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# Comparison of different methods for the determination of total organic carbon and humic substances in Brazilian soils

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## ABSTRACT

Aiming to compare three different methods for the determination of organic carbon (OC) in the soil and fractions of humic substances, seventeen Brazilian soil samples of different classes and textures were evaluated. Amounts of OC in the soil samples and the humic fractions were measured by the dichromate-oxidation method, with and without external heating in a digestion block at 130 °C for 30 min; by the loss-on-ignition method at 450 °C during 5 h and at 600 °C during 6 h; and by the dry combustion method. Dry combustion was used as reference in order to measure the efficiency of the other methods. Soil OC measured by the dichromate-oxidation method with external heating had the highest efficiency and the best results comparing to the reference method. When external heating was not used, the mean recovery efficiency dropped to 71%. The amount of OC was overestimated by the loss-on-ignition methods. Regression equations obtained between total OC contents of the reference method and those of the other methods showed relatively good adjustment, but all intercepts were different from zero ( $p < 0.01$ ), which suggests that more accuracy can be obtained using not one single correction factor, but considering also the intercept. The Walkley-Black method underestimated the OC contents of the humic fractions, which was associated with the partial oxidation of the humin fraction. Better results were obtained when external heating was used. For the organic matter fractions, the OC in the humic and fulvic acid fractions can be determined without external heating if the reference method is not available, but the humin fraction requires the external heating.

**Key words:** Walkley-Black method, dichromate-oxidation, loss on ignition.

## RESUMO

### Comparação de diferentes métodos de determinação de carbono orgânico total de solos e de substâncias húmicas de solos brasileiros

Com o objetivo de comparar três métodos de determinação de carbono orgânico (CO) do solo e de frações das substâncias húmicas, foram avaliadas amostras de dezessete solos do Brasil, de diferentes classes e texturas. Os conteúdos de CO nas amostras foram medidos pelo método da oxidação com dicromato, com e sem aquecimento externo (bloco digestor, 130 °C, 30 min); pelo método de perda por ignição (450 °C/5 h e 600 °C/6 h); e pelo método de combustão a seco. Este último foi usado como referência para medir a eficiência dos demais. Na análise do CO do solo, o método do dicromato com aquecimento externo apresentou maior eficiência e resultados mais próximos do método de referência. No entanto, quando não foi utilizado aquecimento, sua taxa de recuperação caiu para 71%. O conteúdo de CO foi superestimado pelos métodos de perda por ignição. Equações de regressão entre

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os resultados do método de referência e dos demais mostraram bons ajustes, mas com os interceptos diferentes de zero ( $p < 0,01$ ), sugerindo que maior exatidão pode ser alcançada considerando toda a equação e não apenas um único fator de correção. Nas substâncias húmicas, o método Walkley-Black subestimou os conteúdos de CO, o que foi associado à oxidação parcial da fração húmica. Quando o aquecimento externo foi utilizado, melhores resultados foram obtidos. Nas frações orgânicas, se o método de referência não está disponível, o CO nos ácidos húmicos e fúlvicos pode ser determinado sem aquecimento externo. Para a fração húmica, o aquecimento externo é requerido.

**Palavras chave:** método Walkley-Black, oxidação com dicromato, perda por ignição.

## INTRODUCTION

Nowadays there is a great interest in carbon cycling in the context of climate change. The dynamics of this element has been modified as a result of human activities, primarily due to fossil fuel use and secondarily to changes in land use (IPCC, 2013). Changes in soil organic matter contents are directly linked to the modifications of land-use, affecting this important carbon natural reservoir in the Earth. Hutchinson *et al.* (2007) discussed some management practices that contribute to the mitigation of CO<sub>2</sub> emissions by trapping C in soils, indicating challenges and opportunities for policymakers, farmers, and soil scientists.

It is well known the importance of soil organic matter for soil quality and plant growth. Thus, several methods have been proposed for its measurement (Walkley & Black, 1934; Mebius, 1960) and a number of papers discussed the most common ones. The establishment of the relationship among different methods of organic carbon determination is important for soil carbon inventories focusing "Land Use Change and Forestry" evaluations for the Kyoto Protocol and the fitting of data previously obtained by different analytical methodologies (Lettens *et al.*, 2007).

Regarding the soil organic carbon analysis, the dichromate-oxidation method (Walkley-Black) is preferred for routine purposes or when the soils have high carbonate contents. In this procedure, the heat needed for oxidation can be obtained by the energy released from H<sub>2</sub>SO<sub>4</sub> or by an external source such as a hot plate or a digestion block. The temperature of 120 °C obtained from the reaction of concentrated H<sub>2</sub>SO<sub>4</sub> with water and dichromate may be enough to oxidize the active organic carbon forms, but not the recalcitrant forms (Allison, 1960). Usually, 77% of the total organic carbon is determined by this method. Therefore, a correction factor of 1.30 is recommended to convert the amount of organic carbon measured into total organic carbon (Nelson & Sommers, 1982; Gillman *et al.*, 1986).

However, other correction factors are available to compensate this incomplete oxidation. For example, Mikhailova *et al.* (2003) indicated correction factors from 1.09 to 2.27 for Russian topsoils.

The loss-on-ignition method is an easy procedure for soil carbon analysis, requiring equipment available in most laboratories such as a muffle furnace, a drying oven, and a scale (Konen *et al.*, 2002). This method causes partial oxidization of the organic matter using moderate to high temperatures, and the weight lost during the procedure is related to soil organic content. On the other hand, Grewal *et al.* (1991) demonstrated that this procedure may cause structural water loss, especially from oxy-hydroxides, as well as structural changes in some inorganic compounds.

The dry combustion method measures the CO<sub>2</sub> generated by the complete decomposition of organic material at temperatures at or above 900 °C. This method is currently considered as the most reliable, with recoveries close to 100% (Lettens *et al.*, 2007), and it is used as reference method in several studies. However, these elemental analyzers are expensive, which is the main limiting factor for their use.

The aim of this work was to compare the most commonly used methods to measure total organic carbon in soils and humic substances of Brazilian soil samples, as well as to revise the recovery factor used for the dichromate-oxidation method. Some recently studies have made similar comparisons, but using a fewer number of soil classes and none of them evaluated humic substances.

## MATERIAL AND METHODS

Seventeen Brazilian soil samples, including eleven Oxisols, four Inceptisols, one Spodosol and one Histosol (Table 1) were used in the experiment. Samples were collected from the A horizon, air-dried and crushed to pass through a 2 mm sieve. The standard pipette method was used for particle-size analysis (Embrapa, 2011),

indicating contents of sand, silt and clay varying from 100 to 850 g kg<sup>-1</sup> (average: 574 ± 209 g kg<sup>-1</sup>), 40 to 290 g kg<sup>-1</sup> (average: 132 ± 68 g kg<sup>-1</sup>) and 90 to 690 g kg<sup>-1</sup> (average: 295 ± 213 g kg<sup>-1</sup>), respectively.

Humic substances were fractionated according to the alkaline or acid solubility (Schnitzer, 1982). Soil fulvic (FA) and humic (HA) acids were solubilized after extraction with Na OH 0.1 mol L<sup>-1</sup>, while the humin fraction remained in the residual portion. Then, the alkaline supernatant was acidified (pH < 2), promoting HA precipitation while FA remained soluble. The carbon content in each fraction was measured by the dichromate-oxidation method (Walkley-Black), with (WBH) and without (WB) heating at 130 °C, for 30 min (Nelson & Sommers, 1982). Individual contents of total organic carbon (TOC) in fulvic acids, humic acids and humin (expressed in g C kg<sup>-1</sup> soil) were used to calculate the TOC in humic substances with (THSH) and without (THS) heating in the analysis.

Soil samples were crushed to pass through a 500 µm sieve. Organic C was analyzed by dichromate-oxidation method, with (WBH) and without (WB) heating at 130 °C, for 30 min (Nelson & Sommers, 1982), and by the loss-on-ignition (LOI) method of dry weight after 5 h at 450 °C (Davies, 1974) and 6 h at 600 °C (Goldin, 1987). The LOI results were divided by the factor 1.724 to change from organic matter to organic C. Soil samples were further grounded and passed through a 140 µm sieve to measure total C by the dry-combustion method in a CHN-LECO 600.

**Table 1:** Soil classes of studied samples and their particle size composition

Soil	Soil Class <sup>(1)</sup>	Texture (g kg <sup>-1</sup> )		
		Clay	Silt	Sand
1	Oxisol	450	100	450
2	Oxisol	290	90	620
3	Oxisol	470	160	370
4	Oxisol	680	60	260
5	Oxisol	420	40	540
6	Oxisol	220	60	720
7	Oxisol	690	210	100
8	Oxisol	150	170	680
9	Oxisol	100	50	850
10	Oxisol	110	190	700
11	Oxisol	600	110	290
12	Inceptisol	110	170	720
13	Inceptisol	90	290	620
14	Inceptisol	120	200	680
15	Inceptisol	170	120	710
16	Spodosol	110	90	800
17	Histosol	230	130	640

(1) According Soil Taxonomy

Results obtained with this method were considered as reference and used to measure the efficiency of the other methods.

Percent recovery (%R) of organic C (OC) was obtained by each evaluated method and compared with the dry-combustion (DC) method, according to the following equation:

$$\%R = \frac{OC \text{ by evaluated method}}{OC \text{ by dry combustion}} \times 100$$

The recovery factor (RF) for each method evaluated was obtained using the average of percent recovery ( $\% \bar{R}$ ) considering all samples evaluated by the equation:

$$RF = \frac{100}{\% \bar{R}}$$

All results were expressed on an oven-dry basis. All analyzes were done in triplicate. Data were submitted to regression and correlation analysis using the SAEG, a statistical package developed by the Federal University of Viçosa (SAEG, 2007).

## RESULTS AND DISCUSSION

The amount of organic C obtained showed differences among the studied methods (Table 2) and, as expected, were highly correlated with each order, which is in agreement with the literature (Miyazawa *et al.*, 2000; Brunetto *et al.*, 2006; Jankauskas *et al.*, 2006; Pereira *et al.*, 2006; Bianchi *et al.*, 2008). The highest correlation coefficient with the reference method (DC) was obtained by the dichromate-oxidation (Walkley-Black) with external heating ( $r = 0.98$ ,  $n = 17$ ,  $p < 0.001$ ), which indicates the high concordance between the methods. When heating was not used, the correlation was lower ( $r = 0.92$ ,  $n = 17$ ,  $p < 0.001$ ).

The recovered mean (M) of all soils (Table 2) for the dichromate-oxidation method without heating was lower than some reported values. Considering the average of percent recovery obtained for all samples ( $n = 17$ ), the efficiency of this measurement was low, 71% for all soils and 81% considering only the Oxisols. These results indicate that the amount of OC in Brazilian soils is mostly underestimated by routine laboratories, which largely use the dichromate oxidation without heating. If this methodology is used, the recovery factor should be 1.41, or 1.23 for Oxisols. These values are intermediate to 1.33 suggested by Walkley & Black (1934), without using external heating. When external heating was used, the mean recovery was 102%, which is very close to the reference method (DC). Similar accurate estimation using WB with external heating was observed by Wang *et al.* (2012) for Chinese calcareous soils.

On the other hand, the LOI method showed lower concordance with the DC method, as also expressed by the correlation coefficients [ $r = 0.85$  (LOI450) and  $r = 0.90$  (LOI600),  $n = 17$ ,  $p < 0.001$ ]. Also, the estimated organic C amount (M) was almost two times higher than the reference method (DC), when the average of individual percent recovery ( $n = 17$ ) was considered. This result should be related to loss of structural water from oxy-hydroxides and some mineral compounds due to the high temperature, as suggested by Grewal *et al.* (1991); Dias & Lima (2004); Escosteguy *et al.* (2007), and Segnini *et al.* (2008).

Effects of external heat on humic substances estimation were similar to those of soil samples. Using an additional heating source improved recovery power of the method, and the results were more similar to the DC method. These results will be discussed below.

The recovery factor (RF, Table 2) considering the mean value of percent recovery obtained for all samples evaluated was 1.41, 0.98, 0.59 and 0.53 for dichromate-oxidation with and without external heating, and LOI at 450 °C and 600 °C, respectively. The factor close to 1 obtained for Walkley-Black method with external heating indicates high capacity of this procedure for oxidizing all carbon organic presented in the soil sample.

The relationship between total organic carbon contents of the reference method (DC) and those of the other methods may be better described by regression equations (Table 3). The lowest  $R^2$  coefficients of the methods dichromate-oxidation without external heating and loss-on-ignition indicate no linear association between these methods and the DC method, and that the use of a factor may be not a good choice to the final C content in the soil sample. In addition, the intercepts are significantly different from zero ( $p < 0.01$ ) for all equations obtained, which means that more accuracy can be obtained using not a single factor, but considering also the intercept. Some studies forced the intercept to zero to find a single factor, but this was avoided in this study.

When regressions were carried out for the Oxisols, different equations and  $R^2$  coefficients were obtained, although intercepts were still different from zero. This points out the difficult in using a single factor for all types of soils. According to this statement, La Manna *et al.* (2007) verified effects of vegetation type on soil organic C contents, and Lettens *et al.* (2007) noted that RF can be influenced by land use, soil texture and depth. Maybe for this reason, Konen *et al.* (2002) suggested the inexistence of a universal factor applied to different soils, and De Vos *et al.* (2007) recommended a specific

**Table 2:** Organic carbon content (g C kg<sup>-1</sup> soil) from Brazilian soils and their humic substances according to different methods

Id	Soil <sup>1</sup>					Humic substances <sup>2</sup>	
	DC	WB	WBH	LOI450	LOI600	THS	THSH
g C kg <sup>-1</sup> soil							
1	16.0	13.4(84) <sup>3</sup>	17.5(109)	36.2(226)	46.1(288)	9.1(57)	12.4(78)
2	31.1	20.6(66)	25.2(81)	53.9(173)	59.4(191)	16.0(51)	23.5(76)
3	38.5	19.4(50)	40.9(106)	73.8(192)	78.4(204)	21.1(55)	29.0(75)
4	36.7	33.2(90)	38.3(104)	73.4(200)	80.5(219)	18.1(49)	27.4(75)
5	11.9	13.4(113)	14.2(119)	17.7(149)	19.6(165)	7.9(66)	9.3(78)
6	9.0	9.0(100)	10.1(112)	25.4(282)	28.0(311)	6.4(71)	10.7(119)
7	23.8	25.0(105)	28.5(120)	37.6(158)	42.1(177)	12.0(50)	18.5(78)
8	34.8	29.6(85)	34.0(98)	93.5(269)	87.8(252)	18.0(52)	27.1(78)
9	6.1	5.0(82)	9.3(152)	14.6(239)	17.4(285)	2.9(48)	6.1(100)
10	53.2	39.4(74)	39.7(75)	128.3(241)	143.6(270)	41.6(78)	66.4(125)
11	49.2	21.8(44)	41.3(84)	90.0(183)	97.9(199)	30.7(62)	34.2(70)
12	102.2	43.4(42)	99.4(97)	108.2(106)	111.1(109)	68.2(67)	79.0(77)
13	107.5	44.8(42)	102.2(95)	118.5(110)	122.3(114)	93.9(87)	109.2(102)
14	96.2	46.2(48)	94.7(98)	33.6(35)	86.2(90)	59.1(61)	85.9(89)
15	106.6	47.6(45)	114.2(107)	144.2(135)	149.7(140)	61.2(57)	135.5(127)
16	54.3	33.8(62)	49.4(91)	49.9(92)	51.0(94)	51.5(95)	91.5(169)
17	200.7	153.8(77)	185.5(92)	229.1(114)	243.7(121)	146.1(73)	181.9(91)
M <sup>4</sup>	57.5	35.3(71)	55.6(102)	78.1(171)	86.2(190)	39.0(64)	55.7(94)
RF <sup>5</sup>		1.41	0.98	0.59	0.53	1.57	1.06

<sup>1</sup>Methods: dry combustion (DC), dichromate-oxidation (Walkley-Black) with (WBH) and without (WB) external heating, loss-on-ignition at 450°C (LOI450) and 600°C (LOI600). <sup>2</sup>Organic carbon in soil humic substances measured by dichromate-oxidation with (THSH) and without (THS) external heating. <sup>3</sup>Numbers in the parenthesis refer to the percent recovery in relation to DC method (reference) in each sample. For mean value (M), numbers in the parenthesis are the average of all percent recovery ( $n=17$ ). <sup>4</sup>Mean values. <sup>5</sup>Recovery factor considering average of all percent recovery ( $n=17$ )

RF for each laboratory and soil type in order to standardize the results of soil C organic estimation.

These results suggest that the factor 1.33 for soil organic carbon, as recommended by Walkley & Black (1934) and currently in use in Brazilian laboratories, should be revised and other criteria need to be considered for the correction of partial oxidizing reaction observed when external heating is not used. A possibility to increase accuracy of this procedure in routine analysis is to use the dichromate-oxidation method with external heating at 130 °C, for 30 min.

The large difference between the amount of carbon in the total humic substances measured by the dichromate oxidation without external heating (WB) and the reference method (DC) (Table 2) may be due to the low

capacity of the former to oxidize the organic carbon in the humin fraction (Table 4), which is the humic fraction most resistant to decomposition and protected by mineral fraction. This result means that the humin is the main organic matter fraction underestimated by the Walkley-Black method without external heating. When external heating is used (WBH), the efficiency is nearly 100% (Table 2) and much more humin is recovery (Table 4). Therefore, when it is not possible to use the reference method, the amount of carbon in humin fraction should be measured by dichromate-oxidation method with external heating. The humic and fulvic acid fractions are in the extracted solution, so the mean difference between this method with and without heating is about 11%, which is much lower than 42% for the humin fraction.

**Table 3:** Relationship between total organic carbon (y) from the reference method (dry combustion) and organic carbon values (x) measured by different methods evaluated in soil and humic substances

Method	All soils (n = 17)		Oxisols (n = 11)	
	Equation	R <sup>2</sup>	Equation	R <sup>2</sup>
WB <sup>1</sup>	$y = 0.832 + 1.40x^{**}$	0.85	$y = 0.15 + 1.28x^{**}$	0.85
WBH <sup>1</sup>	$y = -0.313 + 1.04x^{**}$	0.96	$y = -0.42 + 1.12x^{**}$	0.86
LOI450 <sup>1</sup>	$y = 0.511 + 0.71x^{**}$	0.63	$y = 0.40 + 0.42x^{**}$	0.95
LOI600 <sup>1</sup>	$y = -0.722 + 0.77x^{**}$	0.80	$y = 0.28 + 0.40x^{**}$	0.95
THS <sup>2</sup>	$y = 0.871 + 1.28x^{**}$	0.95	$y = 0.57 + 1.34x^{**}$	0.95
THSH <sup>2</sup>	$y = 0.510 + 0.94x^{**}$	0.92	$y = 0.75 + 0.90x^{**}$	0.90

<sup>1</sup>Methods used for soil organic carbon: dichromate-oxidation (Walkley-Black) with (WBH) and without (WB) external heating, loss-on-ignition at 450 °C (LOI450) and 600 °C (LOI600). <sup>2</sup>Total organic carbon in soil humic substances measured by dichromate-oxidation (Walkley-Black) with (THSH) and without (THS) external heating. <sup>\*\*</sup> Significant ( $p < 0.01$ ). All values expressed as g C kg<sup>-1</sup> soil.

**Table 4:** Organic carbon in the soil humic fractions extracted with the dichromate-oxidation method (Walkley-Black) with (WBH) and without (WB) external heating

Id	Humic acids		Fulvic acids		Humin	
	WB	WBH	WB	WBH	WB	WBH
g C kg <sup>-1</sup> soil						
1	3.0	3.2	2.0	1.0	4.1	8.2
2	3.1	3.1	5.0	6.0	8.0	14.4
3	13.0	15.1	3.0	4.0	5.1	9.9
4	6.0	7.3	5.0	5.0	7.1	15.1
5	2.0	2.4	2.0	1.0	3.9	5.9
6	1.0	0.8	2.0	3.0	3.4	6.9
7	3.0	3.4	2.0	2.0	7.0	13.1
8	8.0	8.1	2.0	2.0	8.0	17.0
9	1.0	0.7	1.0	1.0	0.9	4.4
10	8.0	9.1	8.0	8.0	25.6	49.3
11	26.0	26.0	2.0	3.0	2.7	5.2
12	25.0	30.0	14.0	15.0	29.2	34.0
13	26.0	32.3	14.0	16.0	53.9	60.9
14	20.0	22.2	13.0	15.0	26.1	48.7
15	26.0	31.0	9.0	10.0	26.2	94.5
16	11.0	12.1	26.0	32.0	14.5	47.4
17	35.0	41.8	7.0	7.0	104.1	133.1
Mean	12.8	14.6	6.9	7.7	19.4	33.4



## CONCLUSIONS

The dichromate-oxidation method (Walkley-Black) with external heating at 130 °C, for 30 min was less affected by soil organic carbon content and texture and should be the procedure for routine purposes in Brazil. Other methods for the same objective should consider the recovery factor, and regression equations must be generated considering local and regional conditions.

For humic substances, whenever the laboratory is unable to use the dry-combustion, dichromate-oxidation method should be used without external heating for fulvic and humic acid fractions, and with external heating for the humin fraction.

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