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An Evaluation of Spectroscopic and Loss on Ignition Methods for Estimating Soil Organic Carbon in Zambian Soils

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Abstract: Soil organic matter information is useful for assessment of soil fertility status. It is normally determined from soil organic carbon (SOC) analysis. However SOC analytical methods may have limitations and be time consuming. Three base extraction methods: $NaHCO_3$, KOH and KOH-EDTA and a loss on ignition (LOI) method were compared to the standard Walkley-Black (WB) method on 21 Zambian soils with various properties. The regression equations obtained were tested on a different set of 12 soils. The $NaHCO_3$ and KOH extracting solutions were found to be weak in detecting SOC. Good correlations were obtained for the SOC estimates between LOI and WB (R = 0.73**) and between KOH-EDTA and WB (R = 0.46*). When applied to soils, the empirical equation obtained for LOI overestimated SOC in soils with relatively high organic matter while the empirical equation for KOH-EDTA slightly underestimated SOC in the same soils. These equations could be improved by grouping soils into testing groups. In the current format, the KOH-EDTA empirical equation seemed suitable for use when rough estimates of SOC values are sufficient, such as in commercial agriculture.

Key words: Soil organic carbon, spectroscopic methods, base extraction, loss on ignition, Zambian soils

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INTRODUCTION

The organic matter content of soil is very important to its chemical and physical fertility. Therefore it is normally determined in routine soil analysis. Analytical laboratories normally determine SOC which is the main component of soil organic matter (SOM). There are several methods available for this analysis and they can be grouped by two primary approaches: (1) dry or wet combustion in which organic carbon is oxidized to and measured as CO_2 and (2) acid hydrolysis of organic matter in the presence of excess oxidant before titration (Nelson and Sommers, 1982). Out of these methods, the dry combustion is a more quantitative and accurate measure of total carbon. However, it requires expensive equipment which only a few laboratories can afford and the soils need fine grind. The titration methods are generally simple and require only basic laboratory equipment, but they do not oxidize all of the organic carbon. Even so, because they are normally well correlated to total organic carbon, titration methods have remained acceptable and popular.

The titration method developed by Walkley and Black (1934) is widely used by soil testing laboratories, sometimes with modifications. This method uses wet digestion of soil in potassium dichromate ($K_2Cr_2O_7$) and concentrated H_2SO_4 followed by redox titration of unreduced oxidant (Nelson and Sommers, 1982). Even with the simplicity of this method, it can become rather tedious and prone to operator error during the transfer of liquids and titration when there are many samples to analyze. The potential for interference by other ions during titrimetric determinations is also high. Another emerging concern that has not received attention locally is the disposal of chromium and the

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strong acid used in the analysis. Currently, laboratories dispose of analytical wastes into the drain where it has a possibility of re-entering into groundwater. In a recent study in Zambia, Chishala *et al*. (2006) observed that vegetables grown using waste water from industries contained levels of chromium significantly higher than the World Health Organization recommended amounts. Hence uncontrolled disposal of chromium is a potential human health risk.

The numbers of commercial soil samples that are being processed by the University of Zambia Soil Analysis Laboratory have been increasing significantly over the years. Soil organic matter information is usually requested for these. Because the laboratory serves the three functions of teaching, research and public service, it has become necessary to examine analytical options that can expedite the processing of samples while reducing the dangers of soil and water contamination. The objective of this study was (1) to evaluate the effectiveness of three extracting solutions using spectroscopy for SOC determination and the loss on ignition (LOI) method, for estimating SOC and (2) to test the suitability of the empirical equations developed from these methods, for use on a different set of soils, especially when accurate values are not required.

MATERIALS AND METHODS

Soils

The soils used in this study were obtained either from the 0-20 cm or 0-10 cm depth of 21 benchmark soils of Zambia (Table 1). They include Alfisols, Entisols, Inceptisols, Oxisols, Vertisols, Ultisols and Spodosols. Tsuji *et al*. (2005) have described these soils. The samples were finely crushed to pass through 40 mesh sieve and they were stored away in plastic bags. Soil pH was measured in 0.01 M $CaCl_2$ using a soil to solution ratio of 1:2.5. Aluminum and iron oxides in the soils were extracted with 0.2 M ammonium oxalate (pH 3) for amorphous oxides (Sah and Mikkelsen, 1986). Aluminum concentration was determined by atomic absorption spectrometry at

Table 1: Some soil properties and estimated soil organic carbon values for twenty-one benchmark soils of Zambia

		Clay	FeO	AlO	WB	LOI	$NaHCO_3$	KOH	KOH-EDTA
Soil	pH	(g/100 g)	(g kg^{-1})						
Bulozi	3.9	3	480	501	17.7	7.1	14.3	13.0	12.1
Chafukuma	4.2	47	840	1790	16.0	13.8	12.9	10.4	13.1
Chibesakunde	4.9	13	530	745	10.3	10.3	9.7	9.0	9.6
Choma-A	4.7	8	90	278	3.6	6.6	10.6	8.2	7.4
Choma-B	4.1	10	70	323	4.0	6.4	9.3	8.2	7.8
Kabuyu-A	4.7	5	80	302	3.9	6.5	9.7	9.1	10.5
Kabuyu-B	5.4	5	50	207	6.2	7.3	11.5	9.5	10.8
Kafue	6.3	59	940	1490	9.8	5.4	8.8	7.8	12.7
Kahare	5.6	7	130	347	7.6	8.2	9.7	8.9	9.1
Kande	3.3	1	60	158	6.7	11.1	12.4	12.4	10.8
Katito-A	4.1	33	39	1720	10.6	7.5	9.7	9.8	11.2
Katito-B	4.7	34	450	1850	12.9	13.2	9.7	9.2	8.3
Liteta	4.7	11	280	1008	8.2	9.2	10.2	9.3	9.9
Malashi	4.9	48	830	1820	14.4	15.3	9.7	10.3	9.4
Mangango	5.1	33	590	1160	20.1	18.9	9.7	11.1	11.1
Meheba	4.3	39	600	1940	16.5	13.6	10.6	9.7	10.3
Misamfu red	4.4	16	310	960	10.9	9.5	8.8	10.6	15.3
Misamfu yellow	4.2	8	260	733	7.0	9.0	8.8	8.7	8.0
Mpongwe	4.3	28	500	1050	11.4	11.2	8.8	9.8	9.6
Mufulira-A	4.2	19	310	712	9.9	10.6	8.4	8.8	8.3
Mufulira-B	4.7	14	470	850	10.8	11.7	9.3	8.9	9.2
Mufulira-C	4.3	12	340	720	6.4	7.8	8.4	8.4	8.1
Mukumbi	5.0	17	170	815	8.1	9.6	9.3	11.1	7.3
Mushemi	5.6	7	190	469	8.9	9.7	8.4	12.6	8.5
Nakambala	5.0	11	210	540	6.6	9.2	8.8	12.8	8.5
Shilenda	4.3	14	200	717	7.4	7.9	8.8	8.7	8.0

FeO/AlO = Iron and aluminum oxides; WB = Walkley-Black organic carbon; LOI = Loss on ignition

309.0 nm with a nitrous oxide-acetylene flame using a Varian-50 Spectra AA and iron concentration was determined at 248.3 nm. Soil particle size analysis was obtained using the pipette method (Gee and Bauder, 1986).

Between January and March 2006, soil organic carbon (SOC) was determined by the Walkley-Black (WB) potassium dichromate wet digestion method (Nelson and Sommers, 1982). This was considered as reference method for the study. Three other extracting solutions were evaluated, namely: i) 0.5 M $NaHCO_3$ (1 g soil was shaken in 20 mL of solution for 30 min, ii) 0.5M KOH (1 g soil was shaken in 20 mL of solution for 2 h) and iii) 0.025M KOH + 0.05 M Na-EDTA (1 g soil was shaken in 50 mL of solution for 2 h). Suspensions were filtered before further processing. All extractions were done in duplicate, at room temperature using an end to end shaker. The absorbance values of all the filtrates were then obtained on a spectrophotometer set at 550 nm wavelength.

Soil weight Loss on Ignition (LOI) was determined by weighing 10 g sample into tarred crucibles and placing them in a 105°C oven for 2 h. The weights were recorded after allowing the samples to cool down. Then they were transferred into a muffle furnace set at 360°C, for 2 h. A second weight was obtained after the samples had cooled down. The LOI (%) was calculated as the difference in weights between 105 and 360°C divided by the 105°C weight. In order to calculate SOC values by the different methods, the wavelength readings from the extraction methods and the LOI values were regressed against the SOC values obtained by the WB reference method. To validate these regression equations, a set of twelve soil samples, different from those used to develop the equations were analyzed using the LOI and KOH-EDTA methods. The readings obtained were used in the regression equations to obtain SOC values for these soils. Means of the values obtained between the proposed and WB methods were separated using Students t-test.

RESULTS AND DISCUSSION

The SOC values obtained by the WB, LOI, $NaHCO_3$, KOH and KOH-EDTA methods are shown in Table 1. Soil organic carbon values estimated by the WB method ranged from 3.6 to 20.1 g kg^{-1}. These are characteristic of the values observed in most tropical and sub-tropical soils. Generally, WB-SOC was found to be well correlated with clay content ($r = 0.58$), Fe oxide ($r = 0.74$) and Al oxide ($R = 0.66$) at $p \le 0.01$, $n = 26$.

The percentage of soil weight lost on ignition (LOI) was well correlated to SOC values obtained by the WB method ($r = 0.73$, $p \le 0.01$, $n = 26$). This relationship was described by a linear regression equation:

$$SOC = 0.54 + 1.23\,(LOI) \qquad (1)$$

Using Eq. 1, estimated SOC values calculated from the LOI values are presented in Table 1. These estimates ranged from 6.5 to 13.8 g kg^{-1}. The equation suggested that a conversion factor of 1.23 could be used to estimate SOC of the soils in the study area when the LOI method is employed. A t-test of the intercept and slope values revealed that an intercept of 0.54% is significantly different from zero at the 0.01% probability level. This observation, termed intercept factor by Grewal *et al.* (1991) and also observed by Goldin (1987) is an indication that there are soil factors other than SOC which contribute to soil weight loss when subjected to high temperatures. The extent of evaporation of hygroscopic and interlayer water adsorbed by clay and sesquioxides and the decomposition of carbonates and bicarbonates are suspected to contribute this intercept factor (Grim, 1967). Consequently, in this study it was observed that LOI was positively correlated with soil clay content ($R = 0.44$), Fe oxide ($R = 0.48$) and Al oxide ($R = 0.53$) at $p \le 0.01$. We did not explore the probability of fitting a quadratic equation to correct for the intercept factor as did Grewal *et al.* (1991), because their results did not show any significant improvement to the linear equation and the quadratic term (LOI^2) has no direct physical interpretation. Therefore the linear equation is more applicable, on physical grounds.

The current observation gave a good indication that LOI could be used as a suitable estimate for SOC. However, among the soils, LOI did not properly capture SOC in the Bulozi, Choma, Kabuyu, Kafue, Kande, Katito and Nakambala soils. These soils have vertic, spodic or siliceous properties. When they were excluded, the correlation of SOC from LOI with WB was improved ($R = 0.95$, $p \leq 0.01$, $n = 16$) and the new empirical equation obtained was:

$$\text{SOC} = -0.29 + 1.25\ (\text{LOI}) \qquad (2)$$

In some cases, LOI underestimated SOC in soils with high clay, Fe oxide or Al oxide content. This could be as a result of differences in the strengths of adsorption that different clay types possess to retain hygroscopic and interlayer water and also because clays may physically protect organic matter from combustion. Ball (1964) reported that most of the water held by soil is lost between 450 and 600°C when the LOI method is used.

Among the extraction methods, $NaHCO_3$ was selected because it can serve a dual purpose in routine phosphorus extraction and analysis. In this study, the $NaHCO_3$ extract was found to be weakly correlated to WB ($R = 0.34$, $p \leq 0.09$, $n = 26$). It underestimated SOC in high clay soils and overestimated SOC in low clay soils (Table 1). Whereas when Savory and Bowman (1986) tested this method they obtained a high correlation with total organic carbon for grassland soils ($R^2 = 0.67$), in a later study, Bowman *et al*. (1991) found the extract too weak for high clay and high organic matter soils. The trend with KOH extraction was similar to that observed for $NaHCO_3$ extraction. Soil organic matter values for the KOH method were weakly correlated with WB values ($R = 0.35$, $p \leq 0.08$, $n = 26$). Again, Bowman *et al*. (1991) reported that KOH overestimated SOC in a Fe-rich acid soil. It has been recognized that lack of pre-treating samples poses a potential limitation to direct extraction with base solutions because clay and $CaCO_3$ can physically protect SOC (Stevenson, 1982).

Out of the three extracting solutions, the KOH-EDTA extraction showed the best correlation with SOC determined by the WB method ($R = 0.46$, $p \leq 0.02$, $n = 26$). Calculated SOC values (Table 1) were obtained from a linear regression equation, vis:

$$\text{SOC} = 0.64 + 1.29\ (\text{KOH-EDTA}) \qquad (3)$$

and they ranged from 7.3 to 13.1 g kg^{-1} with a general conversion factor of 1.29 for all soils. The intercept value of 0.64% was significantly different from zero (t-test, $p = 0.02$), suggesting that KOH-EDTA also may not capture all the SOC. However, the effectiveness of KOH-EDTA is enhanced by the EDTA component which is able to chelate metal cations by disrupting clay-metal-organic matter bridges (Bowman *et al*., 1991). This should expose organic matter and limit any interference effects. Soil organic carbon data in Table 1 show that although KOH-EDTA extraction values were generally lower than WB values, they gave a pattern of SOC distribution most similar to that given by WB method among all soils.

Soil organic carbon for 12 soils different from those used to develop the empirical equations was estimated with the LOI and KOH-EDTA methods (Table 2). The importance of the fineness of sample was tested with the WB method and found to be not significant. Calculated SOC values for LOI and values determined for WB were strongly correlated ($R = 0.77$, $p \leq 0.01$, $n = 12$). However, LOI overestimated SOC in most of the samples with high SOC (such as, Lilayi fallow, Lilayi organic, Lilayi pasture and Chipata). All of these samples were collected from organic cultivation or agro-forestry fallow plots. Soil organic carbon in the soils with high Al and Fe oxides (Meheba and Mufulira) were also overestimated, probably from combustion of these oxides. Soil organic carbon estimated

Table 2: Estimated soil organic carbon values calculated from empirical equations obtained for Zambian soils

Soil	pH	Clay (g/100 g)	WB-C	WB-F	LOI	KOH-EDTA
			(g kg^{-1})			
Chipata	6.42	9.8	11.8	9.7	26.4	9.2
Choma	4.49	8.0	5.1	3.5	9.2	7.2
Field station	7.30	10.8	13.7	12.2	34.8	9.4
Katito	4.75	33.0	15.7	13.9	11.2	10.0
Lilayi fallow	5.95	17.3	24.6	22.1	53.8	14.7
Lil organic	7.15	15.8	39.2	39.0	85.6	15.3
Lilayi pasture	6.54	13.8	38.5	38.8	114.5	14.7
Lilayi rain-fed	4.82	26.8	11.7	10.4	29.4	11.6
Meheba	4.32	39.0	20.3	19.3	50.0	9.9
Mkushi	4.79	10.8	11.1	10.2	23.1	8.7
Mufulira	4.48	19.0	6.0	6.5	53.8	7.6
UNZA farm	5.76	23.8	25.3	26.8	22.8	9.6

WB-C = Walkley-Black using 20 mesh soil; WB-F = 40 mesh soil, LOI = Loss on ignition; Equations used: 1. SOC = 0.54+1.23 (LOI); 2. SOC = 0.64+1.29 (KOH-EDTA)

by KOH-EDTA was well correlated with WB values ($R = 0.81$, $p \leq 0.001$, $n = 12$). In this case, KOH-EDTA values were generally lower than WB values, but they gave closer prediction for more soils. The results suggest that the sensitivity of the proposed base extraction method can be improved when applied to soil testing groups and on low to moderate SOC soils. The high correlations obtained and simplicity of method make the KOH-EDTA option attractive for rapid analysis in commercial laboratories.

In this investigation, a wide range of soils with different properties were used to develop the empirical equations because analytical laboratories in Zambia have not adopted a strategy of grouping soils into testing groups for analysis. The current observations agree with suggestion by Bowman *et al.* (1991) that estimation of SOC is improved when laboratories work with soils within soil testing groups when developing their own empirical equations. Given the good correlation obtained between the WB method and the KOH-EDTA method, it was concluded that the spectroscopic method using KOH-EDTA extraction could be suitable for use in crude estimation of SOC in Zambian soils. This approach is acceptable because SOC values are used as quality indicators that have not been calibrated for crop yields. The empirical equations developed for use could be improved by increasing the number of samples used and limiting the pool of samples to soil testing groups which consider the SOC levels and Al and Fe oxides contents.

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