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Loss on ignition: Measuring soil organic carbon in soils of the Sahel, West Africa

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Traditional methods of measuring soil organic carbon (SOC) are not adequate for accurate evaluation of C sequestration nor to meet environmental safety requirements. Methods that permit accurate assessment of SOC, which are environmentally healthy, and yet feasible for use in developing countries are needed. Initial and ignition temperatures were adjusted so that loss on ignition (LOI) measures of SOC matched measurements by combustion methods. A comparison of combustion, LOI, and Walkley-Black methods was carried out with soils of Mali, West Africa, ranging from 2 - 25 g kg^{-1} SOC. The LOI method, when using initial (preparation) temperatures of 105°C and ignition temperatures of 350°C, related closely with combustion measures (Adj. R^2 of 0.89). The SOC measurements by combustion, however, differed from those of Walkley-Black procedure. The LOI calibration equation was %C (combustion) = 0.03 + 0.36 x %weight loss by LOI, adj. R^2=0.89. The Mali calibration of LOI with combustion SOC was tested on soils of Gambia, however, a systematic over-prediction suggested that re-calibration may be needed to ensure LOI method accuracy with soils different from the calibration set. This need for re-calibration is similar to the Type 2 error, where the sample belongs to a different calibration set.

Key words: Loss on ignition, Sahel, Mali, Africa, carbon sequestration, combustion, Walkley and Black.

INTRODUCTION

Soil organic matter plays a critical role in the conservation of fertility, especially in the extremely coarse textured soils of West Africa (Pieri, 1985). In these soils SOC is both is a source of nutrients and mechanism for nutrient retention, it affects water infiltration as well as increases retention of plant available water, and it provides favorable conditions for soil biota. Several studies have characterized soils of the Sahel in West Africa as poorly buffered, acidic (pH 4.9), sandy and low in soil organic carbon (2 - 5 gkg^{-1}) with a dominance of kaolinite and sesquioxides of Fe and Al (Piéri, 1985, Bationo and Mokwunye, 1991; Kablan et al., 2009). Agricultural

sustainability improves with increases in organic matter in soils of the Sahel so that farmers can continue to grow food and at the same time to maintain a robust and healthy environment (Piéri, 1985). Soil organic carbon, derived from the decay of plants and animals in various forms, is a key component of organic matter playing a great role in increasing soil fertility. Today, a better management of soil organic carbon is needed to enhance soil fertility. As has been emphasized elsewhere, farmers and producers of tomorrow may need to not only farm soil judiciously but to also "farm carbon" (Lal, 2008).

Accurate, rapid and cost-effective soil carbon determination is also important to the development of a soil carbon accounting system (Antle and Uehara, 2002). Unfortunately, soil organic C measurement in developing countries is difficult because accurate methods are either not environmentally acceptable or they are too expensive,

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requiring costly instruments and highly trained technicians. Traditionally soil organic C determination relies on two methods and each method has its own limitations: (1) Oxidation of organic matter by $K_2Cr_2O_7$ (Walkley and Black, 1934), and (2) Dry combustion at 1350°C (Nelson and Sommers, 1996).

The Walkley-Black method only estimates a fraction the soil C (Allison 1935). Moreover, the method is time-consuming, it subjects the analyst to handling highly dangerous concentrated sulfuric acid, and it produces hazardous waste including the chromate ion (Swift, 1996). In addition, the incomplete oxidation of organic carbon often results in erroneous values (Heanes, 1984). Dry combustion, on the other hand, is usually regarded as the most accurate method (McCarty et al., 2002); however, the method is not widely available in the Sub-Saharan tropics because of the high cost of combustion analyzers, developed infrastructure for maintenance and reagent requirements and the need for highly skilled operators.

Loss on ignition (LOI) methods has long been used by soil testing laboratories to measure soil organic matter and soil organic carbon. Mitchell (1932) reported that temperatures of between 350 and 400°C were suitable. Davies (1974) illustrated that even a temperature of 430°C could be used in the presence of calcium carbonate without errors. However, higher temperatures can drive off structural water of clays causing decomposition of carbonates and hydrated salts increasing error associated with higher temperature use. Schulte and Hopkins (1996) developed an LOI method that has been successful in the North Central Region of the USA. Konen et al. (2002), using the method of Schulte and Hopkins (1996), which included pretreatment drying at 105°C and 2 h ignition at 360°C, reported regression relationships with SOC determined by combustion ranging from R^2 of 0.94 - 0.98, but slopes of the regressions differed by nearly a factor of 2 between soil groups of different states (0.57 to 1.14). Konen et al., (2002) concludes that LOI methods are inexpensive, rapid, and require a minimum of expensive equipment, but that unique relationships exist for soils from differing geographic locations, possibly due to varying amounts of soil clay, silt, and differing mineralogy.

This study sought to evaluate a loss on ignition (LOI) method as an alternative to combustion methods and dichromate methods in soils of the Sahel of West Africa, but with the objective of duplicating combustion results. The published studies discussed above reveal that a variety of initial (preparation) and ignition temperatures have been used. The objectives of this study were:

(1) To compare various initial (preparation) and ignition temperatures of the LOI method in order to duplicate combustion method results, which are generally considered the most accurate.
(2) To compare three methods of measuring soil organic carbon (SOC): LOI, dry combustion, and the Heanes (1984) adaptation of the Walkley-Black (1934) dichromate method.

MATERIALS AND METHODS

Soils

Mali: Twenty-five soils from Mali were selected and sampled to provide clay contents ranging from 30 - 600 g kg^{-1} and organic C ranging from 2 to 25 g kg^{-1} C (Table 1). Soils were collected in regions with representative agricultural systems of Mali, including potential C-sequestration sites: Cinzanna (millet); Konobougou (cotton, maize, millet); Oumarbougou (cotton, millet, maize); Niono (irrigated rice); Niessoumana (cotton, irrigated rice) Longorola (cotton, maize, cereal, irrigated rice); Fansirakoro (millet, maize). Prior to analysis soil samples were dried, finely ground to pass a sieve of 60 mesh (0.251 mm).

The Gambia: A set of 144 samples from Gambia were included in this comparison as a test of the LOI calibration on soils from Mali. The Gambian soils were obtained from 72 locations in an experiment to test for effects of *Amenagement en Courbes de Niveau* (ACN) on C sequestration (Doumbia et al., 2009). The soils of Gambia were prepared similar to those of Mali. Rainfall at the Gambian site was substantially higher than that at the Mali sites – a mean of approximately 1200 mm compared to a range between 450 - 1000 mm.

Objective 1. Determine the combination of selected initial (preparation) and ignition temperatures of LOI methods that gives results most similar to the combustion method of measuring soil organic C.

The LOI procedure described by Nelson and Sommers (1996) was used as the basic method. Five gram of sample was placed in 30 ml crucibles that have been pre-weighed and pre-heated at 105°C, then cooled. Crucibles with soil samples were then handled as follows:

(1) Heat in a drying oven at the initial (preparation) temperature over night,
(2) Cool to room temperature in a desiccator over silica gel,
(3) Weigh the crucibles + sample to the nearest mg. Reweigh at 105°C.
(4) Place into a muffle furnace and heat at the ignition temperature for 16 h.
(5) Remove from the furnace and cool to room temperature in a desiccator.
(6) Weigh the ignited samples to the nearest mg and record weight.

The mass of soil loss expressed on a dry weight basis by the following equation:

LOI (g kg^{-1}) = (Weight$_{initial\ T}$ – Weight$_{ignition\ T}$)/ Weight$_{initial\ T}$ x 1000.

Table 1. Selected soils and representative agricultural systems of Mali and their range in soil clay.

Sample	Sampling locations	Depth (cm)	Agricultural systems	Clay, g kg^{-1}
1	Cinzana Sol 1	0-20	Millet	52
2	- « -	20-60	- « -	124
3	Diou	0-20		110
4	- « -	20-60	- « -	40
5	Niessouma bas-fonds	0-20	Cotton, irrigated rice	454
6	- « -	20-60	- « -	520
7	Cinzana Sol 3	0-20	Millet	56
8	- « -	20-60	- « -	182
9	Cinzana Sol 5	0-20	Millet	476
10	- « -	20-60	- « -	538
11	Konobougou	0-20	Cotton, maize, millet	72
12	- « -	20-60	- « -	140
13	Longorola Glacis	0-20	Cotton, maize, cereal	88
14	- « -	20-60	- « -	342
15	Oumarbougou	0-20	Cotton, maize, millet	308
16	- « -	20-60	- « -	96
17	Cinzana Sol 4	0-20	- « -	70
18	- « -	20-60	Maize, Millet	166
19	Fansirakoro	0-20	- « -	104
20	- « -	20-60	Millet	72
21	Cinzana Sol 2	0-20	- « -	22
22	- « -	20-60	Irrigated rice	420
23	Longorola bas-fonds	0-20	Irrigated rice	102
24	Danga	0-20	Irrigated rice	426
25	Moursi	0-20	Irrigated rice	278

The loss in weight of the soil is taken as a raw measure of the organic carbon content.

A set of initial temperatures, usually designed to eliminate of adsorbed water, was selected as was a set of ignition temperatures as follows: Two drying temperatures: 105 and 120°C were employed reflecting differences by existing laboratories. Ignition temperatures: several temperatures have been used ranging from 350 to 600°C (Davies, 1974; Heanes 1984, White and Roth, 1984). A range of ignition temperatures from 350 - 600°C in 50°C increments was tested. This combination of initial, preparatory and ignition temperatures formed the basis of the exploration to calibrate the LOI method with combustion measurements of soil C. The comparison of temperatures was carried out using a Thermolyne (Use of a specific brand name does not imply a recommendation by the University of Hawaii or any institutions of other authors of this manuscript.) muffle furnace, Model F-11730, 220v.

Objective 2. Comparison of methods of determining soil organic carbon: The soil organic carbon in the samples was measured by three methods:

(1) Loss on ignition: The LOI method, developed for Objective 1, was used for the comparison of three methods.

2) Modified Walkley and Black or dichromate oxidation (method: The analyses were performed at the Soil and Plant Laboratory in the Tropical Plant and Soil Science Department of University of Hawai`i using the modified dichromate oxidation method (Heanes, 1984). According to the procedure, $K_2Cr_2O_7$ was added to the soil in an acidic solution for quantitative oxidation of organic C. In an attempt to achieve complete oxidation of organic C, the finely ground soil was digested in test tubes by heating externally in a drying oven at 150°C with 0.167 M $K_2Cr_2O_7$ mixed with concentrated H_2SO_4. The excess dichromate was quantified colorimetrically to assess how much reacted with the soil C. The amount of reacted dichromate was roughly proportional to soil organic carbon content.

(3) Dry combustion: Soils were ashed at 1350°C and the released CO_2 was measured with a LECO CNS-2000 analyzer (Nelson and Sommers 1996).

Statistical analysis

The wet oxidation and the combustion procedures were compared in a 1:1 plot on the base of the mean error, ME = Σ ($X_{pred.}$-$X_{obs.}$)/n, (n is the number of observations) and the root mean squared error (RMSE). If the ME is zero
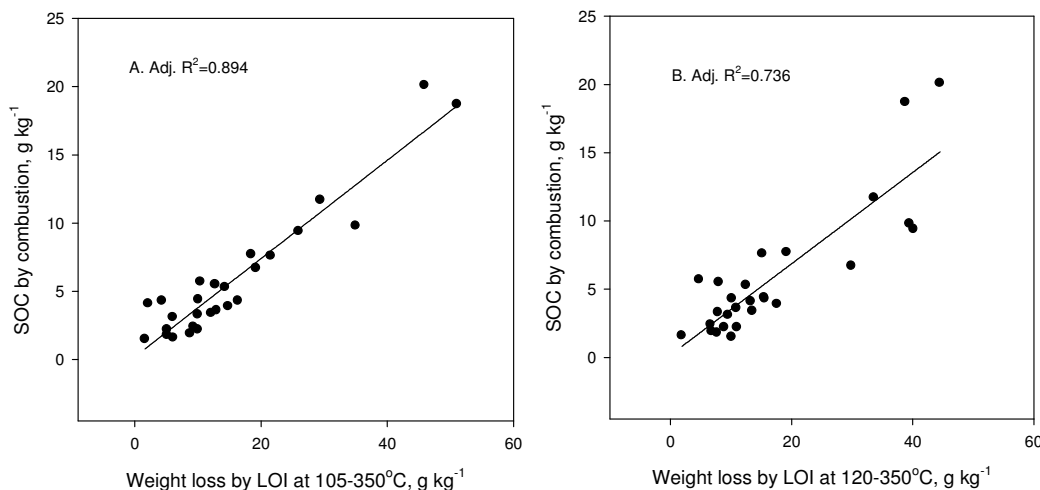
Figure 1. Calibration curve between soil organic carbon SOC (g kg⁻¹) by combustion and the weight loss (g kg⁻¹) by LOI at the two temperatures (A): 105 - 350 °C, (B): 120 - 350 °C.

Table 2. Comparison among LOI initial and ignition temperatures and dry combustion soil C.

LOI temperature combinations: Initial – Ignition (°C)	Intercept[1]	Slope[1]	Adjusted R²	Residual mean square error (RMSE)
120 - 350	0.1733	0.3344	0.736	5.93
120 - 600	0.2239	0.1276	0.693	6.91
105 - 350	0.1958	0.3602	0.894	2.37
105 - 400	0.4641	0.2659	0.736	5.95
105 - 450	0.5553	0.1668	0.684	7.09
105 - 500	0.3969	0.1362	0.702	6.83
105 - 550	0.3351	0.1337	0.731	6.05
105 - 600	0.3828	0.1318	0.744	5.76

[1]Carbon by combustion = intercept + slope x LOI weight loss.

then there is no bias indicating a high accuracy, on the average, for the calibration model, but if the ME is positive then there is a systematic over-prediction, if it is negative then there is systematic underprediction (Loague and Green, 1991). Data were analyzed using Minitab 14. The accuracy of the prediction was determined based on the AdjR² and the root mean square error (RMSE).

RESULTS AND DISCUSSION

Effect of varying initial, preparatory temperatures on LOI estimates of soil C

Figure 1 shows that some soils depart from the regression line, particularly when the initial preparatory temperature was 120°C. In this range weight loss did not correspond with differences in combustion C. The disparity seemed particularly large on soils with LOI in the

range 30 to 40 g kg⁻¹, which may be due to two reasons: differences in organic matter composition among soils or the presence of volatile inorganic components that lose weight during prolonged heating at 350°C (Figures 1 and 2) (University of Georgia, 2003).The Adjusted R² of 0.894 with the 105 to 350°C combination, which was the highest, suggests a minimum of errors associated with those temperatures.

The ignition temperatures may explain much of the variation in the measured organic C content. The weight loss increased when the ignition temperature was higher (Table 2). Ben Dor and Banin (1989) reported that the loss of mass from the soil might be subdivided into several stages:

(i) Hygroscopic water loss (50 to 100°C),
(ii) Organic matter loss (100 to 400°C),
(iii) Thermal reactions such as dehydroxylation of phyllosilicates (200 to 700°C),
(iv) Decarboxylation of carbonates (700 to 1000°C).

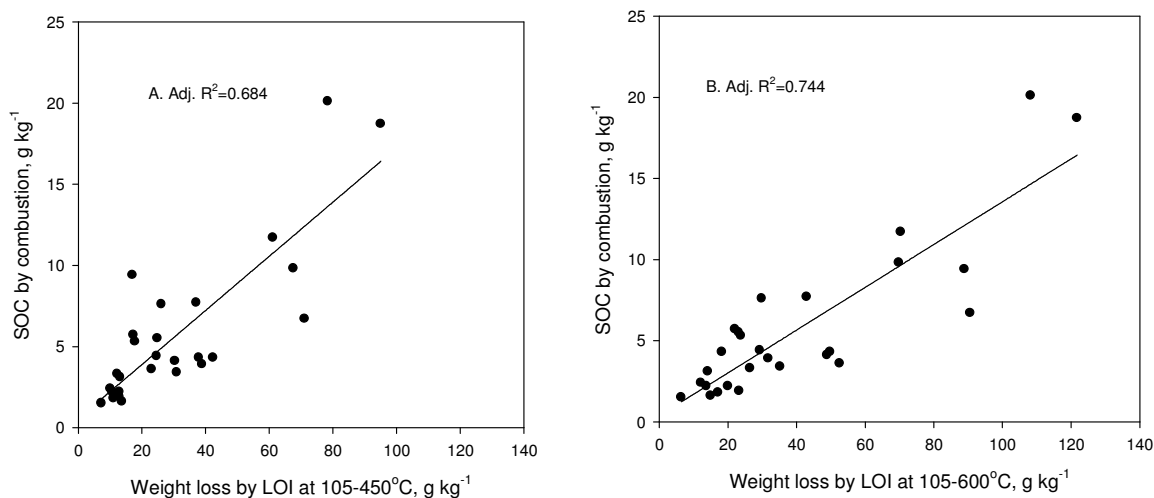**Figure 2.** Calibration curve between soil organic carbon (SOC) (g kg^{-1}) by combustion and the weight loss (g kg^{-1}) by loss on ignition (LOI) at the two temperatures (A): 105 - 450 °C, (B): 105 - 600 °C.
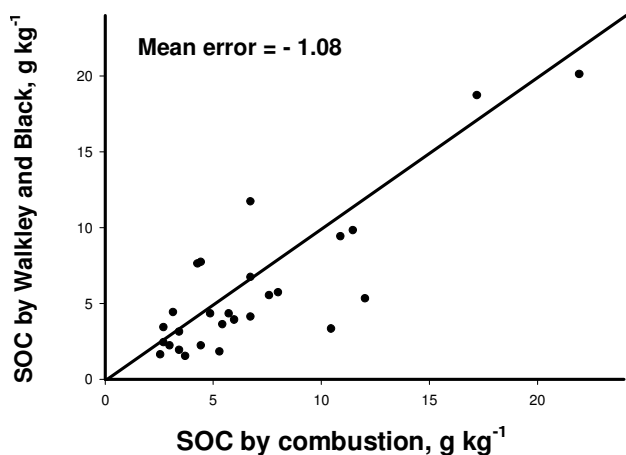

Figure 3. Comparison (1:1 line) between soil organic carbon (SOC) measured by the combustion method and the Walkley – Black method (dichromate) in 25 soils of Mali, West Africa. A mean error of -1.08 indicates that averaged for all samples dry combustion analysis values were 1.08 g kg^{-1} larger than Walkley-Black values.

Effect of varying the ignition temperature

When the LOI was carried out at the highest temperature (600ºC) it resulted in a considerably greater loss of weight than when lower temperatures were used (Figure 2). The Adjusted R^2 of the comparison of LOI at 600 °C with the combustion estimate of C was much lower than the one at 350 °C (Table 2). The higher uncertainty of LOI estimates at the ignition temperature 600 °C was probably due to dehydroxylation and decomposition of inorganic constituents resulting in increased weight losses and subsequent inaccurate estimate of organic matter content (Heanes, 1984; White and Roth, 1984). The uncertainty is more pronounced with high clay soils that contain gibbsite with small amounts of soil carbon, particularly in subsoils (Heanes, 1984; White and Roth, 1984). These authors point out that gibbsite tends to lose much water at 300 °C. Thus, the discrepancy could be due to the fact that hydrated aluminosilicates, carbonate minerals, and some hydrated salts are decomposed at the high temperatures (White and Roth, 1984; Ben Dor and Banin, 1989). The careful control of heating temperatures and the use of an analytical balance with mg accuracy is needed to improve method precision.

Table 2 illustrates the results of the performance of combinations of initial LOI temperatures with increasing combustion temperatures. The Adjusted R^2 for the 105 - 350 °C combination provided the closest approximation of SOC measured by the combustion method. Our results corroborate those obtained by previous researchers, who also found a strong correlation between combustion or wet oxidation and loss-on-ignition (LOI$_{105-350}$ °C) (Ben Dor and Banin, 1989; Davies, 1974). Given the regression Y = 0.36X + 0.03, where X is the weight loss resulting from LOI$_{105-350}$ °C and Y is the soil carbon measured by the combustion method, the 95% confidence limits would cover all data points (data not shown).

Comparison between the Walkley and Black and combustion methods

There was a moderate agreement between the determination of SOC by the Walkley and Black (W and B) and the dry combustion procedures (Figure 3). The
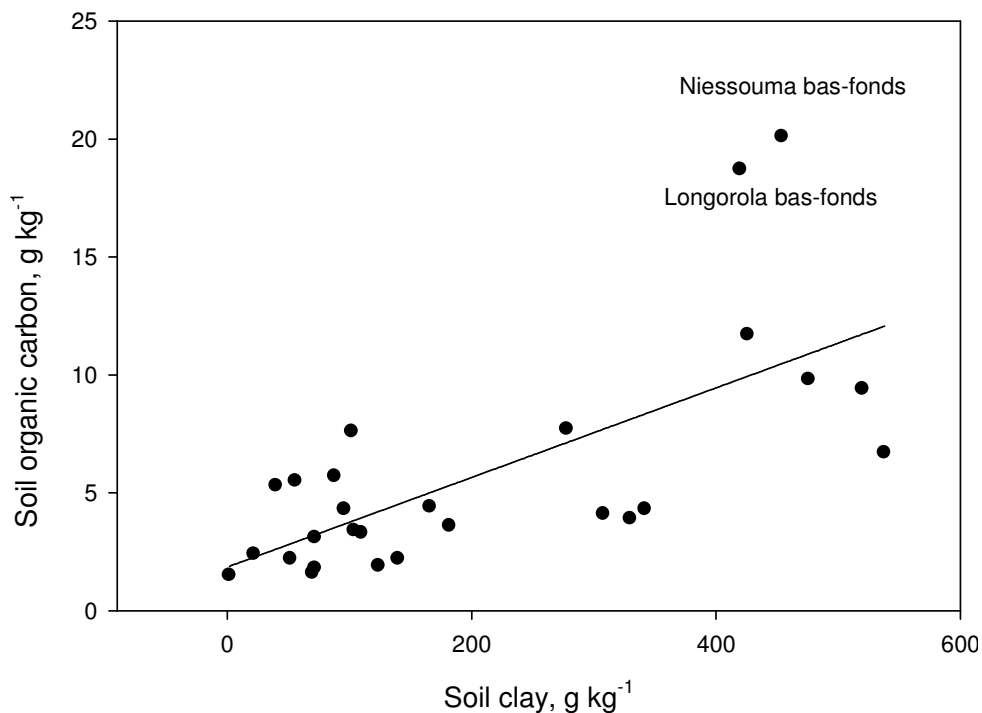
Figure 4. Relationship between C (g Kg^{-1}) by combustion and clay for 25 soils from Mali (SOC = 1.854 + 0.019 Soil Clay, Adj. R^2=0.45.

mean error (ME) of −1.08 g kg^{-1} indicated a systematic underprediction by the W and B method in comparison with the combustion method. The Root Mean Square Error (RMSE) of the relationship between the combustion and W and B = 0.054.

Organic C and soil clay

The amount of soil clay was determined using the pipette sampling method (Gee and Bauder, 1986). Clay minerals play an important role in storage and long-term stability of organic matter in soils (Stevenson, 1982). Clay minerals have a high specific surface area and carry a charge, binding and thereby chemically stabilizing organic matter. The results from Figure 4 with an R^2 of 0.45 illustrate a poor relationship between soil clay and organic C content up to 400 - 500 g kg^{-1} clay. Above 300 to 400 g kg^{-1} of clay the relationship between soil clay and the C content was even less apparent. It may be noteworthy that two soils that exhibited particularly high amounts of soil C than most soils were from poorly drained soils (bas fonds) that may have led to greater amounts of 2:1 clay minerals (Figure 4). Factors such as type of clay mineral have been reported to affect LOI weight loss in soils (Ben-Dor and Banin, 1989). It may be that the water contained in the interlayer caused an increase of the weight loss (phyllosilicate dehydroxylation) (Ben-Dor and Banin, 1989). In addition, the amount of clay-bound organic matter may differ between kaolinite and smectite (Ben-Dor and Banin, 1989; Nelson and Sommers, 1996). It can be seen in the graph that low organic carbon is associated with low clay. Specifically, as clay contents increased a linear increase occurred in organic C concentrations. Only a weak regression (R^2 = 0.45) existed for soil clay versus organic C concentrations. Several previous studies have revealed strong correlations between soil C content and clay content (Nichols, 1984), but the correlation varied among soils with different iron and aluminum oxide content. Kaolinite and gibbsite are the predominant types of clay existing in Mali soils (PIRT, 83; Doumbia, 1994).

Soil carbon prediction of unknown samples

In the study the best calibration model was chosen on the basis of highest regression prediction (R^2 = 0.89) and the lowest values of RMSE (Table 2). The accuracy of C prediction in unknown samples depends on the accuracy of the constructed model, which must also capture the variability in soil C of the sites represented in the original calibration. However, it is clear that the soils differ in degree of weathering, mineral composition and structural water all of which affect LOI results, and which can result in significant error. In order to test the transferability of the LOI calibration developed with Mali to soils in other countries of the Sahel, soil samples from The Gambia
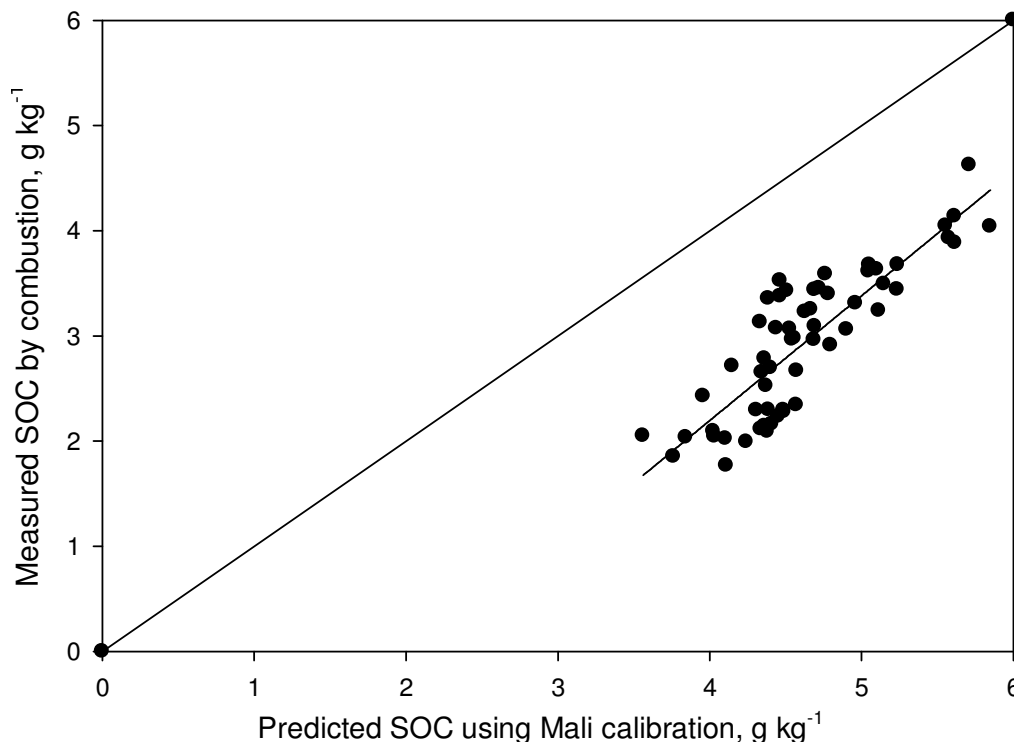
Figure 5. Predicted vs. observed soil organic C, g kg^{-1} for 144 samples from Gambia, using the Mali calibration. Calibration for Gambia samples: Combustion SOC = -2.548 + 1.186*LOI value, Adj. R^2 = 0.73.

were analyzed and LOI C predictions were made.

As illustrated in Figure 5 with the 1:1 line, all the sample results for combustion analyses were below the 1:1 line indicating a systematic over-prediction by LOI based on the Mali model. The Mali model was not able to accurately predict combustion carbon in the soils of The Gambia. This type of error could result from an under-estimate of loss of structural water in soil from The Gambia. The greater presence of minerals with high water contents, such as amorphous oxides, may result in the over-prediction. It is interesting to note, however, that the relationship between combustion and LOI predictions of soil C for the soils of Gambia continued to linear, but shifted downwards, consistent with the possibility of greater amounts of weight loss in the Gambia samples for the same amount of soil organic C as in the Malian case.

The cause of the over-prediction of soil C by the LOI method in Gambian soil subset is not known, but it is a clear warning that accurate prediction of soil organic C by the LOI method requires that the unknown soils be among those in the calibration set, else substantial errors can occur. The over-prediction indicates that some basic properties of Mali soils differ from those of the Gambian subset. These results indicate that re-calibration is needed to apply the LOI method to regional soils of The Gambia. Thus the Gambian soils belong to a differing

population of soils with respect to LOI estimation of soil organic carbon.

One further concern for both the combustion and LOI methods in Sahelian soils is that neither method distinguishes between charcoal, coal fragments, and soil organic matter. It is obvious that C derived from the fragments of coal does not function in the same manner as organic matter in the soil.

Conclusion

The soil carbon in soils of Mali measured by the combustion method in g kg^{-1} can be estimated by an LOI method using a simple calibrated regression equation Y = 0.36X + 0.03, where X is the weight loss in resulting from ignition of the sample from - 105 - 350°C. The 95% confidence limits, ± 2.5 g kg^{-1} would cover all data points (data not shown). The LOI method appears to be useable for the range of soils in this set collected in Mali, but not for soil samples collected in The Gambia. The method is easier and more convenient than the expensive dry combustion method, especially for developing countries. The LOI method, thus calibrated, presents a practical and relatively rapid alternative for determination of soil organic carbon in soils of the Sahel of Africa, although re-calibration will be needed if the unknown sample is from

a different calibration set. Based on these results it appears necessary to check the calibration of the LOI method unless one can be certain that the unknown soils belong to the same population of soils as the calibration set. Thus cautious testing of a new set of soils needs to be considered when using this method.

ACKNOWLEDGMENTS

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