finances and time, the number required to provide statistically valid results should be carefully considered. If sufficient data are generated and verified using standardized measurement protocols, this could be valuable for producers in both nations wishing to document the effects of their operations.

4.1.2. Detection of SOC Change and C Sequestration

It is widely known that SOC changes occur very slowly and often exhibit high spatial, but very low temporal, variability. Therefore, relative to its actual value, detecting any measurable changes can be very difficult. Smith [103] demonstrated, for a range of soil types and land uses by using a model of SOC dynamics, that six to 10 years would be required to detect a 15% SOC increase (regular soil sampling of 10 to 20 samples). Smaller variations would require a very large number of soil samples. To estimate C sequestration, an initial set of SOC measurements must be taken to establish a baseline condition that is assumed to be at a steady state and then any subsequent positive changes in SOC stocks due to management changes can be considered atmospheric C sequestration or alternatively loss due to SOC depletion [72]. The accumulation of C in the soil is dependent on a balance between fresh C inputs and SOC respiration. With a land use change, SOC may increase when C inputs are greater than before but the concomitant increase in substrate for decomposition results in an equilibrium after some time. Current IPCC guidelines state that SOC stocks stabilize or reach a new steady state equilibrium after 20 years [42] following a management change. However, for many soil types, especially those with a high clay content and high SOC levels, a 20-year timeframe is likely to be too short to establish a new SOC equilibrium following either a land use change or land cover change. Furthermore, some conventionally managed soils may, under some conditions, continue to lose C for 50 to 100 years [104]. Spatial variability and the slow rate at which C changes both present major challenges for monitoring and predicting SOC changes [105]. Long-term field experiments (i.e., a diachronic approach) lasting several years or even decades are generally considered to be the best for detecting soil management effects on soil C storage [67], but unfortunately paired plots (i.e., synchronic or chronosequence studies) were found to be the most common within the literature reviewed for both countries. One study by

Costa Junior [106] did examine both strategies and showed that the diachronic approach could more accurately detect soil C accumulation changes since the synchronic approach tended to over-estimate C accumulation.

4.1.3. Sample Collection Method—Pits versus Cores

After selecting a sampling design that is appropriate for the question being asked (i.e., short-term SOC change or long-term C sequestration), another important question is how to collect samples. There are several options for collecting soil samples (e.g., digging open pits, coring with a punch core, using a machine-driven core drill, or for some unique situations relying on explosives). However, for SOC studies, the two most common sampling approaches are: (1) large-excavation of quantitative soil pits (henceforth, "pit"); and (2) coring (i.e., extracting a cylindrical section of soil), as outlined in the GRACEnet protocol [58] or similar documents. The excavation of soil pits has been identified as a widely applicable and universally accepted method [107], and therefore most of the Brazilian studies examined in this review utilized the "pit" method for sample collection. Conversely, all U.S. studies used the coring method. When compared to collecting soil samples from pits, coring is much less time consuming, therefore making it possible to collect a greater number of samples and achieve greater precision, especially in spatially heterogeneous sites. Many researchers [107] agree that the increased number of samples is an acceptable tradeoff for the soil compression that occurs with coring. However, the nearly exclusive use of coring in the U.S. has resulted in a lack of knowledge regarding the distribution or extent of the compaction within different soil core increments and a low level of precision and understanding of the partitioning in compaction that can occur with coring [108]. Methods to overcome sampling-induced compaction have been developed, but other potential sources of inaccuracy persist. For example, underestimating of the coarse soil fractions (>2 mm) can occur if the coring equipment is unable to collect larger rocks [109]. Fortunately, for most agricultural soils with low rock content, this effect is probably negligible.

In comparison to collecting cores, using excavated pits can more readily reveal soil structure and horizon development [110]. Soil pits can also eliminate the accuracy constraint of cores by allowing a more specific measurement of soil mass for bulk density assessment [109]. The major problem associated with pit sampling is site disturbance, and thus the diminished possibility for re-sampling at the same location (i.e., diachronic research). This can affect the precision of SOC estimates when calculating changes in soil C stock [110]. For soil C and nitrogen analyses, some researchers have chosen to supplement pit sampling by collecting additional cores using an auger. However, it can be difficult to identify the exact depth from which the core samples were taken and to prevent cross contamination of soil strata when sampling with augers. This challenge can be overcome by using a core barrel sampler attachment [111], as confirmed by recent research [29] showing that undisturbed cores can be collected from agricultural soils with augers. However, augers should be avoided in ecosystems with variable quantities of stones and coarse roots (e.g., forests) and emerging technologies such as the rotary core device [107] should be used. This recommendation originated from research by Rau [107], who compared the sampling of pits with a rotary core device, and found no consistent bias for either method. However, that is only one comparison study, thus our recommendation is for additional studies comparing traditional coring methods with a combination of soil pits and auger sampling.

4.1.4. Sampling Depth and Increments

Choosing the correct sampling depth for the question being asked is very important because SOC is unevenly distributed throughout the soil profile [29] and several studies have shown that long-term C accumulation can occur in deeper soil layers (e.g., 30 to 100 cm) [112]. Measuring SOC only within the upper portion of the soil profile could yield inaccurate results [67], and either over- or under-estimate temporal changes such as erosion that occurred [113]. As a result, most SOC sampling protocols (e.g., GRACEnet) now advise that samples be taken to a depth of at least 100 cm [11,62].

Other protocols [114] and recommendations [23] suggest that SOC measurements be taken throughout the entire root zone, which for some plant species, can extend to 200 cm. However, current spatial inventories of SOC often focus only on the mineral topsoil, since that is what the Kyoto Protocol specifies [115]. As a result, existing soil C maps that frequently provide the basis for sustainability studies (e.g., LCA of ethanol feedstock production) are often based on data that poorly reflects C pools within deeper soil horizons [29]. Furthermore, it has been found that differences in root distribution between plant species and/or effects of plowing versus no-till operations might be overshadowed if only a portion of the soil profile is examined [112,116].

In addition to choosing the total sampling depth for the question being asked, decisions will be needed regarding appropriate depth increments to calculate SOC changes, redistribution, loss, or sequestration. The most common choices are to sample by horizon, an approach that is often easier with pits than core samples, using a fixed sampling depth (e.g., 0 to 5-, 5 to 15-, 15 to 30-, 30 to 60-, and 60 to 100-cm), or if the comparison involves a tillage change (conventional versus no-tillage), using a fixed sample mass to compensate for compaction-induced volumetric changes. Within the limitations of this review, we were unable to assess the implications of collecting samples from different depth increments on final estimates of SOC stocks in either the U.S. or Brazil. This would have required sample re-analysis which was outside of the scope of our assessment. We do agree that implementing recommendations for deeper sampling in all SOC studies would result in better and more meaningful comparisons, but we are pragmatic enough to realize that ultimately each researcher will be faced with determining exactly how many samples and depth increments can be collected, processed, analyzed, and interpreted based on available fiscal and labor resources. We also acknowledge that in many locations, deeper soils are protected from change. Additionally, soil truncation can vary by site (e.g., even 100 cm of soil erosion can occur in areas with a steep slope) and must be accounted for in the sampling protocol as well. In general, however, the soil science community is lacking information on SOC with depth.

4.1.5. Laboratory Analysis—Walkley-Black Titration Method (WB) versus Dry Combustion (EA) Methods

After addressing all the factors influencing the collection of soil samples for SOC quantification, the next decision point for which there are varying perspectives is the method of laboratory analysis. To illustrate the implications of different analytical procedures, data from four studies carried out in agricultural soils of Brazil comparing WB and EA methods for total C quantification were compiled in Figure 3. Soil analyses from the upper 5 cm of a clayey Oxisol of Central Brazil (Cerrado biome) cropped to different maize rotations were provided by Coser [49], while those from Segnini [117] also corresponded to Oxisols but from the Northern São Paulo state. In a third study [118], soil total C data came from different soils planted to eucalyptus from eastern Minas Gerais, including Oxisols. Finally, a dataset from Fernandes [119] comprehended seventeen top soil samples, including eleven Oxisols, four Inceptisols, one Spodosol, and one Histosol from different locations in Brazil.

In most of the cases, the EA method detected higher concentrations of C than the WB method. The best agreement between the two methods was associated with a low C content. Two factors are hypothesized as reasons for a decreased efficiency of the WB method [33] compared to the EA analysis. First, there is a known strong interaction between organic C and the iron-enriched minerals in Oxisols and the common presence of charcoal in Brazilian soils [120] that could have contributed to differences between the two methods of analysis. Both factors could also explain the greater underestimation and variable results within surface samples from the Oxisols (Figure 3). Hence, underestimation of C content in soil samples from areas subjected to fire (i.e., those producing sugarcane or soybean and maize in Brazil), is expected if WB methods are used. Trends for overestimating soil C content with the WB method were observed by Segnini [117], which could be a result of the high iron content of Oxisols. Using the WB method, they found that C content was underestimated by 18% when compared to the EA method when all samples were compared. A detailed study by Tivet [48] for soils of tropical and subtropical regions in Brazil demonstrated the influence of land cover on the efficiency of the WB method. Effects of clay content and soil depth were also evaluated. Although there was a general agreement between WB and EA methods, Tivet [48] concluded that for a more accurate determination of soil C content, a site-specific calibration factor should be used with the WB method. This is ratified by the absence of a common trend among datasets presented in Figure 3. Based on available literature reviewed in this section, dry combustion analysis using EA is the most accurate method for measuring the soil C content. However, in many cases, the WB method was the only available option for monitoring temporal changes or it was used in the past to generate reference data sets that could now be compared to actual data. Once again, the development of site-specific correction factors would be the best strategy to improve data quality.

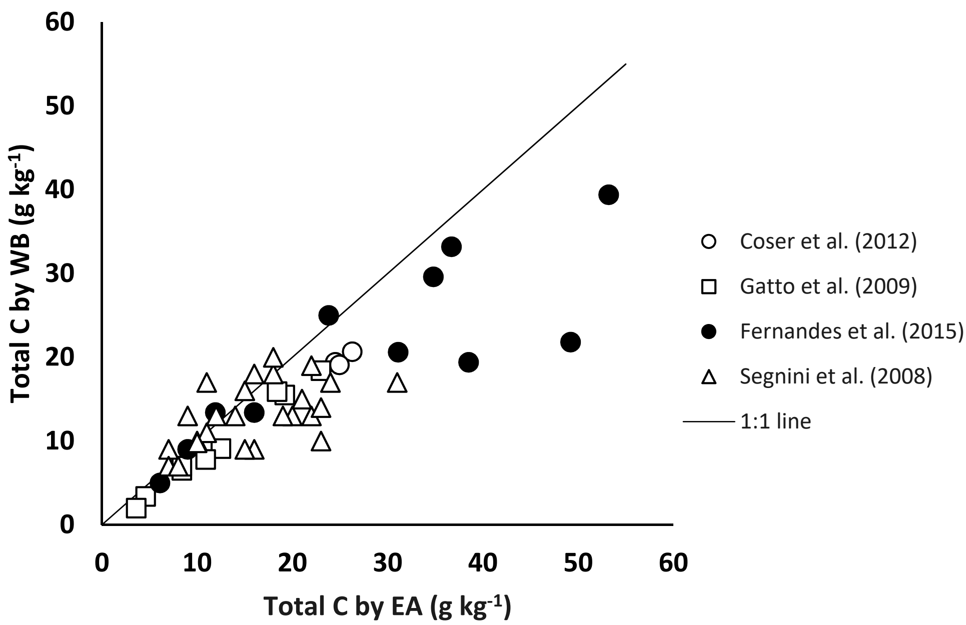


Figure 3. Total soil C content (%) determined by using WB and EA methods. Data came from four published studies whose soil samples were obtained from different soils, depths, and locations in Brazil, most of them from Oxisols.

4.2. Soil Monitoring Networks, Model Simulations, and Implications

Field sampling and laboratory analyses associated with individual studies such as those discussed above serve as the primary source for most regional and global estimates of SOC stocks [121]. Extrapolating the mean but site-specific soil C measurements for broad categories of soil or vegetation types primarily through simulation modeling introduces other uncertainties in addition to the methodological variations discussed above. Some of the additional factors include: variations in SOC stocks due to land use history [122], inadequate accounting of vertical soil C distribution [123], and lack of consensus regarding the appropriate area for extrapolation [124]. Even in well-studied regions, information on the SOC pool and its temporal and spatial dynamics is erratic, and diverse soil sampling approaches exacerbate the issue and cause high uncertainty, even for relatively simple regional comparisons [16,29].

Issues highlighted by global Sustainable Development goals, such as land and soil degradation, climate change, and increasing demand for food, feed, fiber, and fuel resources, have prompted many organizations to initiate efforts to address the high uncertainty associated with current soil C surveys. These initiatives emphasize the need for investment in global, regional, and local high resolution data on soil carbon and nutrients [125], and to improve models and applications using more reliable soil data [14]. Accurate predictions of SOC in response to agricultural or conservation practices are required to improve understanding of interactions among land management, productivity, and climate change (e.g., the USDA-Natural Resources Conservation Service Rapid Assessment of US Soil Carbon) [126]. SOC calculations from global networks and monitoring initiatives such as these are increasingly being utilized to determine the overall GHG impacts of producing bioenergy [127]. Having accurate SOC data is extremely important because it is being used to estimate CO_2 flux from agricultural practices and has become a component of LCAs being used to evaluate bioenergy production systems [8,29,99,128].

U.S. regulations such as the Renewable Fuel Standard and California's LCFS require LCAs of emissions associated with transport fuel options. For biofuels such as ethanol in the U.S., regulatory bodies have relied upon the Greenhouse gases, Regulated Emissions, and Energy use in the Transportation model, GREET [129], to estimate emissions from bioenergy feedstock production and use [130]. These calculations require a soil C factor expressed as C emissions per unit land area per year, to estimate SOC loss because of changing land cover and/or management [131]. For the federal Renewable Fuel Standard, direct soil C emission factors are generally based on the results of Colorado State's CENTURY SOM model [132]. However, CENTURY only considers the 0 to 20 cm layer, even though the IPCC recommends that assessments should focus on at least a 30-cm depth and if possible to 100 cm. These examples of how policies rely on SOC data illustrate the importance of striving for a consistent sampling and assessment protocol. Relying on only the upper soil profile (i.e., 0 to 20 cm) simulated SOC changes may be misleading.

4.3. A U.S. and Brazilian Proposal for Enhanced SOC Assessments

4.3.1. Sampling

The publications analyzed in this review helped identify several factors limiting the quality and reliability of SOC measurement data in both the U.S. and Brazil. These include: (i) limited descriptions of the methods used; (ii) two distinctly different strategies for collecting samples—pit versus core sampling; (iii) significant variation in sampling depth; and (iv) differences in analytical approaches (WB versus EA). We also describe the importance of having accurate, reliable SOC data because of the impact it can have on bioenergy industries through policy-mandated regulations and LCA. Collectively, the differences identified among studies, coupled with the lack of data and supporting documentation, reinforce the need for standardized protocols and a common reporting system for publishing SOC data.

To begin building consensus for more consistent and comparable soil C data, our first recommendation is to invest in field research that makes direct comparisons of the factors influencing SOC measurements in Brazil and the U.S. We recommend using a stratified sampling scheme [58] to capture the inherent soil variability in bioenergy producing regions within both countries. We also recommend a diachronic (long-term) approach [106], but recognize that a synchronic (chronosequence) approach may be more feasible for examining changing land use or management practices. However, if a synchronic experimental design is used, it is essential that a baseline or pre-treatment condition is documented [133]. Whenever possible, pre-treatment baselines should be used, but if they are not available, time-sequence samples should be taken at least once each year [134]. The baseline samples should also be used to assess spatial variability and the appropriate number of replicates [135] using statistical tools. It is also important to account for the spatial arrangement of plants within the sampling area (i.e., whether they are stratified or subdivided). Whether using pits, soil core devices, or augers, the area allocated to each plant should be considered by taking into account inter-row spacing and the in-row distance between plants. Effects of plot size relative to landscape position should also be considered when designing an efficient and reliable sampling strategy [134].

Currently, excavation and sampling from soil pits is recommended [135] as blocks or monoliths can be withdrawn to represent an appropriate soil volume attributed to each plant. We recognize this may not be feasible for some studies, so we offer the following recommendations for using soil cores or augers. First, the choice will likely be influenced by available resources and site characteristics (i.e., avoid augers in ecosystems with variable quantities of stones and coarse roots) [29]. Whenever possible, use a rotary coring device.

Regarding depth increments, we recommend collecting a sample for each 10 cm [58] throughout the effective rooting depth, and for each 20-cm increment below the rooting depth until the full 100 cm profile is sampled [11,43]. This is consistent with GRACEnet protocols [58] and REAP methods [40]. We also recognize that deeper (e.g., 200 cm) or shallower (30 cm) sampling may be more appropriate for some studies and locations [136].

4.3.2. Research Recommendations

Based on this review, our recommended priorities for future soil C research include: (i) comparisons of analytical methods for SOC analysis; (ii) concurrent assessments of other soil quality indicators (biological, chemical, and physical); and (iii) detailed evaluations of SOC fractionation methods [137] and quantification of the stability of various SOC components based on the fractionation method [29]. Quantifying the stability of various SOC fractions is important because of the difference among very active (labile) to stable (non-labile) fractions [137] and their relative contribution to nutrient cycling and biological availability of TC [137,138]. Furthermore, each SOC fraction has a unique impact on soil quality, thus requiring studies to better understand the interaction between SOM and aggregate formation.

Given the current initiatives to measure SOC, and the valuable experiences outside of the U.S. and Brazil, an updated comprehensive review of soil C literature would be useful to examine emerging data sets and new methodologies used around the world. For example, in China, many researchers routinely measure SOC to a depth of at least 40 cm [139]. Cross comparisons and rigorous science-based evaluations of site selection, sampling strategy, processing techniques, analytical measurements, and interpretation should be examined to facilitate a standardized SOC methodology with global relevance. Finally, we recommend that improved and updated approaches be incorporated into international standards (e.g., ISO 13065 [140]) or voluntary certification systems so that the nexus between agriculture and bioenergy production is addressed in a more consistent manner and to facilitate comparable assessment protocols and interpretations of C measurement data. Assessments with implications for bioenergy industries, food security, and global climate forcing, should rely on the best available methods.

5. Conclusions

Soil carbon performs multiple functions that are recognized as being important for the production of food, feed, fiber, and bioenergy, as well as for calculating GHG balances and assessing other ecosystem services. Accuracy and objectivity in SOC measurements are important for both guiding policy and informing public opinion. Although several scientific studies have shown that high yielding agricultural crops (e.g., switchgrass) [141] could, with appropriate management practices, increase C sinks by sequestering soil C and mitigating GHG emissions [4,142], public opinion is still mixed on the relative GHG benefits of bioenergy. Some recent articles asserting SOC losses occur with biomass use for energy add to public uncertainty [143–145]. Additionally, controversies about effects are likely to persist until consensus is reached on standard protocols for measuring SOC and SOC changes over time. Coordinated efforts are needed to improve the assessment and monitoring of the global soil C pool. Reliable and verifiable SOC data are needed to support accurate LCA calculations. Analysts and modelers need access to reliable SOC analyses rather than a mixture of studies reflecting diverse methods. As diverse C trading programs and certification schemes emerge, both will rely more heavily on SOC data [114], underscoring the need for international standards to address inconsistencies such as those highlighted in this review.

We hope that our recommendations support improved protocols and generate the information needed to conduct fair and consistent comparisons of the SOC implications of different energy options. At a minimum, we would like to promote a dialogue about the value of standardized

SOC measurements. The recommendations are a starting point. We recognize that continual improvement based on the new findings is a core concept for more sustainable land management practices. We therefore encourage continued investment in monitoring and analysis to help managers make informed decisions about the options that are most likely to lead to higher productivity and higher SOC concentrations while relying on fewer inputs.

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