



ORIGINAL ARTICLE

Methods for the determination of carbon in Ultisol in eastern Amazon

Métodos de determinação de carbono em Argissolo na Amazônia Oriental

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PALAVRAS-CHAVE

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Perda de massa por ignição
Regressão

ABSTRACT: Soil carbon (C) content is a sensitive indicator of agricultural management impact. Different methods are usually adopted to determine soil C content, which can lead to variable results and therefore hinder the correct interpretation of management impacts. In this study, we tested three commonly used soil C determination methods (dry combustion with elemental analysis, dry combustion with loss-on-ignition, and wet combustion) with soil samples from agricultural sites under different management conditions (fire and fire-free land preparation) and forest. The wet combustion method underestimated soil C content in relation to the standard method (dry combustion with elemental analysis). We found high determination coefficients of predictive equations of total C (dry combustion with elemental analysis) based on concentration values determined either through wet combustion or loss-on-ignition. Thus, we recommend the use of the loss-on-ignition method, which is cheap, fast, and of low environmental impact, to predict total soil C concentration under the studied management conditions.

RESUMO: O teor de carbono (C) no solo é um indicador sensível de impacto de manejo agrícola. Diferentes métodos são atualmente adotados para determinar o teor de C do solo, o que pode levar a resultados variados e, dessa maneira, dificultar a interpretação dos impactos do manejo. Neste estudo, testamos três métodos de determinação de C comumente usados (combustão via seca em analisador elementar, combustão via seca por perda de massa por ignição e combustão via úmida), com amostras de solo de áreas agrícolas sob diferentes manejos (preparo de área com e sem queima) e de floresta. O método de combustão via úmida subestimou o teor de C do solo em relação ao método padrão (combustão via seca em analisador elementar). Encontramos altos coeficientes de determinação das equações de predição do C total (combustão via seca em analisador elementar), baseada em valores determinados tanto por combustão via úmida quanto por combustão via seca por perda de massa por ignição. Portanto, recomendamos o uso do método de perda de massa por ignição, que é barato, rápido e de baixo impacto ambiental, para prever a concentração total de C sob as condições de manejo estudadas.

1 Introduction

Soil carbon (C) is one of the most sensitive indicators of the quality of agricultural systems (VEZZANI et al., 2008). Detection of the impact of agricultural management on soil C content requires precise analytical methods for the determination of this element (CONYERS et al., 2011). However, several studies have demonstrated that there is a difference in sensitivity among the methods currently used in Brazil to determine soil C concentration (GATTO et al., 2009; CARMO; SILVA, 2012; DIAS et al., 2013). The choice of the analytical method is fundamental for the understanding of the dynamics of C in response to soil management over time (BRYE; SLATON, 2003).

Dry combustion with elemental analysis, regarded as a standard method, is based on the combustion of samples in furnace at 1,020-1,500 °C, wherein the organic molecules are oxidized as $N_x O_y$ and $CO_2-H_2O+O_2$. Subsequently, under helium flow, N_2 and CO_2 are separated in a chromatographic column and determined in infrared cells (RHEINHEIMER et al., 2008). Dry combustion with elemental analysis is widely used in reference laboratories, because of its agility, precision, and capacity to quantify total C, that is, the organic and inorganic forms present in the soil sample (GATTO et al., 2009). Nevertheless, the method presents disadvantages related to high cost per analysis, which is a result of the high cost of purchasing and maintaining the elemental analyzer, as well as to the material used in the analysis (GATTO et al., 2009; CARMO; SILVA, 2012).

The Walkley-Black acid digestion method (WALKLEY; BLACK, 1934), based on wet combustion, is currently the most common method used in soil laboratories in Brazil, because it does not require specialized equipment and shows good accuracy since it oxidizes the most reactive fractions of soil organic matter (NELSON; SOMMERS, 1996). It is based on the reduction of dichromate ($Cr_2O_7^{-2}$) with organic C compounds and on the subsequent determination of the remainder by titration or colorimetry (NELSON; SOMMERS, 1996). However, it presents analytical problems due to its limitation to oxidize forms of C that are more protected or complexed with the mineral fraction of the soil, occurring only a partial oxidation of organic matter, excluding coal and carbonates (SEGNINI et al., 2008; NELSON; SOMMERS, 1996; CANTARELLA; QUAGGIO; RAIJ, 2001). The use of this method also brings environmental damage, because it generates toxic wastes containing chromium (Cr^{6+}). This form of chromium can cause cancer by inhalation, heritable genetic damage, and sensitization by skin contact, besides being very toxic to aquatic organisms (CHEMDAT, 2005).

Mass loss-on-ignition (gravimetric method) is a method of dry combustion which consists in quantifying the loss of mass in the form of C dioxide (CO_2), by incineration of the organic material present in the soil sample (MIYAZAWA et al., 2000). It has the advantage of not producing metal residues toxic to the environment and analysts. However, this method has not been widely adopted in laboratories in Brazil because of its analytical slowness and difficulty of automation (MIYAZAWA et al., 2000).

The Amazon region is one of the major agricultural frontiers in the world, and it can contribute as a source or drain of C (MAIA et al., 2010). Because it is an indicator of soil quality in agricultural and forestry systems, soil C concentration should be determined by accurate analytical methods that meet the characteristics intrinsic to the sample. In agricultural systems in the Amazon region, where fire is traditionally used in land preparation for planting (DENICH et al., 2004), the distribution of C into organic and inorganic forms is modified, which might influence the evaluation of the impact of management on soil C dynamics, depending on the analytical method used.

This work is justified by the lack of studies comparing analytical methods for the quantification of C, by dry and wet combustion, in soils of the Amazon. Therefore, the purpose of this study was to assess the dry and wet combustion methods for soil C determination in agricultural sites under different management conditions (fire and fire-free land preparation) in Ultisol in eastern Amazon.

2 Materials and Methods

Soil cores were collected at the Igarapé-Açú Experimental Farm (FEIGA), Federal Rural University of Amazonia (UFRA), in sites located at the geographic coordinates of 1.07155 S and 47.36127 W, in the municipality of Igarapé-Açú, Pará state, eastern Amazon.

The climate in the region is hot and humid (Am_i) according to Köppen classification, with average temperature ranging from 25 to 32 °C and annual rainfall between 2,000 and 3,000 mm (BASTOS; PACHECO, 1999). The soil in the region is classified as Dystrophic Yellow Argisol (Ultisol) with medium texture (EMBRAPA, 2006). The chemical characterization and particle size distribution of the soil in the study area is shown in Table 1.

Two different soil management systems were assessed: (a) fire-free, chop and mulch of successional forest for land preparation, which consists of the rotation of a consortium cycle of maize (*Zea mays*) and cassava (*Manihot esculenta*) or cowpea (*Vigna unguiculata*) and cassava (*Manihot esculenta*), and a cycle of enriched fallow with rapid growth tree species such as ingá (*Inga edulis*) and taxi-branco or carvoeira (*Sclerolobium paniculatum* Vogel); (b) slash and burn of successional forest for land preparation, followed by the planting of food crops, as previously described, with no enrichment of successional forest during the fallow period. These systems of land use were compared with an adjacent 25-year-old successional forest. The treatments used in this study comprise the successional forest and these two management systems, all with areas of 2 ha each.

There were two cultivation periods in the slash and burn treatment (2002 and 2007); the system had been under fallow for 2.5 years at the time of sample collection. There were three cultivation cycles in the chop and mulch (2002, 2005 and 2006); the system had been under fallow for 2 years at the time of collection.

In January 2010, ten composite samples were collected with the aid of a bucket auger for each treatment at 0-5, 5-10, 10-20 and 20-30 cm depths. Five individual samples were combined to form each composite sample. In the laboratory, the samples

Table 1. Chemical characterization and particle size distribution of soil samples.

Treatments	Depth cm	pH ⁽¹⁾ H ₂ O	P ⁽²⁾	K ⁽³⁾	Na ⁽³⁾	Ca ⁽⁴⁾	Mg ⁽⁴⁾	Al ⁽⁵⁾	H+Al ⁽⁶⁾	SB ⁽⁷⁾	Sand	Silt	Clay
Chop and mulch	0-5	4.75	12.12	17.78	10.94	2.55	0.65	3.87	53.95	3.26	808	72	120
	5-10	4.59	6.62	8.43	6.20	1.00	0.46	11.75	57.49	1.49	757	83	160
	10-20	4.6	4.12	5.51	4.73	0.51	0.31	12.37	47.26	0.85	735	85	180
	20-30	4.7	2.25	4.30	3.64	0.49	0.19	12.37	38.58	0.69	670	70	260
Slash and burn	0-5	4.98	2.62	9.46	7.61	1.85	0.48	3.25	38.40	2.36	807	53	140
	5-10	4.81	2.37	6.86	6.44	1.33	0.44	4.87	40.04	1.79	768	112	120
	10-20	4.64	1.62	4.87	4.67	0.71	0.34	7.12	35.91	1.07	747	93	160
	20-30	4.58	1.00	3.72	4.12	0.41	0.20	9.12	29.74	0.63	658	142	200
Successional forest	0-5	4.66	3.43	8.31	10.23	1.04	0.51	9.25	53.98	1.59	759	121	120
	5-10	4.73	2.00	6.13	7.47	0.48	0.19	10.87	42.30	0.69	708	152	140
	10-20	4.79	1.50	5.99	6.07	0.40	0.19	10.62	37.14	0.61	686	174	140
	20-30	4.85	1.12	4.73	5.02	0.40	0.20	10.12	32.41	0.62	574	206	220

⁽¹⁾ potentiometer in the 1:2.5 soil to water ratio; ⁽²⁾ extracted by Mehlich 1 solution and determined by colorimetry; ⁽³⁾ extracted with HCl 0.05 mol L⁻¹ and determined by flame photometry; ⁽⁴⁾ extracted with KCl 1 mol L⁻¹ and determined by atomic absorption spectrometry; ⁽⁵⁾ extracted with KCl 1 mol L⁻¹ and determined by titration with NaOH 0.025 mol L⁻¹; ⁽⁶⁾ extracted with KCl 1 mol L⁻¹ and determined by titration with EDTA 0.025 mol L⁻¹; ⁽⁷⁾ SB = sum of bases. Source: Albuquerque (2013).

were air dried, smashed, sieved through a 10 mesh screen (2.00 mm), crushed in porcelain mortar, and finally sieved through a 60 mesh screen (0.25 mm).

The Walkley-Black method modified by Cantarella, Quaggio and Raij (2001) was used for determination of organic C content (OC_{WC}) by wet combustion. The following were added to each digestion tube: 0.5 g of soil sample, 10 ml of potassium dichromate (K₂Cr₂O₇) 0.5 mol L⁻¹ (99% purity), and 5 mL of concentrated sulfuric acid (H₂SO₄) (99% purity). After that, the tubes were placed in a digestion block pre-heated at 150 °C and kept at this temperature for 30 min. They were then removed from the block and 50 mL of barium chloride 0.4% (BaCl₂) (99% purity) was added to each tube. Next, 200 ml of distilled water was added to the tubes which, after manual agitation, were kept at rest overnight. Measurements were performed in maximum transmission spectrophotometer with a 650 nm filter.

For determination of total C concentration by dry combustion with loss-on-ignition (TC_{LOI}) (BEN-DOR; BANIN, 1989), approximately 10 g of soil sample were weighed in porcelain crucibles previously dried for 1 h at 105 °C. Subsequently, the samples were put in a muffle furnace (Quimis, model Q 318M). After stabilization of temperature at 400 °C, the samples were kept in the oven for 4 h. After incineration, the samples were dried at 105 °C for 24 h, placed in a desiccator to cool, and reweighed. Organic matter content was determined by the difference between the dry masses before and after ignition. The carbon content was estimated assuming that soil organic matter contains 58% C (NELSON; SOMMERS, 1996).

For determination of total C concentration by dry combustion with elemental analysis (TC_{EA}) (NELSON; SOMMERS, 1982), 0.2 g of soil sample was weighed on porcelain trays and put in a combustion furnace at the approximate temperature of 1,350 °C for oxidation in the presence of oxygen (O₂) with high purity. The analysis was carried out in an elemental analyzer (LECO; model CNS 2000).

The results of C content were subjected to analysis of variance using SigmaPlot 11.0 software program, to test the effect of the different methods of quantification at each depth per land use system. When necessary, data were transformed (logarithm) to meet the requirements of normality and homogeneity of variances. The results, however, were presented as original mean and standard deviation. When significant statistical effect was observed ($p \leq 0.05$), the means were compared by the Tukey test ($p \leq 0.05$). In addition, linear regression analysis of the C concentration obtained by each method was performed.

3 Results

The total organic C content determined by wet combustion (OC_{WC}) was lower at all depths and systems compared with the total C determined by dry combustion with elemental analysis (TC_{EA}) and by dry combustion with loss-on-ignition (TC_{LOI}) (Figure 1). In the successional forest, the average concentrations of TC_{LOI} were higher at all depths (Figure 1A), followed by TC_{EA} and OC_{WC}. In the system with fire for land preparation, the highest averages of TC_{LOI} occurred at the depths of 5-10 and 20-30 cm, not differing significantly from TC_{EA} at the other depths evaluated (Figure 1B). TC_{LOI} was higher at the depth of 20-30 cm in the system with cut and trituration for land preparation compared with the C contents determined by the other methods. At the other depths assessed, TC_{LOI} did not differ significantly from TC_{EA} (Figure 1C).

The analyzed methods showed similar sensitivity in differentiating the systems regarding C concentration at the 0-5, 10-20 and 20-30 cm layers (Figure 1). At the 0-5 cm depth layer, regardless of the method used for determination of C, the chop and mulch system showed the highest C content, and it did not differ significantly from the area of successional forest, which, in turn, did not differ statistically from the area with fire for land preparation (Figure 1). At the 5-10 cm depth layer, TC_{LOI} and TC_{EA} were higher in the chop and mulch system

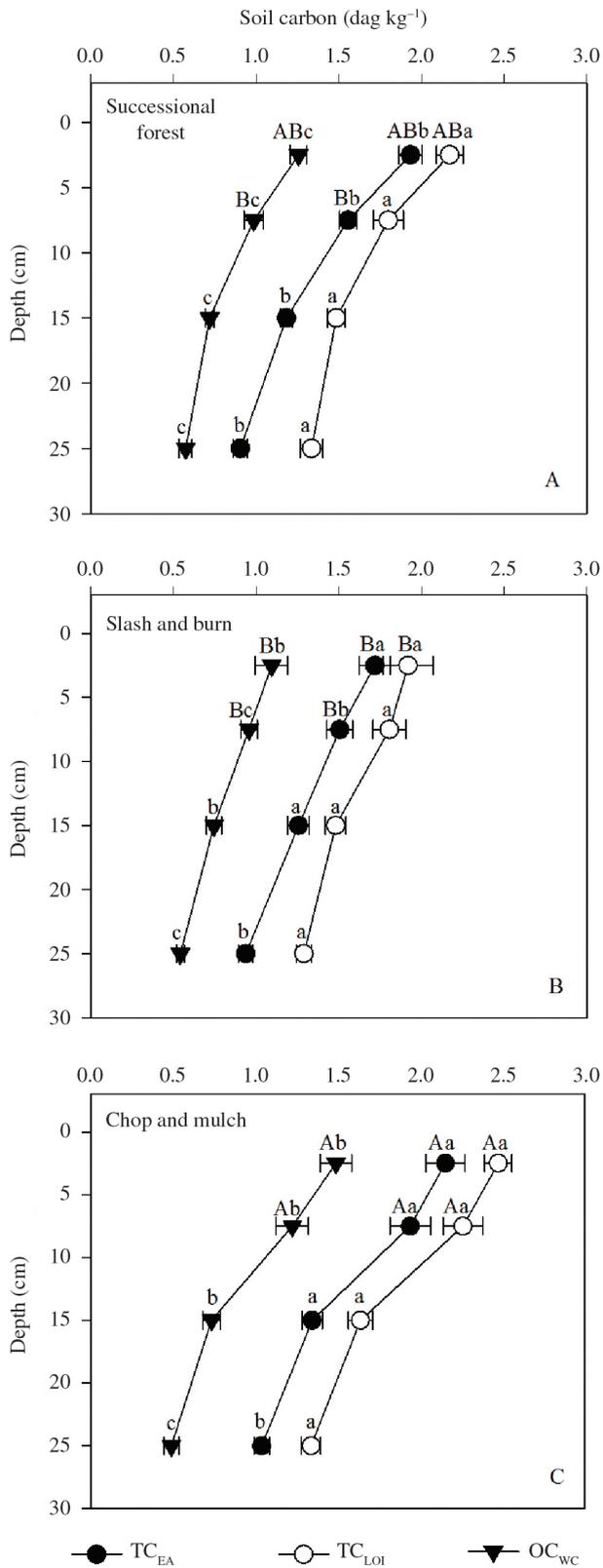


Figure 1. Carbon content in different layers of soil in (A) successional forest, (B) slash and burn system and (C) chop and mulch system, determined by the method of dry combustion with elemental analysis (TC_{EA}), dry combustion with loss-on-ignition (TC_{LOI}), and wet combustion (OC_{WC}). Data are mean \pm standard error ($n = 10$). In a given layer, means in lowercase between methods and uppercase between systems do not differ by the Tukey test at 5% probability.

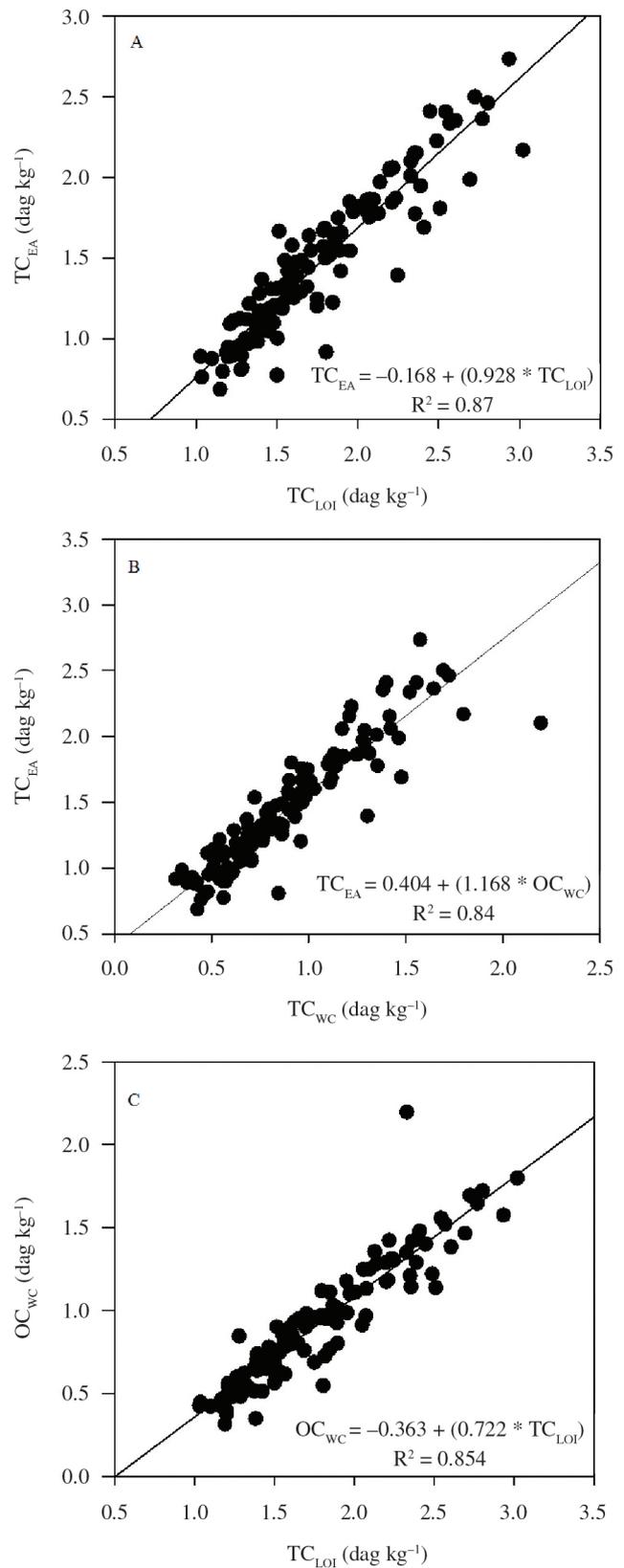


Figure 2. Simple linear regression between carbon concentrations obtained by dry combustion with loss-on-ignition (TC_{LOI}) and dry combustion with elemental analysis (TC_{EA}) (A); wet combustion (OC_{WC}) and TC_{EA} (B); and TC_{LOI} and OC_{WC} (C); $n=120$.

compared to the slash and burn system, and the successional forest (Figure 1); at this layer, the systems did not differ with respect to OC_{wc} .

All methods evaluated presented high determination coefficients (R^2) of regression equations of total C concentrations (Figure 2).

4 Discussion

In this study, carbon concentrations ranged from 0.49 (wet combustion) to 2.48 dag kg^{-1} (loss-on-ignition), staying within the variation range for soils of the Amazon (DESJARDINS et al., 2004). Other studies carried out in the same region found C concentrations, analyzed by wet combustion, ranging from 1.2 to 1.4 dag kg^{-1} in 18-month-old successional forest soils and from 0.8 to 1.8 dag kg^{-1} in alternative cropping system (ARAGÃO et al., 2012); and from 0.92 to 2.61 dag kg^{-1} , at the 0-25 cm layer, in slash and burn and chop and mulch systems, using the dry combustion method (COMTE et al., 2012).

The C concentrations determined by dry combustion and loss-on-ignition were higher than those determined by wet combustion, as observed in other studies (GATTO et al., 2009; SEGNINI et al., 2008; CONYERS et al., 2011; COSER et al., 2012; DIAS et al., 2013). These results are related to the capacity of dry combustion to oxidize all forms of C found in soil, with different forms and levels of protection or complexation with minerals, including C from coal. The wet combustion method is not capable of oxidizing the soil C forms that are more protected or complexed with the mineral fraction and also the inorganic forms (coal, elemental carbon, and carbonates) (NELSON; SOMMERS, 1996; CONYERS et al., 2011). Therefore, the use of wet combustion for quantification of C in soil samples from areas that have a history of fire use can generate underestimated results, leading to erroneous conclusions regarding the impact of soil management and/or use systems in these agroecosystems.

In general, the loss-on-ignition method generated the higher values of C concentration in all systems evaluated in comparison with other methods. This method is applicable for determination of C in Brazilian soils, because they are highly correlated (MIYAZAWA et al., 2000). In these soils organic matter contains a large number of functional groups with oxygen atoms (high degree of oxidation), which favors the total oxidation of organic compounds (MIYAZAWA et al., 2000).

In the superficial layers, where the accumulation of organic matter is more intense, the methods tended to show the same sensitivity in differentiating the land use systems investigated (Figure 1). This response is directly related to the soil organic matter that is available in more labile forms in superficial layers, especially in systems where there is surface deposition of leaf litter (ROSCOE; MACHADO, 2002).

Although the results of OC_{wc} concentrations were lower when compared to the TC_{LOI} and TC_{EA} methods, the sensitivity of these methods in differing the systems was similar. The wet combustion method recovered lower contents of C compared with the dry combustion method, but both showed the same response to distinguish the management systems assessed in

studies of light fractions of organic matter in Yellow Latosol (Oxisol) (COSER et al., 2012).

The methods allowed the adjustment of models with high carbon predictive capacity, especially the loss-on-ignition method, which showed a high determination coefficient in linear regression when related to both the wet combustion and dry combustion methods. The application of the loss-on-ignition method, under the conditions of this study, is a viable and promising alternative, because it provides less unsanitary conditions to analysts and generates less toxic waste compared with the wet combustion method (PIMENTEL et al., 2006), which is the method usually adopted in soil testing laboratories in Brazil. Furthermore, the loss-on-ignition method is more economically viable than the dry combustion method with elemental analysis. Because this is a localized study, the applicability of the results presented in this article in other soil conditions should first be confirmed with samples of the specific areas of interest.

5 Conclusions

The method of wet combustion underestimated soil C contents. The total C determined by dry combustion with elemental analysis can be precisely estimated based on the C content determined by the loss-on-ignition method, which is a relatively inexpensive alternative for obtaining total soil C concentration.

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